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Impact of the thickness of phthalocyanine films and its metal complexes on optical and electrical properties

The paper presents the results of a study of the structural, optical and electrophysical characteristics of phthalocyanine films and its metal complexes with different thicknesses. Films of phthalocyanine and its metal complexes were obtained on the conductive surface of the FTO by thermal evaporation in vacuum. It is shown that the observed broadening of the B and Q bands and the hypsochromic shift of the maxima in the absorption spectra are associated with the central atom. With a decrease in the thickness of MPC films, there is a decrease in the value of the half-width of the absorption spectra. The effect of the thickness of phthalocyanine films on the efficiency of generation and transport of charge carriers in the FTO/MPC/Al cell was studied. It is shown that the VAC increases almost 2.5 times compared to a photocell based on a metal-free phthalocyanine. From the obtained impedance measurement data, it was found that the electrophysical parameters of phthalocyanines depend on the values of optical density, broadening of absorption bands in the short-wave and visible spectral regions, which is consistent with the data of the volt-ampere characteristic.

Keywords: phthalocyanine, metal phthalocyanines, solid films, absorption spectra, impedance meter, current-voltage characteristics.

Introduction

Phthalocyanines are currently widely studied molecular semiconductors, which is due to both applied and scientific reasons. Phthalocyanines are large aromatic organic compounds constructed from four isoin-dole units connected by nitrogen atoms. Phthalocyanines are chemically and thermally stable, most of them are organic p-type semiconductors and have photoconductivity, easily forming ordered thin films [1, 2]. This determines their use as media for optical recording of information, for the manufacture of light-emitting diodes (LEDs), solar cells, gas sensors, high-speed magnetic field sensors [3-5].

In recent years, there has been great interest in phthalocyanines with central atoms of various metals (metallophthalocyanines — MPCs). Groups of metal phthalocyanines are formed as a result of the introduction of divalent metal atoms (e.g. Zn, Cu, Co and Ni) into the phthalocyanine ring. The physical and chemical properties of MPCs are well regulated by optimizing the central metal ions and their structure [6]. Also, the correct choice of morphology and polymorphism of the MPCs thin film structure can have a significant impact on its optical and electrical properties [7].

Due to the remarkable electronic and optical properties, MPCs thin films have become a promising material for organic electronic devices [8, 9]. For example, in recent years MPCs have been implemented in perovskite solar cells as hole transfer materials (HTM), achieving in some cases efficiency exceeding 20 % [10-11]. MPCs are also attractive building blocks in molecular donor-acceptor (D-A) systems due to their ability to efficiently absorb light in the visible region, as well as their ability to act as electron (D) donors during photoexcitation in various D-A systems [12, 13].

Thus, phthalocyanine and its metal complexes have a wide range of potential applications in electronics, energy, chemistry and medicine. Research in this area is ongoing, and it may lead to new and innovative applications of these materials.

In this work, films of phthalocyanine and its metal complexes with different thicknesses were obtained by thermal spraying in vacuum. We studied the effect of film thickness on structural, optical and electrical properties, as well as conducted a detailed analysis of charge transfer processes in phthalocyanine films.

Results and Discussion

Structural analysis of the prepared films

Figure 2 shows SEM images of the surface and transverse cleavage of phthalocyanine films and its metal complexes. As can be seen from the figure, the morphology of the film surface depends on the thickness of the sample deposition. For a film of nonmetal phthalocyanine (H2Pc) and zinc phthalocyanine (ZnPc), a continuous fine-grained surface is observed, with a decrease in the thickness of the film, the surface becomes more homogeneous. Cobalt phthalocyanine (CoPc) and copper phthalocyanine (CuPc) films have a coarser-grained structure, with a decrease in film thickness, a smooth and fine-grained surface is observed. All MPc films were obtained by vacuum spraying under the same conditions, and as a result, the thickness of the vacuum-deposited films is approximately the same.

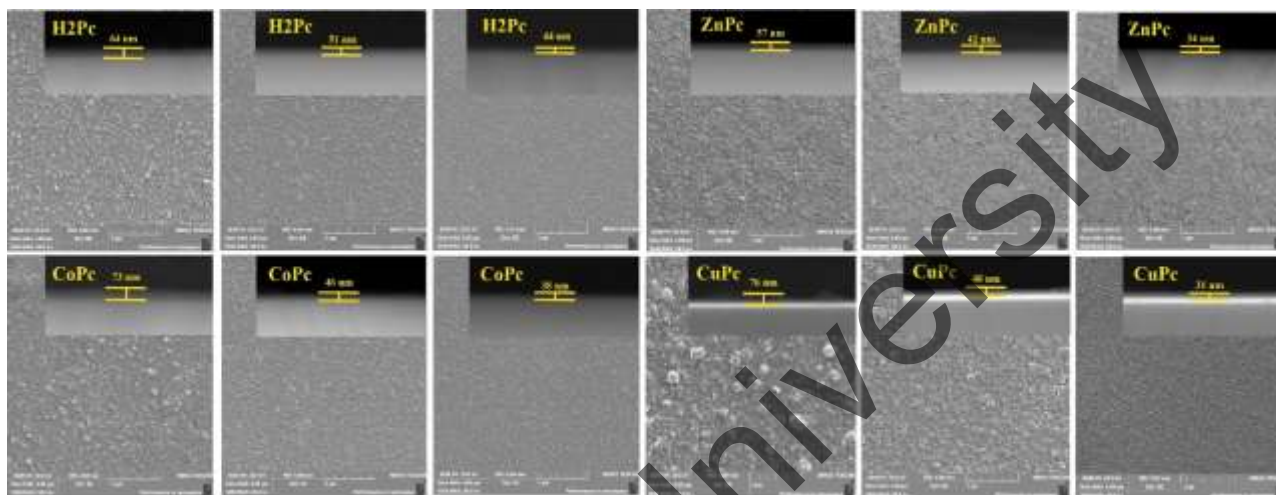


Figure 2. SEM images of phthalocyanine films and its metal complexes with different thicknesses.

Optical properties

Figure 3 shows the absorption spectra of vacuum-deposited MPc films. There are two intense bands in the absorption spectra: in the region of 300-450 nm (B-band), which correspond to the mixed π - π^* and n- π transitions $a_{2u} \rightarrow 2e_g$ and $b_{2u} \rightarrow 2e_g$, and the absorption band in the region of 550-700 nm (Q-band), which corresponds to the π - π^* transition $a_{1u} \rightarrow 2e_g$ [14-15]. As can be seen from the figure, the absorption band of H2Pc, ZnPc and CoPc films in the B-band region has a maximum at a wavelength of $\lambda = 340$ nm. In the Q-band, two maxima are also observed at wavelengths $\lambda = 630$ nm and $\lambda = 690$ nm. In turn, for CuPc films, a hypsochromic shift of absorption maxima $\lambda = 227$ nm is observed in the B-band region, as well as a shift of maxima in the Q-range $\lambda = 485$ nm and $\lambda = 553$ nm. With a decrease in the thickness of MPc films, there is a decrease in the value of the half-width of the absorption spectra. This is due to the fact that a decrease in the thickness of the films leads to a decrease in the local environment of the absorption center, through which energy is absorbed. A decrease in the local environment of the center in the films leads to a greater probability of photon scattering and, as a result, to a decrease in the half-width of the absorption spectra. Table 1 shows the characteristics of the absorption spectra of the B and Q ranges of MPc films.

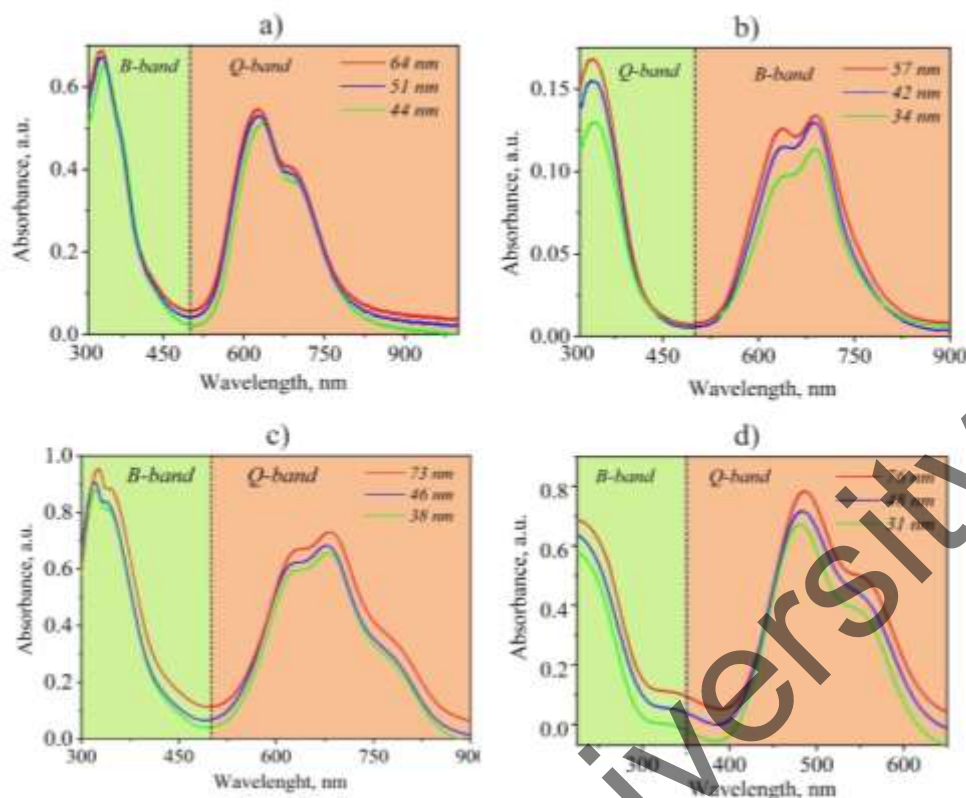


Figure 3. Absorption spectra of H2Pc(a), ZnPc (b), CoPc (c) and CuPc (d) films with different thicknesses.

Table 1

Characteristics of the absorption spectra of the B and Q ranges.

Film, thickness	B-band			Q-band		
	λ_{\max} , nm	D (optical density)	FWHM, nm	λ_{\max} , nm	D (optical density)	FWHM, nm
H2Pc -64nm	$\lambda=333$	D=0.68	110	$\lambda_1=627$ $\lambda_2=694$	D ₁ =0.55 D ₂ =0.41	138
H2Pc -51nm	$\lambda=336$	D=0.67	108	$\lambda_1=628$ $\lambda_2=692$	D ₁ =0.53 D ₂ =0.39	114
H2Pc -44nm	$\lambda=340$	D=0.65	98	$\lambda_1=632$ $\lambda_2=694$	D ₁ =0.51 D ₂ =0.37	96
ZnPc -57nm	$\lambda=339$	D=0.17	94	$\lambda_1=636$ $\lambda_2=688$	D ₁ =0.12 D ₂ =0.13	141
ZnPc -42nm	$\lambda=339$	D=0.16	83	$\lambda_1=638$ $\lambda_2=685$	D ₁ =0.11 D ₂ =0.12	130
ZnPc -34nm	$\lambda=344$	D=0.13	80	$\lambda_1=638$ $\lambda_2=688$	D ₁ =0.09 D ₂ =0.11	117
CoPc -73nm	$\lambda_1=325$ $\lambda_2=348$	D ₁ =0.97 D ₂ =0.88	99	$\lambda_1=630$ $\lambda_2=682$	D ₁ =0.68 D ₂ =0.72	148
CoPc -46nm	$\lambda_1=320$ $\lambda_2=341$	D ₁ =0.90 D ₂ =0.85	91	$\lambda_1=623$ $\lambda_2=678$	D ₁ =0.62 D ₂ =0.69	126
CoPc -38nm	$\lambda_1=323$ $\lambda_2=345$	D ₁ =0.87 D ₂ =0.82	89	$\lambda_1=627$ $\lambda_2=680$	D ₁ =0.59 D ₂ =0.64	108
CuPc -76nm	$\lambda=227$	D=0.69	110	$\lambda_1=485$ $\lambda_2=553$	D ₁ =0.79 D ₂ =0.52	156
CuPc -48nm	$\lambda=227$	D=0.62	96	$\lambda_1=482$ $\lambda_2=558$	D ₁ =0.71 D ₂ =0.48	128
CuPc -31nm	$\lambda=227$	D=0.58	83	$\lambda_1=476$ $\lambda_2=544$	D ₁ =0.67 D ₂ =0.39	102

Along the edge of the absorption spectra, the width of the optical band gap B and Q of the MPc films was estimated (Fig. 4a, b). The estimation of the width of the optical band gap of MPc films was carried out by the TaucPlot method. As can be seen from Figure 4a, the width of the band gap in the B-band was for H2Pc films: $U_p=3.10$, ZnPc: $U_p=2.94$, CoPc: $U_p=2.99$, CuPc: $U_p=3.25$. The width of the optical band gap in the Y-band showed values for H2Pc films: $U_p=1.62$, ZnPc: $U_p=1.66$, CoPc: $U_p=1.62$, CuPc: $U_p=1.65$ (Fig. 4b).

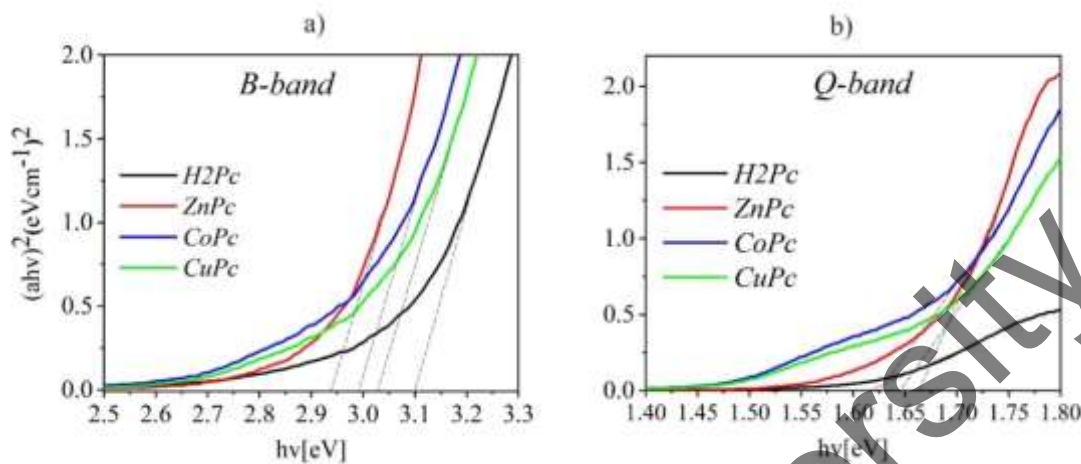


Figure 4. The width of the optical band gap B (a) and Q(b) ranges of phthalocyanine films and its metal complexes

Photoelectrical characterizations

Photovoltaic cell samples consisting of a glass substrate, a transparent conductive layer of polycrystalline FTO (anode), a photoactive MPc layer and an aluminum electrode were prepared for photovoltaic measurements (Fig. 5a). Figure 5b shows a diagram of MPc energy levels. It can be seen from the diagram of energy levels that the values of the band gap width and the position of the energy levels of HOMO and LUMO phthalocyanines differ slightly. The width of the forbidden zone varies in the range from 1.6 to 1.9 eV. The shift of the energy levels of HOMO and LUMO in phthalocyanines depends on the central metal atom.

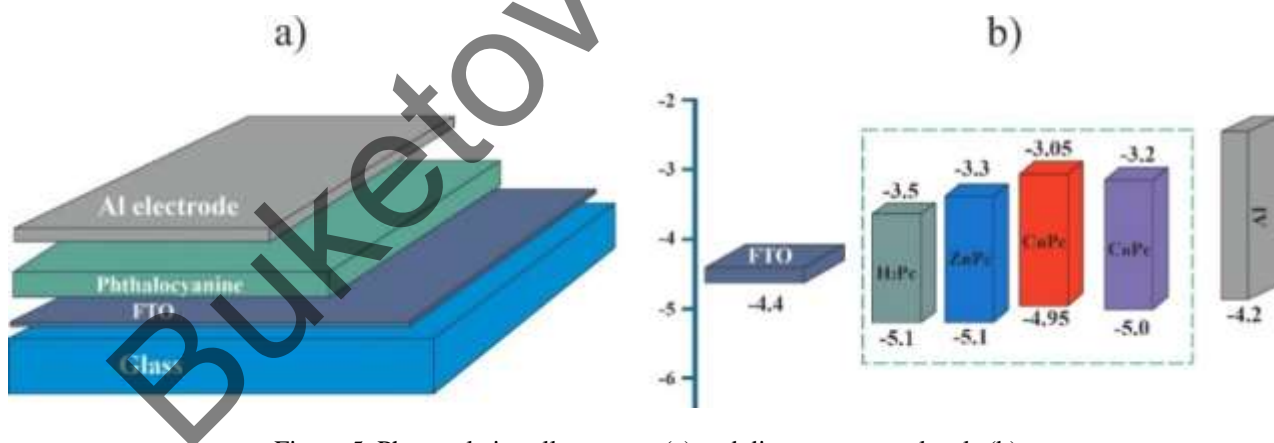


Figure 5. Photovoltaic cell structure (a) and diagram energy levels (b)

Figure 6 shows MPc current-voltage characteristics with different thicknesses. As can be seen from the figure, the current-voltage characteristics are nonlinear. The photosensitive cell based on H2Pc has the lowest values of electrical characteristics. The efficiency of charge carrier generation in a solid H2Pc film is low due to the weak broadening of the absorption bands in the B- and Q-bands (Fig. 3a). This is indicated by the low value of the short-circuit current density $J_{sc} = 3.7 \text{ mA/cm}^2$. The maximum no-load voltage $V_{oc} = 0.48 \text{ V}$ has been achieved for films with a thickness of 51 nm. The ZnPc-based photocell has improved characteristics, such as the short-circuit current density $J_{sc} = 12.3 \text{ mA/cm}^2$ and the maximum value of the no-load voltage $V_{oc} = 0.90 \text{ V}$. These values were obtained for films with a thickness of 34 nm. For CoPc films, the current density value $J_{sc} = 9.2 \text{ mA/cm}^2$ and the maximum voltage $V_{oc} = 1.26 \text{ V}$ were obtained for films with a thickness of 73 nm. The current-voltage characteristics of CuPc-based cells showed a value of J_{sc}

$=8.2 \text{ mA/cm}^2$, while the maximum voltage value at a film thickness of 48 nm is $V_{oc} = 0.90 \text{ V}$. This fact is due to an increase in the optical density and a broadening of the absorption bands in the short-wave and visible spectral regions. The analysis of the current-voltage characteristics in the figures shows that the values of J_{max} and J_{sc} cells based on phthalocyanine with a central metal atom increase by almost 2.5 times, compared to a photocell based on nonmetal phthalocyanine, while the dependence of J_{sc} on the thickness of films in all MPCs does not change. However, there is a dependence of V_{oc} on the thickness of MPC films (Table 2).

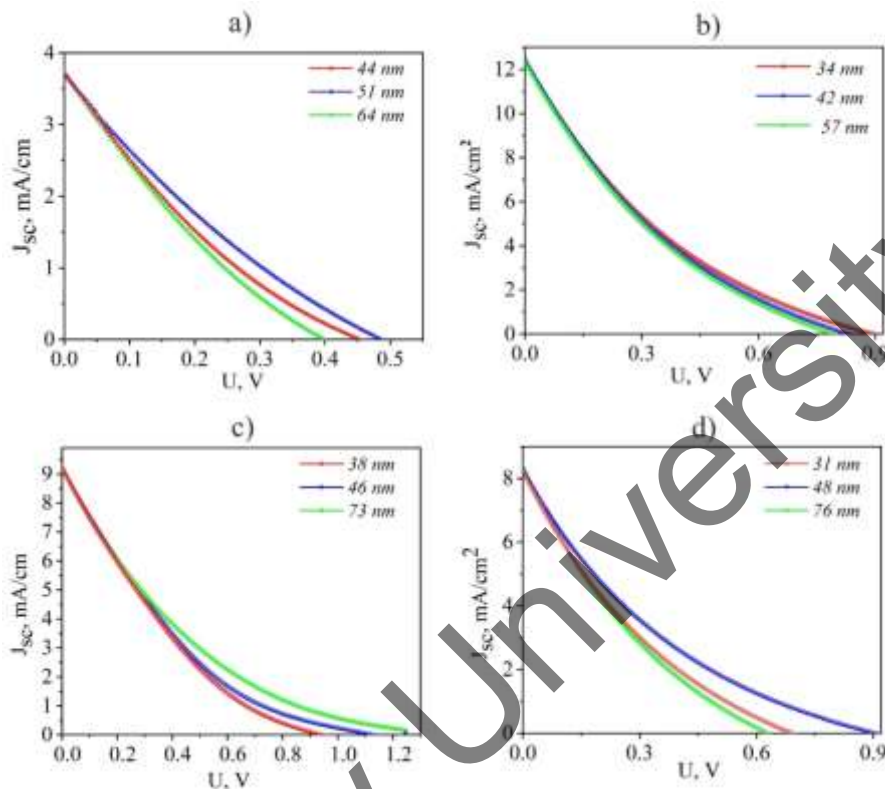


Figure 6. Current-voltage characteristics of H2Pc (a), ZnPc (b), CoPc (c) and CuPc (d) with different thicknesses

Table 2

Photovoltaic characteristics of phthalocyanine and its metal complexes

Film, thickness	V_{oc} (V)	J_{sc} (mA/cm ²)	V_{max} (V)	J_{max} (mA/cm ²)	FF
H2Pc -64 nm	0.39	3.7	0.20	1.3	0.15
H2Pc -51 nm	0.48	3.7	0.23	1.5	0.19
H2Pc -44 nm	0.45	3.7	0.21	1.4	0.19
ZnPc -57 nm	0.78	12.3	0.32	4.6	0.15
ZnPc -42 nm	0.83	12.3	0.33	4.7	0.16
ZnPc -34 nm	0.90	12.3	0.34	4.8	0.14
CoPc -73 nm	1.26	9.2	0.44	3.1	0.11
CoPc -46 nm	1.11	9.2	0.42	3.2	0.13
CoPc -38 nm	0.94	9.2	0.41	3.4	0.16
CuPc -76 nm	0.69	8.2	0.30	3.2	0.16
CuPc -48 nm	0.90	8.2	0.33	2.9	0.13
CuPc -31 nm	0.62	8.2	0.29	2.8	0.14

Figure 7 shows the impedance spectra of cells of the FTO/MPC/Al structure with different thickness of the MPC layer. As can be seen from the figure, the cell based on H2Pc has the highest values of electrophysical characteristics. The resistance of the H2Pc film with a layer thickness of 64 nm is 5697.8 ohms, the recombination resistance is 1.57-105 ohms. When the layer thickness decreases to 51 nm, a decrease in the resistance value is observed, and at a thickness of 44 nm, the film has minimal electrophysical characteristics for this type of phthalocyanine, $R_w = 4389.3 \text{ ohms}$, $R_{rec} = 1.39-105 \text{ ohms}$. The conditional mobility of charge

carriers increases with a decrease in the thickness of H₂Pc. The data of the impedance spectra are consistent with the data of the current-voltage characteristics and the characteristics of the optical absorption spectrum. For CoPc films, a device with a layer thickness of 46 nm has optimal characteristics. $R_{rec} = 1.34 \cdot 10^6$ ohms, $R_w = 368.6$ ohms. For a 76 nm thick CuPc film, the resistance parameters are equal to $R_{rec} = 0.42 \cdot 10^5$ ohms and $R_w = 4012.7$ ohms. However, the ZnPc-based device has the best characteristics. For a ZnPc film with a thickness of 34 nm, the minimum resistance value $R_w = 173.4$ ohms has been achieved, at this thickness the recombination resistance is $1.37 \cdot 10^6$ ohms, the effective lifetime of charge carriers is 81 ms and is the highest among all the studied samples, D_{eff} is $34.2 \cdot 10^{-8} \text{ cm}^2/\text{s}$, and the conditional mobility of charge carriers it is maximum and equal to $1.4 \cdot 10^5 \text{ cm}^2 \text{V}^{-1} \text{ s}^{-1}$. The dynamics of changes in electrophysical parameters is caused by changes in the values of optical density, broadening of absorption bands in the short-wave and visible spectral regions and is consistent with the data of the current-voltage characteristics (Table 3).

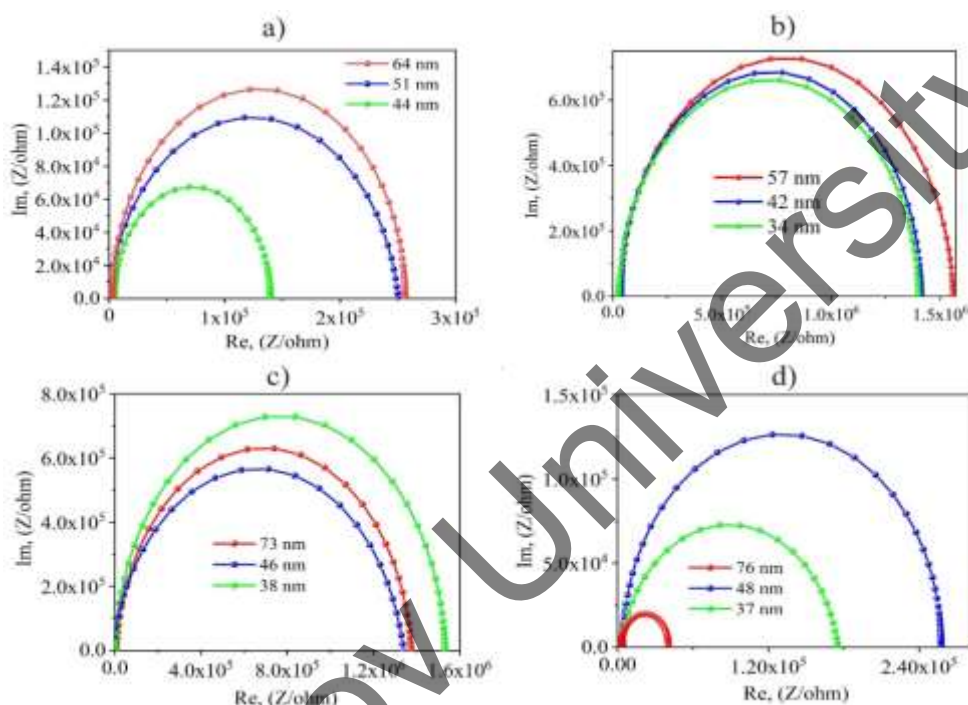


Figure 7. Impedance hodographs in Nyquist coordinates for sprayed films H₂Pc (a), ZnPc (b), CoPc (c) and CuPc (d) with different thicknesses

Table 3

Charge transfer parameters of phthalocyanine films and its metal complexes with different layer thicknesses

Film, thickness	D_{eff} , ($\text{cm}^2 \cdot \text{s}^{-1}$)	k_{eff} , (s^{-1})	τ_{eff} , (ms)	R_{rec} , (ohm)	R_w , (ohm)	μ , ($\text{cm}^2 \text{V}^{-1} \text{ s}^{-1}$)
H ₂ Pc -64 nm	$28.7 \cdot 10^{-5}$	25.5	39	$1.57 \cdot 10^5$	5697.8	$0.1 \cdot 10^{-5}$
H ₂ Pc -51 nm	$20.9 \cdot 10^{-5}$	26.4	37	$1.46 \cdot 10^5$	4789.7	$0.9 \cdot 10^{-5}$
H ₂ Pc -44 nm	$17.2 \cdot 10^{-5}$	28.1	35	$1.39 \cdot 10^5$	4389.3	$0.7 \cdot 10^{-5}$
ZnPc -57 nm	$23.8 \cdot 10^{-7}$	15.5	64	$1.5 \cdot 10^6$	316.3	$1.3 \cdot 10^{-5}$
ZnPc -42 nm	$17.5 \cdot 10^{-7}$	14.2	70	$1.42 \cdot 10^6$	203.2	$1.1 \cdot 10^{-5}$
ZnPc -34 nm	$34.2 \cdot 10^{-8}$	12.3	81	$1.37 \cdot 10^6$	173.4	$1.4 \cdot 10^{-5}$
CoPc -73nm	$29.7 \cdot 10^{-7}$	15.3	65	$1.38 \cdot 10^6$	378.1	$0.2 \cdot 10^{-5}$
CoPc -46 nm	$10.5 \cdot 10^{-7}$	13.7	72	$1.34 \cdot 10^6$	368.6	$0.8 \cdot 10^{-5}$
CoPc -38 nm	$79 \cdot 10^{-6}$	13.3	75	$1.52 \cdot 10^6$	370.1	$0.5 \cdot 10^{-5}$
CuPc -76 nm	$15.5 \cdot 10^{-5}$	25.7	38	$0.42 \cdot 10^5$	4012.7	$0.2 \cdot 10^{-5}$
CuPc -48 nm	$21.1 \cdot 10^{-5}$	17.5	57	$2.11 \cdot 10^5$	4025.7	$0.1 \cdot 10^{-5}$
CuPc -37 nm	$2.5 \cdot 10^{-5}$	4.3	23	$1.73 \cdot 10^5$	4063.7	$0.6 \cdot 10^{-5}$

Conclusions

As a result of vacuum spraying under the same conditions, MPc films were obtained, the SEM image of the transverse cleavage of the films showed that the thickness of the vacuum-deposited films is approximately the same. The measured absorption spectra showed that the maximum of the B-band for H2Pc, ZnPc and CoPc films is located at a wavelength of $\lambda = 340$ nm. In the Q-band, two maxima are observed at wavelengths $\lambda = 630$ nm and $\lambda = 690$ nm. For CuPc films, a hypsochromic shift of absorption maxima $\lambda = 227$ nm is observed in the B-band region, as well as a shift of maxima in the Q-range $\lambda = 485$ nm and $\lambda = 553$ nm. Based on the results obtained, it was found that the value of the half-width of the absorption spectra decreases with a decrease in the thickness of MPc films. Along the edge of the absorption spectra, the width of the optical band gap of the B and Q ranges of MPc films is determined. The photovoltaic properties of FTO/MPc/Al cells with different MPc thicknesses were measured. The measured of the current-voltage shows that the values of J_{\max} and J_{sc} cells with metal phthalocyanine increase almost 2.5 times compared to the H2Pc-based photocell, while the dependence of J_{sc} on the thickness of films in all MPCs does not change, however, there is a dependence of V_{oc} on the thickness of MPc films. The ZnPc-based photocell is characterized by improved characteristics of the short-circuit current density $J_{\text{sc}} = 12.3$ mA/cm² and the maximum no-load voltage $V_{\text{oc}} = 0.90$ V, which are obtained for films with a thickness of 34 nm. The measurement of the impedance spectra of the cells confirms that the effective lifetime of the charge carriers for the ZnPc film was 81 ms and is the highest indicator among all the samples studied. The dynamics of the electrophysical parameters for all MPc films is due to changes in the optical density values, broadening of the absorption bands in the short-wave and visible spectral regions and is consistent with the data of the current-voltage characteristic.

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Фталоцианин мен оның металл кешендері қабыршақтары қалыңдығының оның оптикалық және электрлік қасиеттеріне әсері

Мақалада әртүрлі қалыңдықтағы фталоцианин және оның металл кешендері қабықшаларының құрылымдық, оптикалық және электрфизикалық сипаттамаларының зерттеу нәтижелері ұсынылған. Вакуумда термиялық булану арқылы өткізгіш FTO бетінде фталоцианин және оның металл кешендерінің қабықшасы алынды. В және Q жолақтарында байқалатын кеңею және жұтылу спектрінің максимумдарының гипохромдық ығысуы орталық атоммен байланысты екені көрсетілген. МРС қабықшаларының қалыңдығы азайған сайын жұтылу спектрінің жарты ені азаяды. FTO/MPC/Al ұяшығында фталоцианин қабықшаларының қалыңдығы заряд тасымалдаушылар генерациясының және тасымалдануының тиімділігіне әсері зерттелді. Металлсыз фталоцианин негізіндегі фотоэлементпен салыстырғанда ток кернеуінің сипаттамасы шамамен 2,5 есе өсетіні көрсетілген. Алынған импеданс нәтижелерінен фталоцианиндердің электрфизикалық параметрлері, оптикалық тығыздық мәндері мен спектрдің қысқа толқынды және көрінетін аймақтарындағы жұтылу жолақтарының кеңеюіне байланысты екені анықталды, бұл вольт-амперлік сипаттамасына талдау деректерімен сәйкес келеді.

Кілт сөздер: фталоцианин, металлдар фталоцианині, қатты қабықшалар, жұтылу спектрлері, импедансметр, вольт-амперлік сипаттамалар.

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Влияние толщины пленок фталоцианина и его металлокомплексов на оптические и электрические свойства

В статье представлены результаты исследования структурных, оптических и электрофизических характеристик пленок фталоцианина и его металлокомплексов различной толщины. На проводящей поверхности FTO методом термического испарения в вакууме были получены пленки фталоцианина и его металлокомплексов. Показано, что наблюдаемое уширение полос В и Q и гипохромный сдвиг максимумов спектра поглощения связаны с центральным атомом. С уменьшением толщины пленок МРС происходит снижение значения полуширины спектра поглощения. Изучено влияние толщины пленок фталоцианина на эффективность генерации и транспорта носителей заряда в ячейке FTO/MPC/Al. Показано, что ВАХ увеличивается почти в 2,5 раза по сравнению с фотоэлементом на основе безметаллового фталоцианина. Из полученных данных измерения импеданса установлено, что электрофизические параметры фталоцианинов зависят от значений оптической плотности, уширения полос поглощения в коротковолновой и видимой области спектра, что согласуется с данными анализа вольт-амперной характеристики.

Ключевые слова: фталоцианин, фталоцианины металлов, твердые пленки, спектры поглощения, импедансметр, вольт-амперные характеристики.