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INFLUENCE OF FULLERENOL DOPANT ON LIGHT ABSORPTION BY POLYVINYL ALCOHOL-BASED COMPOSITE FILMS

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Abstract. The study of the effect of fullerenol ($C_{60}(OH)_{44}$) dopant of different concentrations on the optical properties of polyvinyl alcohol (PVA) films has been presented. The addition of even a small concentration of fullerenol was found to lead to significant light absorption in the UV spectral range. As the concentration of fullerenol increased from 1 to 10 wt.%, the light transmission edge of the PVA : $C_{60}(OH)_{44}$ composite films shifted from 230 to 440 nm. The dependences of the extinction coefficient (EC) on the fullerenol concentration in the films were calculated for several wavelengths; these curves decreased with $C_{60}(OH)_{44}$ concentration increasing from 1 to 3 wt.%, but then EC values stopped decreasing. The light absorption in the studied samples was shown to cause by the fullerenol molecules' excitation during indirect HOMO → LUMO transitions. The E_g value estimated from their spectra, was 2.2±0.3 eV and depended little on the fullerenol concentration.

Keywords: polyvinyl alcohol, fullerenol, extinction coefficient, optical spectrum, energy gap

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ВЛИЯНИЕ ДОБАВОК ФУЛЛЕРЕНОЛА НА ПОГЛОЩЕНИЕ СВЕТА КОМПОЗИТНЫМИ ПЛЕНКАМИ НА ОСНОВЕ ПОЛИВИНИЛОВОГО СПИРТА

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Аннотация. Представлено исследование влияния добавок фуллеренола $C_{60}(OH)_{44}$ различной концентрации на оптические свойства пленок поливинилового спирта (ПВС). Установлено, что добавка даже небольшой концентрации фуллеренола приводит к значительному поглощению света в УФ-области спектра. По мере увеличения концентрации фуллеренола с 1 до 10 масс.% граница пропускания света композитными пленками ПВС:$C_{60}(OH)_{44}$ смещается от 230 к 440 нм. Для ряда длин волн рассчитана зависимость коэффициента экстинкции (КЭ) от концентрации фуллеренола в пленках; при повышении концентрации $C_{60}(OH)_{44}$ от 1 до 3 масс.% КЭ снижается, но далее его значение стабилизируется. Показано, что поглощение света в изученных объектах связано с возбуждением молекул фуллеренола при непрямых переходах HOMO → LUMO. Согласно оценке, значение разрыва HOMO–LUMO составляет 0,3±2,2 эВ и мало зависит от концентрации фуллеренола.

Ключевые слова: поливиниловый спирт, фуллеренол, коэффициент экстинкции, оптический спектр, энергетический зазор

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Introduction

Polymer composite materials are widely used in electronics as structural and functional materials. Water-soluble polymers, in particular polyvinyl alcohol (PVA), are of special interest. PVA is characterized by low cost, environmental safety, transparency, good dielectric characteristics and excellent film-forming properties. PVA is a thermoplastic amorphous crystalline polymer where regions with a high degree of ordering and dense packing of molecules (crystallites) are randomly oriented within amorphous regions [1]. Composite materials based on PVA are intended for manufacturing of organic field-effect transistors [2], thin-film polarizers [3], polymer coatings [4], optoelectronic devices [5].

Introducing even a small amount of dopant into PVA can significantly modify its physical properties. Additives that do not impair the water solubility of PVA, such as fullerenols, i.e., water-soluble polyhydroxylated derivatives of fullerene, are also of considerable interest [6–8].

Due to the presence of hydroxyl groups in fullerenol and PVA molecules, hydrogen bonds arise between them [6]. It was found in [8] for concentrations of fullerenol aqueous solutions close to its maximum solubility in water (58.9 mg/mL [9]) that the compound tends to form clusters in the PVA matrix, so the composite material consequently exhibits properties inherent to pure fullerenol. Determining the concentration of fullerenol at which such properties begin to appear is an important objective both in fundamental and applied terms. It should be noted here that the tendency to form molecular aggregates is observed not only in PVA : $C_{60}(OH)_{44}$ composite material, but also in fullerene C_{60} embedded in the polymethylmethacrylate (PMMA) matrix. The aggregates formed in PMMA have a certain degree of ordering, but it is far from that of the crystalline phase [10]. According to [10], such phenomena are associated with segregation of fullerene molecules during film formation as the solvent evaporates.

The goal of this study was to analyze the effect from introducing an aqueous solution of fullerenol $C_{60}(OH)_{44}$ in various concentrations on the optical properties of PVA films.

Experimental procedure

The experimental samples were films made of the 16/1 grade PVA (GOST 10779-78). A 5% aqueous solution of PVA was prepared by dissolving PVA in distilled water at a temperature of 85 °C. Aqueous solutions of fullerenol with different concentrations were added to the resulting PVA solution. Fullerenol synthesized by the technique described in [11] was used to prepare the composite films. The composite films were prepared by solution casting of the mixture onto a glass surface (a Petri dish 9 cm in diameter), followed by air drying at room temperature. Seven samples of PVA : $C_{60}(OH)_{44}$ composite film with different concentrations of fullerenol (see Table) and one control sample of PVA film without fullerenol were prepared. After preparation, all films were first kept in a thermostat at 120 °C for 2 hours and subsequently slowly cooled to room temperature for 8 hours. The entire heat treatment process was carried out in vacuum at a pressure of about 10 Pa.

Table

Characteristics of prepared PVA+fullerenol film samples

Thickness, μm	79±1	54±1	73±3	59±3	76±5	74±1	85±2	68±2
Fullerenol concentration, wt.%	0.0	1.0	1.5	2.0	2.5	3.0	5.0	10.0

The optical transmission spectra of the films in the wavelength range of 190–1100 nm were measured using an SF-56 spectrophotometer.

Experimental results and discussion

Fig. 1 shows the optical transmission spectra $T(\lambda)$ (T is the transmittance, λ is the light wavelength) of the studied films. Evidently, the PVA film without added fullerenol is characterized by transparency not only in the visible but also in the near-UV spectral range. The band observed in PVA at a wavelength of 280 nm can be attributed to $(-CH{=}CH-)_2CO-$ structures formed in the polymer [12].

Adding even a small amount of fullerenol to PVA causes the film to turn yellow-brown, which is accompanied by a significant increase in light absorption in the UV spectral range. No characteristic absorption bands are observed on the $T(\lambda)$ dependences considered. Notably, the yellow coloring of the polymer film is accompanied by the appearance of absorption in the blue spectral range. As fullerenol concentration in the composite film increases, its color becomes more saturated, with predominantly brown hues, while the transmission edge progressively shifts towards the longer-wavelength region of the spectrum.

Comparing the attenuation of light by PVA : $C_{60}(OH)_{44}$ composite films of various thicknesses, let us consider the effect of fullerenol concentration on their extinction coefficient (EC), defining it as

$$\varepsilon(\lambda) = -\lg[T(\lambda)]/lC, \tag{1}$$

where l, μm, is the film thickness, C, wt.%, is the fullerenol concentration.

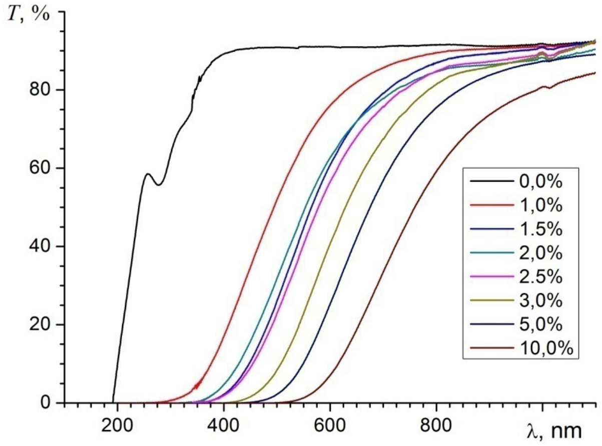

Fig. 1. Transmission spectra for samples of pure PVA films and PVA : $C_{60}(OH)_{44}$ composites at different fullerenol concentrations

The ε(C) dependences are shown in Fig. 2. They were calculated using Eq. (1) and the T(λ) dependences for different wavelengths. Apparently, these curves are smooth at wavelengths of 700 and 800 nm, and the ε values found at lower λ are slightly higher than at longer wavelengths. It should be noted that the EC decreased with an increase in $C_{60}(OH)_{44}$ concentration from 1 to 3% and its value stabilized with a higher fullerenol concentration. The EC values determined at wavelengths of 480 and 580 nm are characterized by both significantly higher magnitudes and considerable scatter in the fullerenol concentration range of 2–3 wt.%.

We believe that the observed feature of the ε(C) curve at a fullerenol concentration C = 2–3% is caused by concentration-dependent aggregation accompanied by the formation of clusters of fullerenol molecules. At the initial stage of cluster nucleation, their sizes are rather small. As the fullerenol concentration in the solution increases, they transform randomly; for this reason, the EC, depending on the size of the cluster, also varies randomly. Once a certain concentration of fullerenol molecules is reached, an average cluster size can be defined that no longer depends (or depends only weakly) on the further increases in concentration.

No visual opacification of the films was observed with increasing dopant concentration. We can therefore conclude that the decrease in the intensity of light transmitted through the film (and, consequently, in the extinction coefficient ε) is due to absorption of light energy rather than its scattering in the composite film.

The following relationship is often used to determine the energy gap between the levels of the highest occupied molecular orbital (HOMO) and the lowest unoccupied molecular orbital

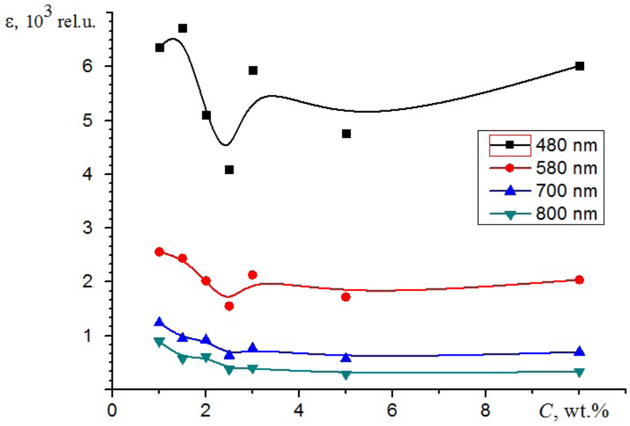

Fig. 2. Dependences of extinction coefficient for PVA : $C_{60}(OH)_{44}$ composite films on fullerenol concentration at different light wavelengths λ

(LUMO) in organic structures (known as the HOMO–LUMO gap), in particular in polymer composite materials [5, 13–15]:

$$(h\nu/l)\cdot\ln(1/T) = B(h\nu - E_g)^p, \qquad (2)$$

where h is the Planck constant; ν is the light wave frequency; B is a constant independent of the photon energy $h\nu$; E_g is the HOMO–LUMO energy gap, $E_g = E_{\mathrm{LUMO}} - E_{\mathrm{HOMO}}$ (E_{HOMO}, E_{LUMO} are the energies of the HOMO and LUMO levels, respectively); p is a constant characterizing the behavior of the HOMO → LUMO transition (i.e., the fundamental absorption).

Its value is $p = 1/2$ for an allowed direct transition, and $p = 2$ for an indirect transition associated with phonon emission or absorption [15].

Eq. (2) allows to determine the energy gap based on the data for the transmittance $T(\lambda)$. It implies that the E_g value can be found by extrapolating the linear part of the graph

$$[(h\nu/l)\cdot\ln(1/T)]^{1/p} \qquad (3)$$

to the zero value.

Eq. (2) was used in [5] to calculate E_g in PVA films modified with extract from black tea leaves. The E_g obtained in [5] for $p = 1/2$ (i.e., assuming direct transitions) for pure PVA was 5.38 eV. If 10 ml of tea extract was added to an aqueous solution containing 1 g of PVA, E_g decreased to 3.1 eV and further decreased with increasing volume of the added extract. The E_g value for added 60 ml of the extract was 1.85 eV. According to [5], the tea extract solution contains a sufficient number of conjugated double bonds, hydroxyl, carboxyl groups, polyphenols and polyphenols conjugates, promoting the formation of complexes with polar groups of PVA, which leads to a decrease in E_g for PVA molecules.

The trend towards a decrease in E_g if sodium iodide (NaI) was added to PVA was also established in [15]. Estimates of E_g using Eq. (2) in our study were carried out assuming both direct and indirect transitions were possible. The E_g value for pure PVA determined for $p_1 = 2/1$ was 5.4 eV, and 4.9 eV for $p_2 = 2$. The values of 5.10 and 4.65 eV, respectively, were obtained for the PVA : NaI (90:10) composite films. With an increase in NaI concentration in the composite (70:30), the E_g value in the PVA : NaI composite decreased to 4.90 eV for direct transitions with an increase, and to 4.40 eV for indirect ones.

Thus, an increase in the dopant concentration in PVA is always accompanied by a decrease in the E_g value determined by Eq. (2).

In this work, we calculated the E_g values for both direct and indirect transitions based on the optical transmission spectra of pure PVA and PVA : $C_{60}(OH)_{44}$ composite films with different fullerenol concentrations.

As an example, Fig. 3 shows the calculated dependences for the quantities $[(h\nu/l)\cdot\ln(1/T)]^2$ and $[(h\nu/l)\cdot\ln(1/T)]^{1/2}$ (see Eq. (3)) on photon energy $h\nu$ (Fig. 3, *a* and *b*, respectively) for pure PVA, from which E_g can be determined for this polymer. The value of E_g obtained for direct transitions was 5.7±0.2 eV (here and below, the uncertainty in the determination of E_g arises from the chosen number of points used in the linear extrapolation), and 5.6±0.25 eV for indirect transitions.

Within the experimental error, the E_g values are virtually identical and thus cannot be used to determine the dominant type of transition. On the other hand, the obtained E_g values are in good agreement with the data known from the literature on the HOMO–LUMO gap in PVA [5, 15].

The E_g values in fullerenol-doped PVA films were found by a similar technique but the error in determining E_g increased to ± 0.3 eV due to the limited number of points at which linear approximation was performed.

The dependence of E_g on the fullerenol concentration in PVA : $C_{60}(OH)_{44}$ composite films for both direct and indirect HOMO → LUMO transitions is plotted in Fig. 4.

It can be seen that the E_g values determined for two types of transitions differ significantly in magnitude, which is especially noticeable at dopant concentrations below 5%. However, regardless of the assumed type of transition, the obtained E_g values for the composite material are significantly lower than those found for pure PVA. While the E_g values found for direct transitions exhibit a decreasing trend with increasing fullerenol concentration, this behavior is not observed for indirect transitions and the E_g values are approximately the same for all dopant concentrations.

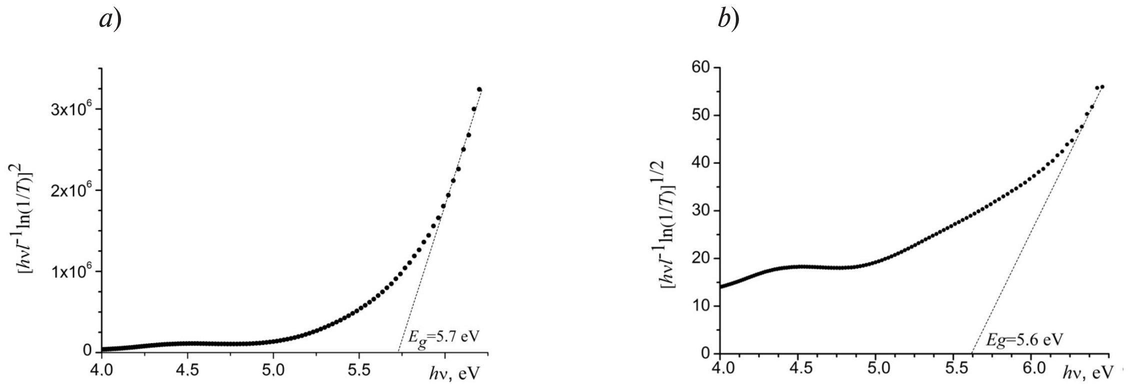

Fig. 3. Calculated dependences for Eq. (3) at $p_1 = 1/2$ (*a*) and $p_2 = 2$ (*b*) for pure PVA film

The dotted tangent lines illustrate the method used to determine the E_g

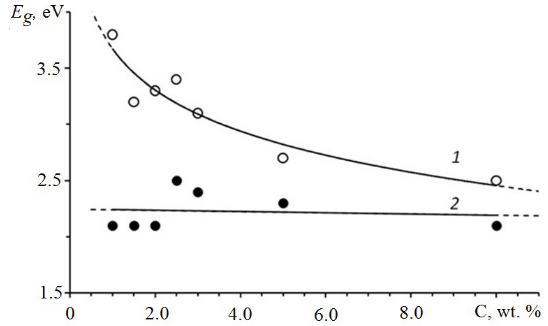

Fig. 4. Calculated dependences of HOMO—LUMO gap in PVA composite films on fullerenol concentration for direct (*1*) and indirect (*2*) HOMO → LUMO transitions

The data were obtained based on the experiments on optical transmission of samples

Before further analyzing the results in Fig. 4, we should note that PVA is a molecular solid formed by hydrogen (weak) intermolecular bonds, therefore, PVA macromolecules remain distinct entities in condensed phase. PVA : $C_{60}(OH)_{44}$ composite films were prepared by mixing aqueous solutions of PVA and fullerenol. We did not use any special methods to initiate the crosslinking of macromolecules, so strong covalent bonds could not form between the PVA and fullerenol molecules [6]. In our case, hydrogen bonds dominate between the molecules, which are much weaker than covalent bonds and cannot significantly transform the electronic structure of both PVA and fullerenol molecules. Consequently, the position of the HOMO and LUMO energy levels determining the E_g value remains unchanged. In any case, the decrease in E_g of PVA molecules during their interaction with fullerenol molecules should not be as substantial as the data in Fig. 4 indicate for direct HOMO → LUMO transitions.

In our opinion, the absorption of light by PVA : $C_{60}(OH)_{44}$ composite films is not associated with excitation of PVA molecules whose electronic structure was modified by interaction with the dopant. Instead, it is caused by indirect HOMO → LUMO transitions in fullerenol molecules, for which the E_g value is approximately 2.2 eV and virtually does not depend on the dopant concentration.

Unfortunately, there are no data in the literature on the electronic structure of fullerenol $C_{60}(OH)_{44}$, but the results of theoretical calculations of the electronic spectrum and estimates of the E_g value are known for fullerenol with a smaller number of hydroxyl groups ($C_{60}(OH)_{26}$), for which the E_g was determined to be about 1.95 eV [16]. For an isolated molecule of fullerene C_{60}, the band gap is 1.9 eV [17], which, within the measurement error, coincides with the E_g value we determined in this study.

Conclusion

This paper reports on measurements of light absorption by PVA films doped with fullerenol $C_{60}(OH)_{44}$ molecules. We established that adding even a small concentration of fullerenol leads to significant absorption of light in the UV spectral range. As the concentration of fullerenol increases from 1 to 10 wt.%, the light transmission edge of PVA : $C_{60}(OH)_{44}$ composite films shifts from 230 to 440 nm. The extinction coefficient decreases with an increase in fullerenol concentration from 1 to 3%, remaining almost unchanged at high dopant concentrations. Presumably, fullerenol molecules form clusters, such that the HOMO–LUMO gap (E_g) of fullerenol molecules in clusters remains virtually unchanged starting from a certain cluster size (increasing with increasing dopant concentration), becoming close to the corresponding values in condensed state. In other words, the value of the extinction coefficient stabilizes and stops increasing with increasing dopant concentration, despite the continued increase in cluster sizes.

The absorption of light in the composite films is most likely due to excitation of fullerenol molecules during indirect HOMO → LUMO transitions. The value of the HOMO–LUMO gap E_g, found from the measured transmission spectra of the film samples, lies within 2.2 ± 0.3 eV according to our estimates, and obviously does not depend on the fullerenol concentration in the composite film.

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