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Langmuir and Langmuir–Schaefer Films of Poly(3-hexylthiophene) with Gold Nanoparticles and Gold Nanoparticles Capped with 1-Octadecanethiol

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ABSTRACT: This paper reports on the production of Langmuir films of regioregular poly(3-hexylthiophene) (P3HT) and their characterization by surface pressure isotherms and UV–vis in situ spectroscopy aiming at the best spreading conditions. Hybrid films containing the polymer and gold nanoparticles were obtained through the use of the optimized parameters for neat P3HT. Two types of hybrid films with P3HT and gold nanoparticles were studied. In the first type, gold nanoparticles in an aqueous solution were used in a subphase of a Langmuir trough and the polymer spread on the air/water interface. In the second type, 1-octadecanethiol capped gold nanoparticles (AuNpOctathiol) were used in a mixture with P3HT in chloroform and spread on the air–water interface. The Langmuir films were transferred to several solid substrates by the Langmuir–Schaefer (LS) method. The surface morphology was characterized by Scanning Electron Microscopy with Field Emission Guns (SEM-FEG), and the presence of gold nanoparticles in the films was confirmed. The growth of the layers in the films was monitored by transmission UV–vis spectroscopy, and the existence of optical anisotropy was investigated by polarized UV–vis spectroscopy. The results imply that the polymer backbone adopts a preferential orientation during the compression, which has a strong component parallel to the trough barrier, and is influenced by the presence of the nanoparticles in the films. The photostability of the films was studied by their exposition to a white light, and the hybrid films formed with P3HT and AuNpOctathiol showed an increase in the photostability, in comparison to the neat P3HT. Such an increase indicates that somehow the AuNpOctathiol molecules provide some protection against the photodegradation of P3HT.

INTRODUCTION

Conjugated polymers are materials that exhibit insulating, semiconducting, or conducting properties, depending on their structure and doping levels. The conductivity characteristics of such compounds are related to the presence of delocalized π electrons along the polymer chain, which enables numerous applications to areas dedicated only to other materials, as metals. Polythiophene derivatives are conjugated polymers particularly interesting because they have high chemical and thermal stability and ease of functionalization by the insertion of different groups in the thiophenic ring at positions 3 and/or 4. Substituted polythiophenes are those that combine the properties of the conjugated chain with the properties of the substituents.

Conjugated polymers, especially polythiophenes, have been largely studied due to their specific properties, such as conductivity, luminescence, and chromism, which enable several applications, as photovoltaic devices, solar cells, and sensors of various types. However, these polymers are susceptible to photodegradation by the simultaneous action of light and oxygen. The behavior of the photodegradation of polythiophenes is similar to that of poly(p-phenylenevinylene) (PPVs). In the initial stages, the intensity of the visible light absorption decreases and color changes can be observed by the naked eye. In the following stages, the wavelength of maximum absorption decreases until the sample has become colorless. This is a serious drawback for technological applications, which still needs to be properly addressed.

The incorporation of metal nanoparticles in these polymers is an alternative to improve the properties of polythiophenes. Nanoparticles, specially gold and silver, have been used for the optical amplification and electrical signals of conjugated polymer-based devices.

In general, metal nanoparticles may have different sizes and shapes, such as spheres, rods, and pyramids, which take to different properties, and may be capped in a wide variety of substances, such as alkanethiols, amines, and various polymers, including conjugated polymers. Spherical nanoparticles are found in most reports in the literature due to their ease of synthesis and can be obtained in either homogeneous aqueous phases (using reducers as citrate and borohydride or amines) or aqueous/organic phases.

The hybrid materials from polythiophene derivatives and gold nanoparticles, synthesized in our study, were used for the...
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EXPERIMENTAL SECTION

Synthesis of Gold Nanoparticles and Gold Nanoparticles Capped with 1-Octadecanethiol. Gold nanoparticles were prepared by sodium citrate reduction in an aqueous solution by the Turkevich method.35 35 μL of an aqueous solution of gold chloride (solution commercially obtained from Sigma-Aldrich — gold (III) chloride solution, 30 wt %, in diluted HCl 99.99%) was added to 100 mL of water in a 250 mL reaction flask attached to a reflux condenser. The solution was heated and stirred until boiling, and 25 mL of 0.1 M sodium citrate solution was added. After 10 min, the heating was interrupted, and the suspension formed was left to cool to room temperature so that an AuNp suspension could be obtained.

The synthesis of gold nanoparticles capped with 1-octadecanethiol was based on the work of C. S. Weisbecker and collaborators.36 One mL of the AuNp suspension, prepared by the Turkevich method, and 1 mL of aqueous NaOH 10−3 M were mixed. The mixture was centrifuged at 10,000 rpm for 20 min, and the supernatant was removed. The precipitate was washed with 1 mL of the NaOH solution, stirred, and centrifuged again. This process was repeated three times. One mL of a 0.2 mM solution of 1-octadecanethiol (Sigma-Aldrich) in ethanol was then added to the precipitate. The mixture was stirred and centrifuged under the same conditions, and the supernatant was discarded. The precipitate was washed with ethanol three times, centrifuged, and dried in a vacuum oven at 40 °C for 24 h. The product formed could be solubilized in chloroform (AuNpOctathiol).

All suspensions of nanoparticles were characterized by absorption and emission in the UV−vis and Field Emission Scanning Electron Microscopy (SEM-FEG).

Preparation of Langmuir and Langmuir−Schaefer Films (LS). The Langmuir and Langmuir−Schaefer films (LS) were prepared in a KSV 5000 trough in a 10,000 class clean room at constant internal temperature of 22 °C. AuNp in aqueous solution was used as a subphase of the Langmuir trough, and P3HT was spread on the surface (chloroform solution of 0.1 g/L). The AuNp concentration in the subphase was varied, as described in Table 1, and the volume of the spreading P3HT solution remained constant.

### Table 1. AuNp Concentration Used in P3HT Langmuir Films

<table>
<thead>
<tr>
<th>isotherm</th>
<th>concn of AuNp</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>0.05 g/L</td>
</tr>
<tr>
<td>2</td>
<td>0.10 g/L</td>
</tr>
<tr>
<td>3</td>
<td>1.00 g/L</td>
</tr>
<tr>
<td>4</td>
<td>2.00 g/L</td>
</tr>
</tbody>
</table>

*In all measurements, the amount of spreading P3HT was 350 μL.*

Another type of hybrid film was obtained by chloroform solutions of P3HT and AuNpOctathiol mixtures prepared from neat 0.1 g/L solutions of two compounds. The mixtures were prepared with different ratios between the components but keeping the final concentration of 0.1 g/L, as shown in Table 2.

### Table 2. Ratios of P3HT and AuNpOctathiol, in Mass %, of the Mixtures in Chloroform

<table>
<thead>
<tr>
<th>mixtures (0.1 g/L)</th>
<th>[P3HT] (g/L)</th>
<th>[AuNpOctathiol] (g/L)</th>
<th>MM average (g/mol)</th>
</tr>
</thead>
<tbody>
<tr>
<td>P3HT75%/AuNpOctathiol(25%)</td>
<td>0.075</td>
<td>0.025</td>
<td>197.70</td>
</tr>
<tr>
<td>P3HT50%/AuNpOctathiol(50%)</td>
<td>0.050</td>
<td>0.050</td>
<td>245.25</td>
</tr>
<tr>
<td>P3HT25%/AuNpOctathiol(75%)</td>
<td>0.025</td>
<td>0.075</td>
<td>324.50</td>
</tr>
<tr>
<td>P3HT10%/AuNpOctathiol(90%)</td>
<td>0.010</td>
<td>0.090</td>
<td>403.75</td>
</tr>
<tr>
<td>P3HT4%/AuNpOctathiol(96%)</td>
<td>0.004</td>
<td>0.096</td>
<td>451.30</td>
</tr>
</tbody>
</table>

The calculation of the molar ratio of the fractions was based on the molar mass of the repeating unit of the polymer (166 g/mol) and the molecular weight of AuNpOctathiol (considering one molecule of 1-Octadecanethiol per gold molecule, 483 g/mol). The spreading volume was 350 μL for all samples.

The Langmuir films were characterized by surface pressure isotherms, in situ absorption spectroscopy in the UV−vis, in situ fluorescence microscopy, and Polarization Modulation Infrared Reflection Absorption Spectroscopy (PM-IRRAS). They were transferred to solid substrates by the Langmuir−Schaefer method for the inference about the organization of P3HT molecules and study of the influence of gold nanoparticles on such an organization. Two-layer LS films were prepared in 2.5 cm² square glass substrates at surface pressure of 30 mN/m and always keeping one side of the substrate parallel to the compression barrier.

Characterizations. UV−Vis Spectroscopy. The Langmuir films were characterized by UV−vis in situ using an HR2000...
Ocean optics spectrophotometer, ranging from 200 to 1100 nm, with a QR600-7-SR125BX optical fiber perpendicular to a planar mirror placed at the bottom of the Langmuir trough. The background spectra were recorded with subphase of ultrapure water or gold nanoparticles. The optical anisotropy was analyzed by transmission UV–vis with incident light horizontally polarized. The LS films were rotated with the aid of a special holder, and the measurements were performed on a Hitachi U-2001 spectrophotometer.

Fluorescence Spectroscopy. The photoluminescence of the solutions was analyzed in a Shimadzu RFWC3501 spectrophotometer. The Langmuir films were characterized under a Fluorescence microscope (Olympus BXFM-F with a Q-Color 5 camera) coupled to a Langmuir trough.

PM-IRRAS. The infrared spectra were taken on the surface of the Langmuir films under pressure of 30 mN/m, with an incidence angle of 80°, PEM wavelength of 6666.5, 0.550 retardation, and 6000 scans, in PMIS50 equipment of KSV Instruments (8 cm⁻¹ resolution).

SEM-FEG. The samples were prepared on glass and iron substrates in the form of cast films for the characterization of the nanoparticles in solution. The presence of nanoparticles was observed in the LS film. The images were obtained on Philips model XL30 and FEI Magellan 400 L instrument.

Photodegradation. The photodegradation experiments were conducted using the illumination of a white halogen light (50W, 12W - Osram) of 17 mW/cm² power placed 30 cm from the surface of the LS films.

RESULTS AND DISCUSSION

Gold Nanoparticles (AuNp and AuNpOctathiol). The synthesis of gold nanoparticles described in this paper has been previously reported in the literature, and the ratio of one mol of gold to two mol of sodium citrate enables the obtaining of spherical nanoparticles of approximately 20 nm diameter determined by the absorption band in the UV–vis (520 nm) and the images obtained by SEM-FEG.

The product from the encapsulation of AuNp by 1-octadecanothiol showed solubility in chloroform due to the presence of thiolates formed when the sodium citrate was removed by a treatment with a strong base (sodium hydroxide, NaOH), and 1-octadecanethiol molecules were incorporated in the gold nanoparticles. The solution exhibited a blue color, and its absorption spectra showed a broad band centered at 540–570 nm range. The size and distribution of the AuNpOctathiol spin-coated films were analyzed by SEM-FEG on silicon substrate. Sphere nanoparticles of approximately 20 nm diameter and some triangular nanoparticles were obtained. The blue color of the solution of nanoparticles is due to the presence of nanoparticles of different shapes and a solvatochromic effect, since a solvent less polar than water was used (chloroform).

The UV–vis spectra and the SEM-FEG and TEM images are shown in the Supporting Information in Figures 1 and 2, respectively.

Langmuir Films – P3HT and Aqueous AuNp. The formation of a polymer Langmuir film at an air–water interface of the Langmuir trough is very dependent on the spreading conditions, such as type of solvent used and concentration of spread solution (isotherms of neat P3HT are shown in Figure 3 in the Supporting Information). Therefore, the Langmuir film of neat P3HT was studied in different concentrations, and the best spreading concentration was determined by surface pressure isotherms and in situ absorption spectroscopy in the UV–vis. The film formed by spreading the solution with 0.5 g/L showed the formation of aggregates visible to the naked eye and a film somewhat homogeneous. The film formed from the solution of 0.1 g/L showed no formation of visible aggregates.
agglomerates; therefore, this concentration was used in the studies of hybrid films.

First, the hybrid films were prepared with gold nanoparticles in the trough subphase and spreading the polymer on the surface. Figure 1 shows the isotherms obtained from P3HT and different concentrations of gold nanoparticles in the subphase. The parameters used in all isotherms were the same, i.e. molar mass of the repetitive unit of P3HT, concentration of P3HT in chloroform, and spreading volume. The behavior of the P3HT isotherm in the water subphase is comparable to that reported in the literature for the same polymer in a chloroform solution. No collapse was observed, and the value of the surface pressure exceeded 45 mN/m. The package of neat P3HT showed a mean area of approximately 9.5 Å^2 per repeating unit, while the area calculated, based on the molecular arrangement of the polymer backbone parallel to the water interface and the thiophene ring positioned vertically, is 14.7 Å^2. The lower area values can be indicative of either a bilayer formation or the folding of the polymer main-chain.

The presence of gold nanoparticles in the subphase shifts the isotherm to a higher area per repeating unit, shown in the inset of Figure 1. Such a shift is proportional to the increasing concentration of nanoparticles until the value at which there is a “saturation” of the interaction between P3HT and AuNp (10:1 AuNp/P3HT). Therefore, the concentration of AuNp 1.0 g/L was used for obtaining the LS films. This shift can be due to either the penetration of nanoparticles in Langmuir film or the Van der Walls interaction of the nanoparticles in the polar part of the polymer chains, which can cause an expansion of the monolayer, observed by the displacement of the pressure isotherm to higher areas.

The same study was conducted with a solution containing sodium citrate in acid medium in the subphase, with the same pH and concentration of the nanoparticle suspension. Under such conditions, the isotherm of neat P3HT was identical to the one obtained with ultrapure water subphase. This result indicates the increase in the area by the use of AuNp in the subphase is not due to differences in the pH or ionic force of the subphase but to interactions between the polymer and the AuNp and incorporation of the nanoparticles in the film. This behavior can be proven by the in situ analysis of the absorption in the UV–vis range while keeping the film at a 30 mN/m surface pressure (UV–vis in situ spectra are shown in Figure 4 in the Supporting Information). The comparison between the wavelength of maximum absorption (λ_max) of the spectra recorded for neat P3HT on water (525 nm) and AuNp (485 nm) subphases shows there was a 40 nm blue shift in the film containing AuNp. The spectra remained unchanged for over 2 h, which indicates that the films were stable in both cases during this period.

**Langmuir Films – P3HT and AuNpOctathiol.** Mixtures of P3HT and AuNp capped with 1-octadecanethiol (AuNpOctathiol) in chloroform were prepared from solutions of the neat compounds at 0.1 g/L concentration and characterized by UV–vis spectra (Figure 5 in the Supporting Information). P3HT and AuNpOctathiol exhibited λ_max at 450 and 555 nm, respectively, and in the spectra of the mixtures containing 96, 90, and 75 mol % AuNpOctathiol the presence of these two bands is clearly observed. The spectra of mixtures containing 50% and 25% of AuNpOctathiol revealed only one λ_max corresponding to the absorption band of P3HT, because its intensity is much more pronounced than AuNpOctathiol (UV–vis spectra in Figure 5 in the Supporting Information).
according to their molar ratios, as described in the equation below:

\[ A_{(12)} = x_1 A_1 + x_2 A_2 \]

where \( A_{(12)} \) is the average area occupied by the film of the mixture, \( x_1 \) and \( x_2 \) are the mole fractions of neat compounds, and \( A_1 \) and \( A_2 \) are the areas occupied by the films of the neat compounds under the same surface pressure. To study the interaction between molecules in the mixtures, we built a graph that relates the MMA at 15 mN/m (maximum surface pressure observed for the AuNpOctathiol isotherm) to the mass ratio of P3HT (Figure 2). The points represented with squares correspond to an ideal behavior, in which components are completely immiscible. Any diversion from an ideal curve shows evidence of miscibility. The star-shaped points correspond to the experimental areas of mixtures of P3HT and AuNpOctathiol. In all cases, the area of the mixture decreased in comparison to the ideal area indicating an attraction between molecules of P3HT and AuNpOctathiol, due to the Van der Walls forces between the aliphatic chain of 1-octadecanethiol molecule and the side chain of P3HT, which is more pronounced in higher P3HT proportions.

The mixtures with ratios of 10 and 4% of P3HT showed the lowest deviation from the ideal curve; therefore, the other characterizations were performed only for the mixtures with 75, 50, and 25% P3HT, which were also used for the obtaining of Langmuir–Schaefer (LS) films.

The Langmuir films of the mixtures were characterized by \textit{in situ} UV–vis spectra. Only one band with \( \lambda_{\text{max}} \) at 535, 550, and 564 nm for the films containing 75, 50, and 25% of P3HT, respectively, was observed. The spectrum of the film with the lowest proportion of P3HT (25%) showed one shoulder at approximately 690 nm, corresponding to the absorption of AuNpOctathiol, which was tenuous in the other two films (with 75 and 50% of P3HT). (UV–vis \textit{in situ} spectra are shown in Figure 6 in the Supporting Information.)

Polarization Modulation Infrared Reflection Absorption Spectroscopy (PM-IRRAS) is a technique of infrared reflection–absorption for the detection of the functional groups on thin surfaces, such as Langmuir films and observation
of the orientation of the groups at the air–water interface (perpendicular or parallel to the plane). The spectra in Figure 3a, acquired at a surface pressure of 30 mN/m, show two bands around 2900 and 2800 cm⁻¹ corresponding to the stretching of axial asymmetric deformations of aliphatic CH. There occurs a displacement of the bands of the mixtures with a higher proportion of AuNpOctathiol in comparison to the spectrum of neat P3HT. This behavior is not observed in the Fourier Transform Infrared spectroscopy (FTIR) of the cast films in transmission mode. The spectrum of neat AuNpOctathiol could not be obtained, because no stable Langmuir film had been formed. The two peaks related to the CH stretching are pointed upward in the spectra, which indicates the hydrocarbon side-chain of both P3HT and 1-octadecanethiol are somewhat perpendicular to the surface.

The Langmuir films of neat P3HT and P3HT containing gold nanoparticles are stable at high surface pressures; therefore, they can be easily characterized by in situ bright field and under fluorescence microscopes. The images obtained are shown in Figure 4. Areas of higher and lower concentrations (bright and dark regions, respectively) and homogeneous. The Langmuir films of neat P3HT and P3HT containing AuNp in the subphase were observed. Aggregates of polymers are clearly seen in the fluorescence images (excitation range 400–440 nm; emission range 450–900 nm). For the mixtures of P3HT and AuNpOctathiol, those regions also appear, but by increasing the ratio of AuNpOctathiol, the Langmuir films become more homogeneous.

**Langmuir–Schaefer (LS) Films.** LS films were obtained in square glass substrates of 2.5 cm² and surface pressure of 30 mN/m. Two layers were deposited on the substrate parallel to the compression barrier of the Langmuir trough. The films were used for the investigation of the influence of gold nanoparticles on the orientation of the molecules in the Langmuir and LS films with consequent optical anisotropy of the P3HT films. The absorption spectra in the UV–vis were acquired with linearly polarized incident light, and the films were placed in different rotatational positions with the aid of a rotating holder. The 0° position was defined as the direction parallel to the compression barrier; therefore, the film was fixed with the side aligned to the barrier at the 0° position.

The relation between the maximum absorbance, at 550 nm wavelength, and the position of the films in the rotating holder are shown in Figure 5. The behavior of all curves is sinusoidal, and the mutual absorbance values were found at positions 90 and 270°. As the light used was horizontally polarized, the angles of strongest absorptions imply that the polymer main-chains, responsible for the visible light absorption, are parallel to the compression barrier in the Langmuir films. In the curves of neat P3HT and P3HT containing AuNp in the subphase, the behavior of the optical anisotropy of the P3HT molecules is similar. For the mixture of P3HT and AuNpOctathiol, the increase in AuNpOctathiol causes a decrease in the difference between the maximum and the minimum absorbances, which disappears almost completely for the films obtained with the mixture of 75% AuNpOctathiol, due to the interference of the nanoparticles in the organization of the polymer at the interface.

The results can be also represented numerically by the dichroic ratio (DR), which, in this case, is the ratio between the values of the minimum and maximum absorbances (0 and 90°, respectively). The values of DR for the LS films of neat P3HT and P3HT containing AuNp were approximately 1.55 and 1.57, respectively, and indicate that the films have some orientation, and there is practically no difference in the orientation of the polymer when gold nanoparticles are present. Another way to calculate the anisotropy is to use the in-plane order parameter S, given by $S = (A// − A\perp)/(A// + 2A\perp)$. For the LS film of neat P3HT and P3HT containing AuNp, parameter $S$ is 0.2, which is an evidence of the noninterference of the AuNp in the molecules orientation in the film.

In films containing AuNpOctathiol, the dichroic ratios were approximately 1.44, 1.28, and 1.04, and parameter $S$ was 0.16, 0.12, and 0.05 for the films containing 75%, 50%, and 25% of P3HT, respectively. The increase in the concentration of nanoparticles capped with 1-octadecanethiol in the film decreases the DR and parameter $S$; therefore, the presence of these nanoparticles decreases the organization of the polymer molecules.

Images by scanning electron microscopy (SEM-FEG) (Figure 6) were obtained for the LS films to confirm the presence and morphology of the nanoparticles. The image of neat P3HT film shows nonnanometric structure (Figure 6—image A). The films of P3HT containing AuNp show spherical nanoparticles of approximately 25 nm diameter (Figure 6 - images B and C), which correspond to the values found for the SEM measurements of the gold nanoparticles previously mentioned in this paper. The distribution of the nanoparticles in the films is uniform, as shown in Figure 6 — image C recorded from the same film with lower magnification. The films of mixtures of P3HT and AuNpOctathiol (Figure 6—images D, E, F) show nanoparticles forming agglomerates with no homogeneous distribution throughout the film. The same behavior was observed for the images obtained from a cast film of a chloroform solution of neat AuNpOctathiol.

Conjugated polymers are susceptible to degradation by oxygen and light. Photodegradation experiments were performed on the LS films for the study of possible enhancements in the photostability of the P3HT films due to the presence of AuNps. The films were continuously exposed to a white light at room temperature for approximately a week, and their absorption spectra were recorded in different periods of exposition. In the first stages of the photodegradation of polythiophene, the absorption intensity of the visible light decreased but with no color film fading perceptible to the naked eye. In subsequent stages, the value of the wavelength of maximum absorption decreased until the sample had become colorless. In general, the process of photodegradation of the films begins on the surface and affects the inner layers of the film afterward. Figure 7 shows the decay curve of absorbance (ABS, normalized by the absorbance value before exposure to light − ABS₀) of all films at a 540 nm wavelength. After 100 h of exposure, the values of films of neat P3HT and P3HT containing AuNp showed half the initial intensity. The degradation was similar for both films, which indicates that the presence of gold nanoparticles in the films does not significantly affect the degradation of P3HT. For the mixed films of P3HT and AuNpOctathiol, the higher the concentration of AuNpOctathiol, the lower the decrease in absorbance, which shows the presence of AuNpOctathiol minimizes the photodegradation process. This fact may be related to the interaction between molecules of P3HT and AuNpOctathiol, previously discussed in the explanation of Figure 2, which indicated an attraction between molecules of P3HT and AuNpOctathiol, due to the van der Waals force.
CONCLUSIONS

Hybrid Langmuir–Schaefer films of P3HT and AuNP or AuNP–thiol have been obtained and characterized. The presence of gold nanoparticles in these films leads to a modification in the orientation of the P3HT molecules in the films and consequent interference in their optical anisotropy. The maximum absorbance values were found at positions 90° and 270°, and the behavior of the curves is sinusoidal. As the light used was horizontally polarized, the angles of strongest absorptions imply that the polymer main-chains are parallel to the compression barrier in the Langmuir films. The behavior of the optical anisotropy of the P3HT molecules is similar in all the cases, in which the anisotropy decreases with the increase in the AuNP–octathiol concentration in these films.

The presence and morphology of nanoparticles in the LS films were confirmed by analyses of SEM–FEG. Such a morphology corresponds to the values found for the SEM measurements of AuNP and AuNP–octathiol. Photodegradation was observed for all the films after 100 h of exposure. The films of neat P3HT and P3HT/AuNP showed a similar degradation, indicating that the presence of gold nanoparticles in the films does not significantly affect the degradation of P3HT. For the mixed P3HT/AuNP–octathiol films, the higher the concentration of AuNP–octathiol, the lower the decrease in absorbance, which indicate the interaction between molecules of P3HT and AuNP–octathiol minimizes the photodegradation process.

ASSOCIATED CONTENT

* Supporting Information
Some figures and explanations for a better comprehension of results. This material is available free of charge via the Internet at http://pubs.acs.org.

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Notes
The authors declare no competing financial interest.

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