

## Self-assembling and spectroscopic properties of soluble linear acenes\*

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**Abstract:** The gelling and spectroscopic properties of new soluble *disubstituted acenes* (anthracene, tetracene, pentacene) are reported. It is shown that the 2,3-alkoxy derivatives gel a large variety of organic solvents, a process that is related to the linear shape of the molecule. The gelation involves the formation of *nanofibers* through noncovalent interactions (van der Waals,  $\pi$ - $\pi$  stacking), weaving a 3D supramolecular network or a deposit made of isolated fibers on surfaces (mica or graphite). Transport of photons through this type of nanostructure has been evidenced by the observation of an efficient energy transfer occurring from an anthracene light-harvesting matrix to a tetracene energy trap doping agent.

**Keywords:** aromatic compounds; gels; pentacene; tetracene; anthracene.

### INTRODUCTION

Linear polycyclic acenes are of interest in various fields due to their unique properties which encompass photoreactivity ([4+4] cycloaddition...) [1–7], strong  $\pi$ - $\pi$  absorption coupled with high luminescence yields [8], large charge mobility in the solid phase [9], etc. These properties could be exploited for designing new types of materials such as photochromic devices [10,11], fluorescent sensors [12], organic field effect transistors, and other inexpensive plastic optoelectronic systems [13]. However, these potential applications are strongly limited by the very low solubility of the compounds in the usual organic matrices (and solvents) which hampers their processing into thin films or matrices of given geometries. This detrimental insolubility increases with the number of rings in the linear acene (i.e., anthracene, tetracene, pentacene...), whereas the properties of the material become more attractive for applications (higher charge mobility, large red-shift of the absorption and emission spectra toward the IR region...). In addition, this insolubility makes more difficult or precludes the study of the photoreactivity in fluid solutions; therefore, tetracene and pentacene have been mainly investigated in the solid state [14–16].

We have already shown that the substitution of the anthracene nuclei by two long linear alkoxy chains (O-*n*-C<sub>10</sub>H<sub>21</sub>) greatly improved the solubility of the materials, which can therefore be easily dissolved in a large variety of common organic solvents. In addition, the corresponding photodimers dis-

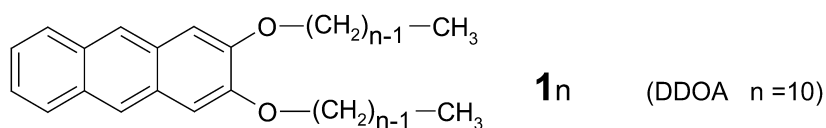
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played also a remarkably high solubility (even in  $\text{CCl}_4$  or alkanes), whereas the parent compound (anthracene photodimer) is practically insoluble in the usual organic solvents [17,18]. Moreover, we discovered that the 2,3-di-*n*-decyloxy derivative (DDOA) was able to gel a series of solvents (including supercritical  $\text{CO}_2$  [19]) at very low concentration [20,21], leading to a 3D supramolecular network woven with nanosized fibers formed from DDOA aggregates [22]. As reported in the present paper, the phase transition (sol-gel) is accompanied with large and specific spectral changes (UV, fluorescence, IR), which give first-hand information on the molecular organization within the fibers. Due to this specific molecular organization, these fibers act as light-harvesting antenna in which energy-transfer processes are particularly efficient. Besides, the disubstitution of tetracene with two long alkoxy chains produces soluble materials, displaying, as shown for the 5,12 derivative, interesting photochromic properties [23,24]) and for the 2,3 isomer organogelating properties similar to those of DDOA. Along these lines, the solubility of pentacene is greatly improved by substituting the nucleus with two alkoxy chains, giving also materials endowed with gelling properties. In this account, the gelling and spectroscopic properties of some 2,3-di-*n*-alkoxy-anthracene, tetracene, and pentacene, respectively, are reviewed and an example of energy transfer is briefly illustrated.

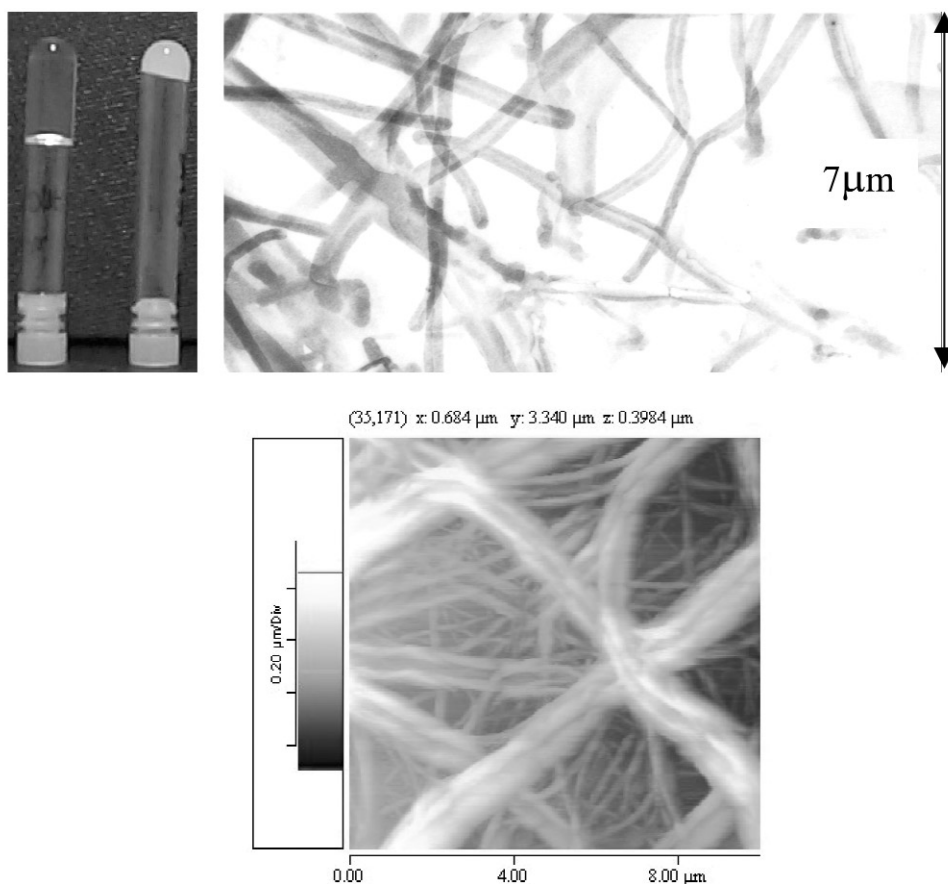
### 2,3-DI-*n*-ALKOXYANTHRACENES

It was shown that 2,3-di-*n*-alkoxyanthracenes (**1n**) ( $n \geq 6$ ), Scheme 1, are able to gelify, at low concentration, a large variety of organic fluids at room temperature (see illustration, Fig. 1) [21]. This chain-length-dependent thermoreversible gelification is only observed with linear polymethylene sequences and exclusively with the 2,3-disubstituted derivatives (branched chains and extra substitution on the anthracene nucleus were shown to give inactive compounds, under the same conditions). In addition, the presence of an oxygen atom between the chain and the aromatic nucleus is required. It generates a small dipole moment (ca. 1.9 D) inducing dipole-dipole interactions, and forces the two chains into a specific conformation (see Fig. 5) favoring intermolecular rather than intramolecular interchain interactions.



**Scheme 1**

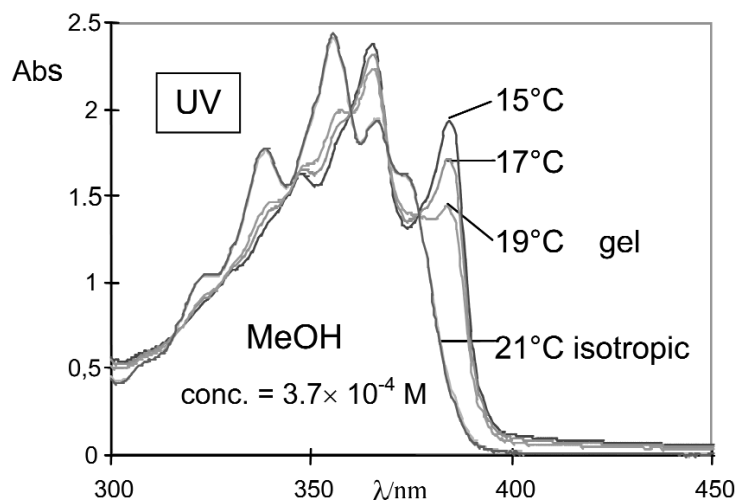
If protic solvents such as alcohols (methanol, ethanol...) and aliphatic amines are gelified by DDOA and its derivatives, other organic fluids such as toluene, acetone, acetonitrile, methylene chloride, dimethyl sulfoxide (DMSO), dimethylformamide (DMF), propylene carbonate, alkanes, etc. are also readily rigidified. This underlines the fact that the proticity of the solvent able to contract directional H bonds is not necessary for constructing the 3D supramolecular framework imprisoning the fluid. Therefore, the driving force is mainly due to  $\pi$ - $\pi$  stacking, van der Waals, and other hydrophobic interactions. That DDOA is able to gel supercritical  $\text{CO}_2$  for giving organic aerogels [19] strongly supports this aggregative mechanism.



**Fig. 1** Ethanol gels of DDOA. Top left: photographs of the translucent and opaque materials corresponding to low ( $<10^{-3}$  M) and high ( $>10^{-2}$  M) concentrations of gelator, respectively. Top right: TEM image of the gel (conc.  $\approx 10^{-3}$  M), note the hollow character of the fibers. Bottom: AFM image (tapping mode) of the gel obtained in *n*-heptane (conc.  $\approx 10^{-3}$  M).

The classical techniques used for the structure investigation of gels give information on the shape and mechanical properties of the soft material, but provide no straightforward evidence on the molecular packing in the fibers. Therefore, except for one example [25], no such information is actually available in the literature; only hypothetical packing has been postulated, implying that gel and crystal display identical structures (which is certainly not always the case) [26,27].

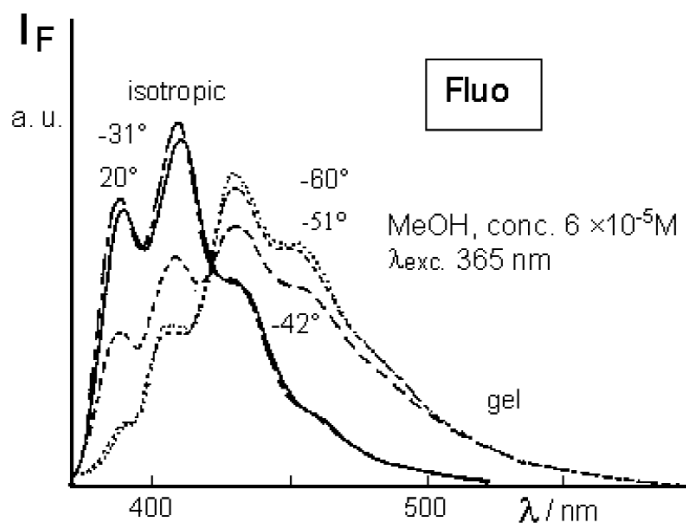
As spectroscopic and photophysical properties of anthracene are unique and very sensitive to various parameters (solvent, concentration, substitution, quenchers...), this chromophore could act as a very powerful probe able to reveal weak molecular interactions and information on aggregation processes (via excimer species formation...). In the present situation, anthracene is expected to play both the role of an actor (gelling agent) and a self-sensor (UV, IR, fluorescence spectroscopies) on its own molecular aggregation. Indeed, the sol-gel phase transition induces specific, and reversible, spectroscopic changes of the medium as exemplified by the UV and fluorescence spectra (Figs. 2 and 3, respectively).



**Fig. 2** Electronic absorption spectra of DDOA in methanol vs. temperature.

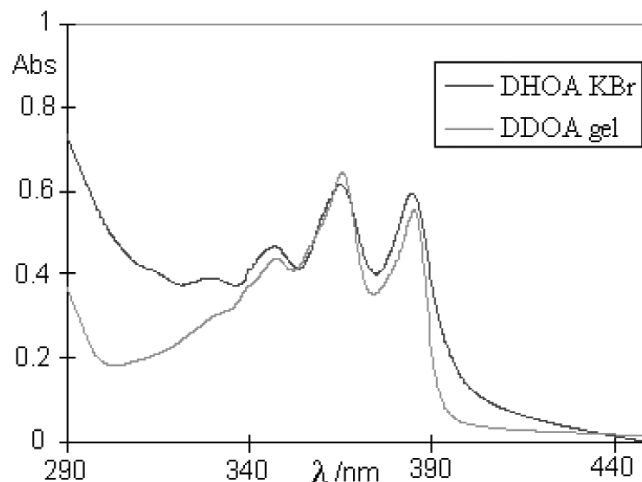
The clear bathochromic shift of the lower energy transition band (300–400 nm), coupled with the splitting of the higher energy transition ( $^1B_b$  band pointing around 250 nm, not shown here [21]) indicates a  $\pi$ - $\pi$  stacking of the chromophores in the gel, the anthracenes experiencing some degree of parallelism along their long axis (this fact is supported by the fine structure of the lower energy band associated with the disappearance of the  $^1L_b$  transition, which is polarized along the long axis of anthracene, as well as the 250-nm split band in the gel) [21,28,29].

The analysis of the fluorescence spectra supports the proposal for the structure of aggregates: The structured band of the isotropic solution is red-shifted upon gelification and the fluorescence decays are not monoexponential, these facts being connected with the occurrence of multimolecular emitting species in the gel phase [30,31].

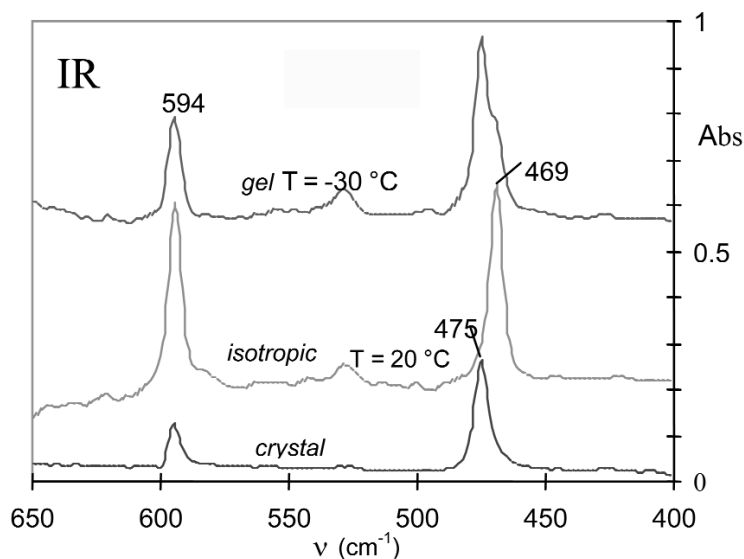


**Fig. 3** Fluorescence spectra of DDOA in methanol vs. temperature.

Besides, UV and IR spectra of DDOA display similar spectroscopic signatures both in the gel phase and in the solid, pointing to the close resemblance of the local environment of the anthracene sub-unit in these two media (Fig. 4). IR spectroscopy also reveals that the alkyl chains exhibit a *trans* configuration within the gel (in contrast to the solution, in which numerous kinks are present).



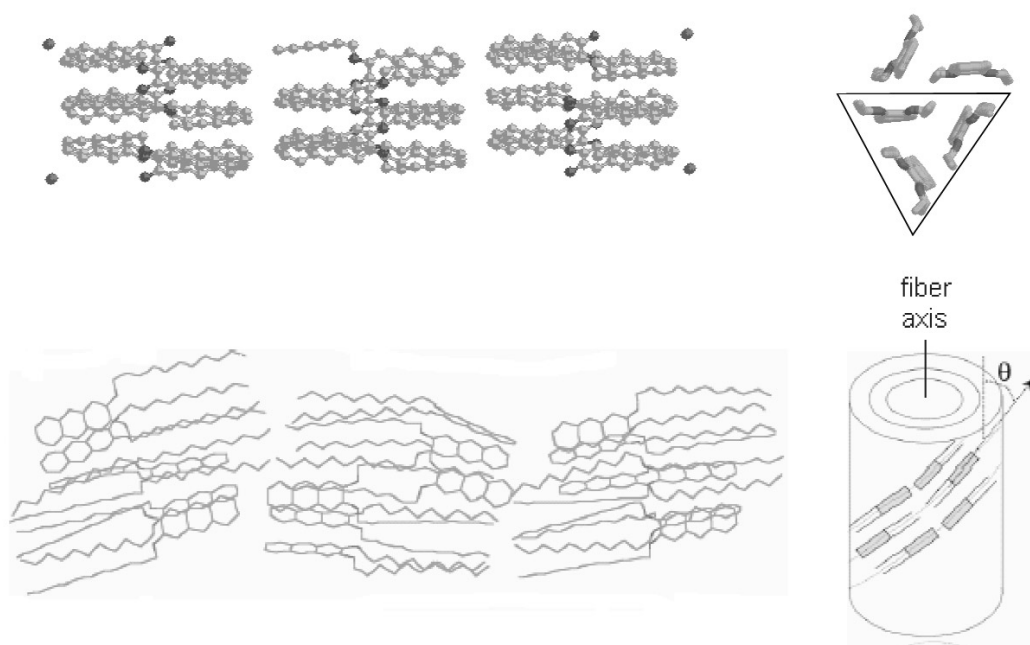
**Fig. 4a** UV absorption spectra of DHOA (crystal) and DDOA (gel in methanol). DHOA gel displays the same spectrum as DDOA. Adapted from ref. [21], reproduced by permission of The Royal Society of Chemistry on behalf of the Centre National de la Recherche Scientifique (CNRS).



**Fig. 4b** IR spectra of DHOA (**16**) in gel phase ( $\text{CCl}_4$ ), isotropic solution ( $\text{CCl}_4$ ), and in the crystal, respectively. Adapted from ref. [22] with permission; copyright © 2003 American Chemical Society.

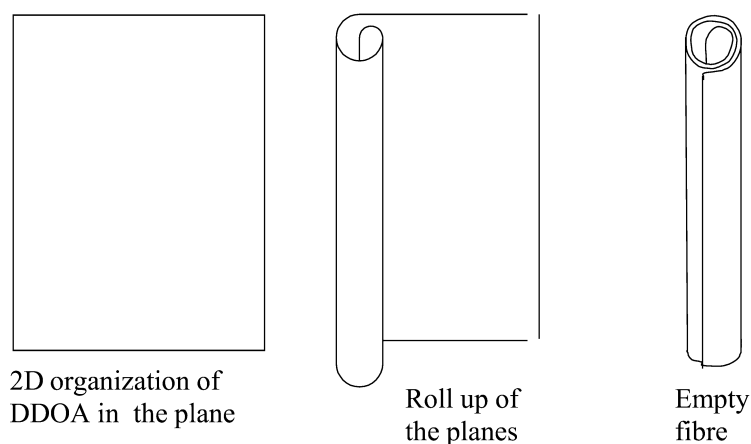
Finally, inspection of the spectroscopic properties of 2,3-dihexyloxyanthracene (DHOA, **16**), which gives both single crystals and gel, indicates that the structure in the gel is comparable to that of the crystal packing; in the latter, the DHOA forms layers in which the molecules are arranged in tri-

ads (Fig. 5, top). A detailed analysis of aligned aerogel fibers of DDOA with polarized light (IR and fluorescence dichroism), coupled with molecular modeling allowed us to propose a 3D arrangement for the gelator as depicted in Fig. 5 (bottom) [22]. This molecular organization is reminiscent of the crystal packing of DHOA [32] as the molecules are arranged in triads forming layers, but with conformational changes imposing an arrangement along helicoidal coils in concentric cylinders. This model was recently supported by a fine analysis of the strong birefringence of magnetic aligned DDOA fibers [33].



**Fig. 5** Top: molecular packing of DHOA (**16**) in the crystal. Bottom left: AMBER-optimized arrangement of 18 molecules of DDOA. Right: proposed molecular organization of DDOA molecules within the fibers. Adapted from ref. [22] with permission; copyright © 2003 American Chemical Society.

Taking into account the preceding data (and the specificity of the 2,3-dialkoxyanthracenes to pack into layers), coupled with the hollow character of fibers (see Fig. 1), a hypothetical model for formation of the fibers could be proposed (Fig. 6). It is supported by the homogenous shape of the fibers and their “spontaneous” formation (microscopic observations) without apparent increase in length.

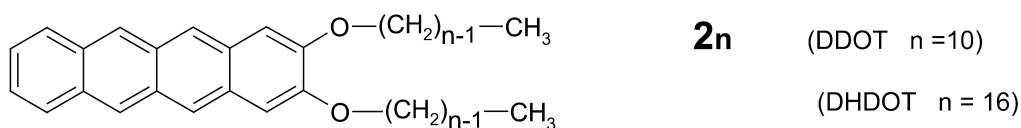


**Fig. 6** Proposed model for DDOA fiber formation.

As indicated in the introduction, our investigation was extended to other aromatic substrates such as tetracenes and pentacenes which are of great interest for future optoelectronic devices in order to examine whether these compounds follow the same behavior as DDOA and if these compounds could be easily processed into nanofibers or other organized structures.

### 2,3- DI-*n*-ALKOXYTETRACENES

2,3-Didecyloxytetracene (DDOT) and 2,3-dihexadecyloxytetracene (DHDOT) **2n**, Scheme 2, produce orange-colored materials [34] soluble in a large variety of solvents in contrast to the parent molecule (tetracene) which cannot be easily dissolved in common organic fluids even upon heating.

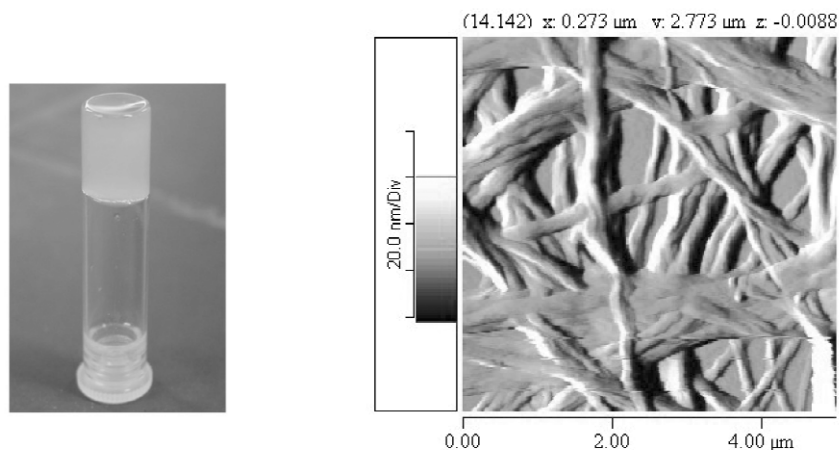


**Scheme 2**

As shown in Table 1, these two compounds are able to rigidify organic fluids at low concentration, especially DHDOT ( $n = 16$ ) (see illustration, Fig. 7) which is more efficient than DDOT ( $n = 10$ ) and could be considered as a supergelator [35,36]. That DHDOT was found to be a better gelator than DDOT could be assigned to the best balance between the chain length and the aromatic nuclei size (for DDOA, which possesses an anthracene subunit, decyloxy was preferable to hexadecyloxy). It is also remarkable that non-H-bonding solvent such as linear alkanes or hydrocarbons could be easier to gel than alcohols. Here again, hydrophobic forces (van der Waals interactions,  $\pi$ - $\pi$  stacking) are the main driving forces controlling the gel formation.

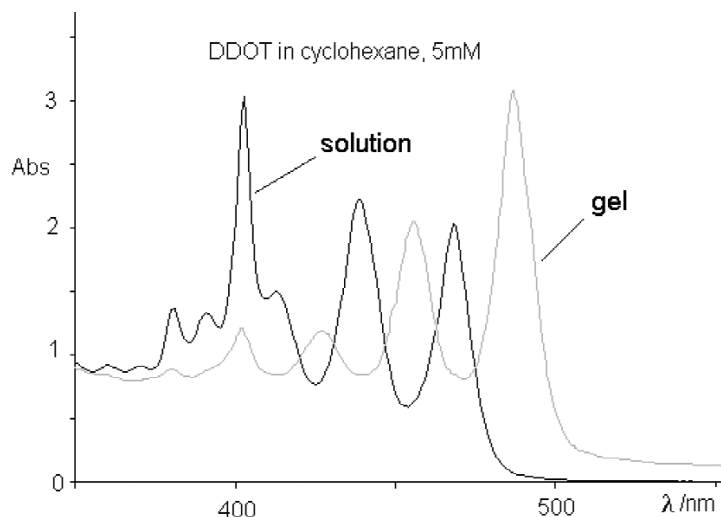
**Table 1** Gelling ability at room temperature of DDOT and DHDOT. **Bold figures:** critical concentrations; nd: not determined

Solvent	DDOT ( <i>n</i> = 10)		DHDOT ( <i>n</i> = 16)	
	[mg/ml]	Conc. ( $10^{-3}$ M)	[mg/ml]	Conc. ( $10^{-3}$ M)
<i>n</i> -Hexanol	0.92	<b>1.71</b>	1.24	<b>1.75</b>
<i>n</i> -Octanol	2.69	4.98	0.70	<b>0.987</b>
<i>n</i> -Decanol	2.49	4.61	0.97	<b>1.37</b>
<i>n</i> -Hexane	5.43	10.1	0.85	<b>1.20</b>
<i>n</i> -Heptane	nd	nd	0.61	<b>0.86</b>
<i>n</i> -Nonane	nd	nd	1.12	<b>1.58</b>
<i>n</i> -Tetradecane	nd	nd	0.993	<b>1.40</b>
Cyclohexane	0.21	0.4	0.28	<b>0.40</b>
Me-cyclohexane	5.5	10	0.68	<b>0.995</b>
Dichloromethane	nd	nd	5.31	7.49
Acetone	nd	nd	2.45	3.46
DMSO	1.14	<b>2.1</b>	1.49	<b>2.1</b>

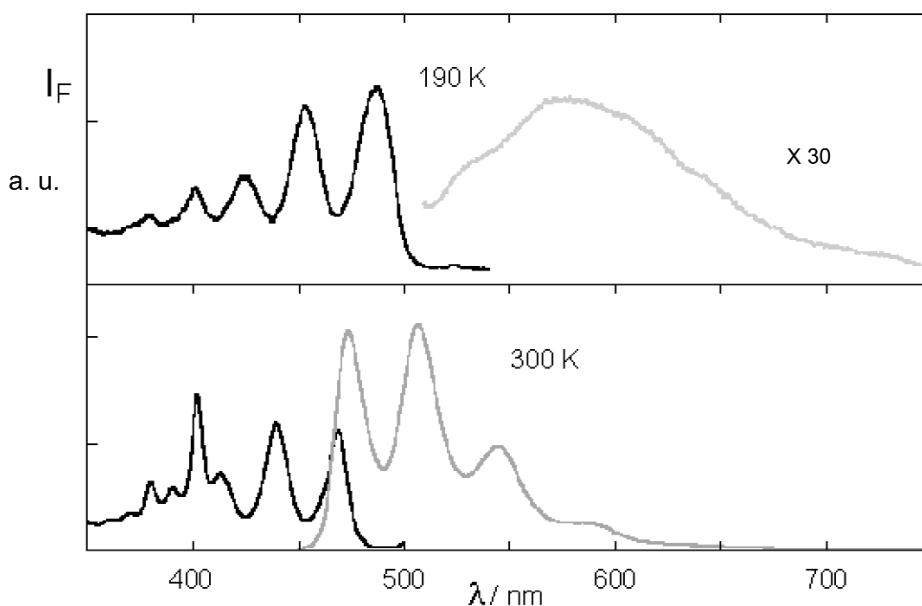
**Fig. 7** Left: photograph of a nonane gel of DHDOT ( $\approx 2 \times 10^{-3}$  M). Right: Topography or signal error images of DHDOT fibers on mica surfaces obtained by AFM in tapping mode (2 mg/ml of nonane partially evaporated). The smaller fibers typically have a width of about 150–200 nm. Adapted from ref. [34] with permission; copyright © 2005 American Chemical Society.

As shown in Fig. 8 for DDOT, the UV spectrum of tetracene derivatives is strongly modified upon gelification. In addition to a large bathochromic shift ( $\approx 790 \text{ cm}^{-1}$ , for the long-wavelength vibronic transition) indicating the stacking of the molecules, the band peaking at ca. 400 nm, ascribable to the  $^1L_b$  transition, has almost completely disappeared; this strongly suggests some degree of parallelism between the tetracene long axes in the gel fiber (exciton splitting) [21,28,29]. These spectral changes are reminiscent of those found for DDOA. Besides, the fluorescence emission spectra are affected in the same way as DDOA, the well-structured spectrum of the fluid solution is transformed for the gel phase into a red-shifted nonstructured band (Fig. 9), accompanied by a net intensity drop. This might be related to tetracene aggregate formation displaying a weaker fluorescence intensity than the monomer species (not subject to self-quenching in the diluted fluid solution as occurring in the fibers) [30,31]. Thus, these data support a molecular organization in the gel similar to that prevailing with DDOA, the driving force being here again mainly  $\pi$ - $\pi$  stacking and dipole-dipole interactions.





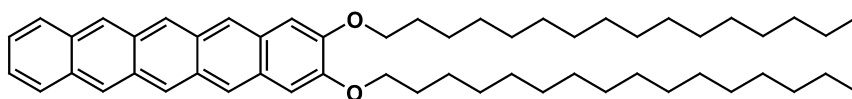
**Fig. 8** Absorption spectra of DDOT in cyclohexane at room temperature (gel state) and above melting temperature (isotropic solution). Typically, the absorption spectra of the gels undergo a bathochromic shift as compared to the isotropic solution (up to 20 nm). In the gel state, the absorption maxima are independent of the solvent (the same spectroscopic behavior is observed for DHDOT). Adapted from ref. [34] with permission; copyright © 2005 American Chemical Society.



**Fig. 9** Excitation and emission spectra of DHDOT ( $10^{-4}$  M) in methycyclohexane. A nonstructured emission is observed for the gel phase at 580 nm (top), strongly bathochromic and of lower intensity as compared to that of the isotropic solution at 300 K (bottom). The excitation spectra reflect the absorption spectra of the gel and the isotropic solution, respectively. (Emission spectra,  $\lambda_{\text{exc}}$ : 440 and 488 nm for the isotropic and gel phase, respectively; excitation spectra,  $\lambda_{\text{obs}}$ : 507 and 580 nm, respectively). Adapted from ref. [34] with permission; copyright © 2005 American Chemical Society.

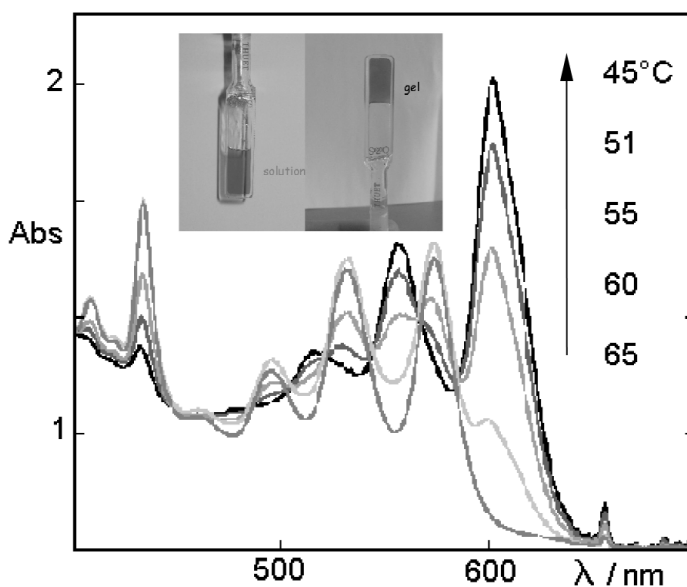
### 2,3-DI-*n*-HEXADECYLOXPENTACENE (DHDOP)

The 2,3-disubstitution of pentacene with a long linear hexadecyloxy ( $\text{O}-[\text{CH}_2]_{15}-\text{CH}_3$ ) chain, DHDOP, Scheme 3, produces a relatively soluble red-colored material [37], very sensitive to the presence of oxygen. The solutions obtained are red-colored. Upon cooling down from 65 to 45 °C, a chloroform solution (conc. DHDOP  $\approx 2 \times 10^{-3}$  M), the red medium turns to gray–purple in connection with the formation of a gel (see Fig. 10). The evolution of the UV spectrum upon the gelification is similar to the tetracene derivatives (vide supra) with a clear bathochromic shift ( $\approx 832 \text{ cm}^{-1}$ , for the longest wavelength vibronic component; it should be noted that this shift is slightly larger than for DDOT or DHDOT, which might be related to the larger sized  $\pi$ -chromophore) is accompanied by the disappearance of the 434-nm band, which is a  $^1\text{L}_b$ -type transition (see Fig. 10). In addition, the atomic force microscopy (AFM) image (not shown) of the soft material is typical of those given by DDOA and tetracene derivatives. As the color change is thermo-reversible, DHDOP is a thermochromic compound [38].



DHDOP

Scheme 3



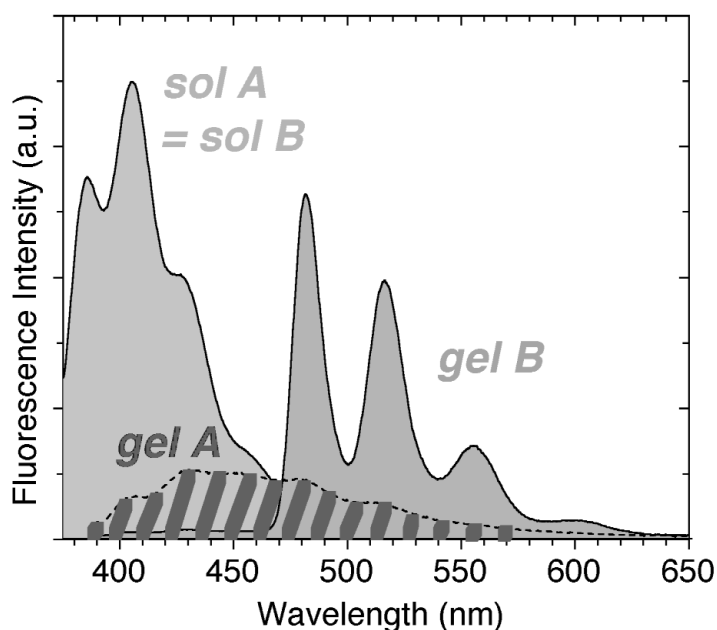
**Fig. 10** Evolution of a chloroform solution of DHDOP ( $2.6 \times 10^{-3}$  M/chloroform) vs. temperature. Inset: photographs of the chloroform solution (red, left) of DHDOP and of the corresponding gel in sealed cells (dark blue, right).

### LIGHT-HARVESTING AND ENERGY TRANSFER IN GEL FIBERS

As precedently shown, when substituted on 2,3 positions with long linear alkoxy chains, anthracene, tetracene, and pentacene become soluble and form gels with a large variety of solvents. From spectroscopic considerations and inspection of AFM or transmission electron microscopy (TEM) images, it emerges that the molecular packing in the fibers is similar for these materials (of note, the crystal packing of the parent arenes is similar and called type A by Stevens [39]; this packing mode controlled by the

oblong shape of the molecule allows only weak  $\pi$ - $\pi$  intermolecular overlaps). Thus, it was of interest to test whether mixed gels and electronic energy transfer could be implemented with these materials.

Figure 11 represents the fluorescence spectra of a diluted solution ( $2.0 \times 10^{-5}$  M) of DDOA in methylcyclohexane in the absence and presence of less 1 % of DDOT. It emerged that DDOA gel fibers act as a very powerful antenna (light-harvesting), i.e., at room temperature (isotropic phase), the fluorescence of the solutions (pure DDOA and DDOA solution containing 0.75 % DDOT) are identical, the light being mostly adsorbed (>99 %) by DDOA and fluorescence emitted from DDOA, no significant energy transfer occurred in these conditions between the two chromophores. In contrast, at low temperature (170 K) when the medium is gelled, a nicely structured spectrum characteristic of tetracene was recorded. The process is thermally reversible. Thus, anthracene donor chromophores in the organogel fibers act as a compact light-harvesting matrix that excites tetracene acceptor luminophores. Imaging of these fibers by fluorescence confocal microscopy confirms that DDOT is closely associated with the DDOA fibers and does not form large agglomerates on the exterior of DDOA. In addition, tetracene derivatives whose the molecular shape does not match that of DDOA (and derivatives which do not give gels, such as 5,12-dicycloxytetracene) do not perturb the fluorescence emission of DDOA gels. For DDOT or DHDOT, the *fine* structure of the doped gel emission must result from a homogeneous energy distribution for the DDOT excited states due to isolated DDOT molecules in DDOA gel matrix, contrasting with the behavior of pure DDOA and DHDOT gels. Of note, the fluorescence spectrum of the last tetracene derivatives in DDOA gel, displays a finer structure than the fluid-diluted solutions of DDOT or DHDOT, indicating, in addition, a specific organization of the dopant within the fibers.



**Fig. 11** Fluorescence spectra in MCH of: (A) DDOA and (B) DDOA+DDOT (0.75 %), [DDOA] =  $2.0 \times 10^{-5}$  M: (light blue) isotropic sol of (A) and (B) at 298 K (the spectra are the same),  $\lambda_{\text{exc}} = 366$  nm,  $\lambda_{\text{em}}^{\text{max}} = 412$  nm; (dark stripes) gel of (A) at 170 K,  $\lambda_{\text{exc}} = 382$  nm,  $\lambda_{\text{em}}^{\text{max}} = 432$  nm; gel of (B) at 170 K,  $\lambda_{\text{exc}} = 382$  nm,  $\lambda_{\text{em}}^{\text{max}} = 482$  nm.

This system shows an energy-transfer efficiency that largely competes with that of other light-harvesting organogelators [40–44].

## CONCLUSION AND PERSPECTIVES

This paper shows that anthracenes, tetracenes, and pentacenes self-assemble into stable nanostructures merely owing to a 2,3-bis-*n*-alkoxy substitution. The fibers forming these 3D supramolecular networks could be aligned with intense magnetic fields [33] or mechanically [45], making these materials of interest for various applications [46]. Moreover, *attractive perspectives* arise from the *very efficient energy-transfer process*. Indeed, the host organogelator (here DDOA) acts as a chromophore-rich light-harvesting matrix and as an organizing scaffold. With their nanometric dimension, the fibers could furthermore act as an optical confining media and thus as a waveguide for the directional transfer of optical information. Besides, one might expect an improvement of the tetracene and pentacene conductivity upon organization into gels, as recently described for other polycyclic aromatic derivatives [47].

## ACKNOWLEDGMENTS

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