A new low molecular mass organic gelator (LMOG) bearing 1,3,4-oxadiazole and azobenzene groups, namely N-(3,4,5-octanoyloxyphenyl)-N'-4-[(4-hydroxyphenyl)azophenyl]-1,3,4-oxadiazole (AOB-t8), was designed and synthesized. The organogelator has shown great ability to gel a variety of organic solvents to form stable organogels with the critical gelation concentration as low as $5.0 \times 10^{-4}$ M and, therefore, can be considered a supergelator. It has been demonstrated that the aggregation morphology and surface wettability produced by organogels strongly depend on the nature of gelling solvents. A structure of upwisting fibers on the substrate and superhydrophobicity were observed in the xerogel formed from 1,2-dichloroethane. The wettability of the xerogel could also be tuned by applying a sol-gel process with different solvents. Cooperation of hydrogen bonding, \(\pi-\pi\) interaction, and Van der Waals force are suggested to be the main contribution for this self-assembled structure. The unique and tunable surface properties such as superhydrophobicity distinguish the obtained organogels as a novel class of functional materials.

**Keywords:** 1,3,4-oxadiazole, Morphology, Organogel, Superhydrophobic surfaces

**Introduction**

In the past decade, low molecular mass organogelators (LMOGs) have attracted broad attention because they can self-assemble into diverse nano/micro-structures such as fibers, particles, tubes, and helical ribbons through specific noncovalent interactions such as hydrogen bonding, \(\pi-\pi\) interaction, hydrophobic interaction, Van der Waals force, and so on (1–3). Supramolecular self-assembly is the spontaneous process of molecular aggregation into ordered nanostructures, which provides a bottom-up approach to obtain structural regularity of various morphologies (4); however, a complete mechanism for the self-assembled supramolecular structures is still beyond our understanding. Recently, there has been a growing interest in tuning morphology by changing molecular structures (5), altering the composition of a binary gel (6, 7) and solvents (8, 9), as well as using ultrasound (10, 11), light (12, 13), and so on. For example, Nandi and co-workers reported for the first time on the hierarchical tuning of one-dimensional morphology from helical bunched fibers to rods and hollow tubes, by changing the composition of riboflavin-melamine in a hydrogel system (2). Li and co-workers demonstrated that the structural transition from organogels to flower-like microcrystals in the dipeptide self-assembling system can be induced easily by using ethanol as a co-solvent (8). Our previous work reported a photo-induced fiber-vesicle morphological transition in an azophenyl hydrazide derivative’s chloroform gel (12). However, full control over the morphologies of self-assembled structures for their implementation in special applications such as superhydrophobic surfaces is still a challenge.

Wettability of a solid surface is an important property for scientific research and demonstrates many practical applications. Superhydrophobic surfaces inspired by biological micro/nanostructures, characterized by water contact angles greater than 150° (14), have recently attracted much attention for their promising applications such as antisticking (15), anticontamination (16), and self-cleaning (17, 18). Compared to altering chemical composition, designing and constructing the appropriate surface roughness plays an important role for the formation of superhydrophobic surfaces (19). Various approaches, such as plasma etching and polymerization (20), chemical vapor deposition (21), laser or plasma etching processes (22), sol-gel (23), solidification (24), phase separation (25), and deposition of nanoparticles (26), have been developed. On the other hand, self-assembled morphologies of low molecular mass organogelators using hydrogen bonding, \(\pi-\pi\) interaction, and Van der Waals force have attracted increasing interest...
in this field (19, 27, 28). Due to the simplicity in their preparation and the diversity of their tunable surface properties, low molecular mass organogel represented a promising type of material for self-cleaning applications. In this paper, we report the synthesis of the gelator, N-(3,4,5-octanoxyphenyl)-N'-4-[(4-hydroxyphenyl)azophenyl] 1,3,4-oxadiazole (AOB-t8) (Scheme 1); the results indicate that its gelation property, the aggregation morphology of organogel, and wettability of the surface strongly depend on the solvents.

**Experimental Section**

**Characterization**

The $^1$H NMR spectra were recorded with an Avance-400 400 MHz spectrometer, using tetramethylsilane (TMS) as an internal standard. Field emission scanning electron microscopy (FE-SEM) images were taken with a JSM-6700F apparatus. Samples for FE-SEM measurement were prepared by wiping a small amount of gel onto a silicon plate, followed by drying in a vacuum for 12 h at room temperature. AFM images of the xerogels were obtained on an Agilent AFM 5500 (Agilent Technologies, USA) apparatus. FT-IR spectra were recorded with a Perkin-Elmer spectrometer (Spectrum One B). The xerogels were obtained by freezing and pumping the organogel of AOB-t8 for 12 h, and then pressed into a tablet with KBr for FT-IR measurement. Contact angle (CA) measurements were performed using the sessile drop method (Dataphysics, OCA 20), in which the water droplets were introduced using a microsyringe, and images were captured to measure the angle of the liquid-solid interface. Each sample was recorded at five different points.

**Gelation Test**

The weighted gelator was mixed in a cap sealed test tube (3.5 cm [height] × 0.5 cm [radius]) with an appropriate amount of solvent, and the mixture was heated until the solid dissolved. The sample vial was cooled to 4°C, and then turned upside down. When a clear or slightly opaque gel formed, the solvent therein was immobilized at this stage. Melting temperature ($T_m$) was determined by the “falling drop” method (29). An inverted gel was immersed in a water bath initially at or below room temperature, and then was heated slowly up to the point at which the gel fell due to the force of gravity, that is, the $T_m$.

**Synthesis**

The compound, N-(3,4,5-octanoxyphenyl)-N'-4-[(4-hydroxyphenyl)azophenyl] 1,3,4-oxadiazole (AOB-t8), was synthesized following the mechanism shown in Scheme 1. The hydrazine derivatives BNB-t8 were prepared by the route reported in our previous work (12). The purified BNB-t8 was dissolved in phosphorous oxychloride (POCl$_3$) and refluxed for approximately 40 h. The excess POCl$_3$ was removed through distillation and the residue was slowly added to ice-water. After removal of the solvent under reduced pressure, the final product AOB-t8 was purified by recrystallization from ethanol for further NMR, FT-IR spectroscopy, and elemental analysis.

N-(3,4,5-octanoxyphenyl)-N'-4-[(4-hydroxyphenyl)azophenyl] 1,3,4-oxadiazole (AOB-t8)

$^1$H NMR (400MHz, DMSO-$d_6$), (ppm, from TMS): 8.35 (d, 2H, $J$=8.4), 8.06 (d, 2H, $J$=8.8), 7.97 (d, 2H, $J$=8.8), 7.40 (d, 2H, $J$=9.2), 7.36 (s, 2H), 4.05-3.93 (m, 6H), 1.88-1.64 (m, 6H), 1.47-1.23 (m, 30H), 0.87-0.84 (m, 9H).

FT-IR (KBr, pellet, cm$^{-1}$): 3426, 2956, 2925, 2855, 1592, 1551, 1494, 1468, 1440, 1386, 1326, 1292, 1227, 1119, 1029, 1009, 980, 855, 839, 746, 727.

Elemental analysis: calculated for C$_{44}$H$_{62}$N$_4$O$_5$: C, 72.69%; H, 8.60%; N, 7.71. Found: C, 72.57%; H, 8.85%; N, 7.63.

**Results and Discussion**

**Molecular Design and Gelation Properties**

The strategy of this work is to build up a system containing hydrogen bonding, $\pi-\pi$ interaction, and Van der Waals force to drive the self-assembly gelation. The aforementioned interactions play mutual balance to modulate the packing arrangement
of molecules and eventually construct a particular superstructure and functional surface. For this purpose, we designed and synthesized the tapered oxadiazole derivative AOB-t8, which contains hydroxyl, azobenzene, and 1,3,4-oxadiazole moieties, and three alkyl chains (Scheme 1). The numerous interactions in AOB-t8 should offer, at least to some extent, the possibility of controlling the aggregation morphology of the organogel. It is evident that AOB-t8 has the ability to gelate a wide variety of organic solvents, such as 1,2-dichloroethane, dichloromethane, cyclohexane, ethanol, chloroform, benzene, and acetone, in a critical gelation concentration range $5.0 \times 10^{-4} - 9.8 \times 10^{-3}$ mol/L (as shown in Table 1). Therefore, compound AOB-t8 can be considered a supergelator. Moreover, the sol-gel transition is fully thermo-reversible even after several cycles of heating and cooling. The organogels are remarkably stable and can be stored for months showing no sign of decomposition.

Fig. 1 shows the gel-sol transition temperature ($T_m$) of AOB-t8 gels in benzene, dichloromethane, and acetone as a function of concentration. The $T_m$ increased until a plateau region was reached (denoted by a concentration-independent $T_m$) as the concentration increased, which were determined by the “falling drop” method (29). After changing the solvent, the value of the $T_m$ in the “plateau region” decreased from 70°C (benzene) to 59°C (acetone), and then to 41°C (dichloromethane). The most dramatic feature is that the morphologies and surface properties of xerogels formed by AOB-t8 display a strongly dependence on the nature of gelling solvent.

**Morphologies of the Xerogels**

In order to investigate the aggregation morphology of the organogels, the xerogels of AOB-t8 were prepared and subjected to scanning electron microscopy (SEM) and atomic force microscopy (AFM). As shown in Fig. 2a and 2b, xerogels of AOB-t8 from acetone and cyclohexane represented 3D cross-linking network, in which flexible wire-like fibers with the width of 70–200 nm were observed. The more entangled and dense fiber morphology was demonstrated for xerogel from cyclohexane, indicating that the interactions between individual fibers are stronger in cyclohexane. Xerogel of AOB-t8 from 1,2-dichloroethane (Fig. 2c) and ethanol (Fig. 2d) consists of twisted fibers 50–80 nm wide and tens of micrometers long. These fibers further assemble into thick fiber bundles, constituting a highly developed, entangled network. The long axis of thick fiber bundles is tilted on the substrate in the xerogel of AOB-t8 from ethanol, however it is slanted to the substrate in the xerogel from 1,2-dichloroethane. Interestingly, SEM image of AOB-t8 xerogel from dichloromethane indicated the formation of two layers (Fig. 2e). The lower one consists of flexible fibers with the width of 50–60 nm as shown in the inset of Fig. 2e; whereas, the upper one exhibits ordered honeycomb matrix with the mesh source of about 0.6 μm and the mesh aperture of about 2.5–3.5 μm, which was consistent with observations in the AFM image (Fig. S1). The average height of honeycomb matrix in Fig. S1 is 400 nm. This two-layer structure could be readily reproduced in our experiment, therefore, it was unlikely obtained by accident. As a contrast, SEM image of AOB-t8 xerogel from chloroform indicates the formation of polydisperse spherical structure, with an average diameter of about 5 nm (Fig. 2f).

On the basis of the aforementioned results, it can be concluded that the morphology of the xerogels strongly depends on the nature of the gelling solvents. The formation of elongated fiber-like and spherical aggregates indicates that the self-assembly of AOB-t8 is driven by strong intermolecular interactions.

**Interactions in the Gels**

To investigate the interactions and alkyl chain conformations in the gels, FT-IR spectroscopy on xerogels of AOB-t8 from 1,2-dichloroethane and chloroform were performed. Fig. 3 shows the comparison of IR spectra for gel and xerogel of AOB-t8. For the 1,2-dichloroethane xerogel, the hydrogen-bonded O-H stretching vibration mode was located at 3427 cm$^{-1}$, whereas the

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**Table 1.** Gelation properties$^a$ of AOB-t8.

| Solvent                  | State | CGC       | Solvent          | State | CGC       |
|--------------------------|-------|-----------|------------------|-------|-----------|
| 1,2-dichloroethane       | G     | $2.5 \times 10^{-3}$ | chloroform       | G     | $9.8 \times 10^{-3}$ |
| dichloromethane          | G     | $3.4 \times 10^{-3}$ | benzene          | G     | $5.0 \times 10^{-4}$ |
| tetrahydrofuran          | S     | —         | acetone          | G     | $4.3 \times 10^{-3}$ |
| cyclohexane              | G     | $3.8 \times 10^{-3}$ | acetonitrile     | P     | —         |
| ethanol                  | G     | $5.7 \times 10^{-3}$ | DMSO             | S     | —         |

$^a$G: stable gel formed at room temperature; P: precipitate; S: solution; CGC: critical gelation concentration (mol/L), the minimum concentration necessary for gelation of solvents.
peak shifted to slightly higher wavenumbers of 3459 cm$^{-1}$ in the related gel. This shift suggests that the hydrogen bonds in the gel are somewhat more disordered than that in the solid state (30).

On the other hand, the $\nu_{s}(\text{CH}_2)$ and $\nu_{as}(\text{CH}_2)$ in both the xerogel and the gel of AOB-t8 appeared at around 2855 and 2925 cm$^{-1}$, respectively, implying that the alkyl chains are closely packed to form quasicrystalline domains (31). It is worth pointing out that there is no appreciable difference between IR spectra given by the xerogel from different gelling solvents. Electronic UV-vis absorption spectra of the gels were studied to obtain information about the aggregated state of azobenzene on the molecular scale. With the concentration of AOB-t8 in 1,2-dichloroethane of $2.5 \times 10^{-3}$ M, the $\pi-\pi^{*}$ absorption maximum of the azobenzene group of AOB-t8 (Fig. 4) was located at 354 nm in the sol state at 50$^\circ$C, which red-shifted to 359 nm in the gel state in ethanol at 5$^\circ$C. This indicates that the azobenzene units were arranged into J-type aggregates through $\pi-\pi$ interactions in gels (32).

According to the obtained UV-vis and IR data, it is reasonable to suggest that the aggregates of AOB-t8 in the gel and xerogel states do not significantly depend on the nature of gelling solvent. The supermolecular structure of the gel may result from the combined effects of hydrogen bonds, aggregation of azobenzene moieties, and Van der Waals force.

![Fig. 2. SEM images of AOB-t8 xerogels from (a) acetone ($4.5 \times 10^{-3}$ M), (b) cyclohexane ($4.0 \times 10^{-3}$ M), (c) 1,2-dichloroethane ($2.5 \times 10^{-3}$ M), (d) ethanol ($6.0 \times 10^{-3}$ M), (e) dichloromethane ($3.5 \times 10^{-3}$ M), and (f) chloroform ($9.8 \times 10^{-3}$ M).]
Gelling Solvent Dependence of Surface Wettabilities

The wettabilities of 1,2-dichloroethane, dichloromethane, and chloroform xerogels were characterized by measurements of water contact angle on their surface. The organogels were drop-casting on silicon plate, and dried in a vacuum for 12 h at room temperature. The shapes of a water droplet sitting on xerogel films obtained from different gelling solvents are shown in Fig. 5. The static contact angle of water on the thin film of 1,2-dichloroethane xerogel was measured to be 151.3° (Fig. 5c). In contrast, the thin film of dichloromethane xerogel manifested a smooth layer with a low surface roughness (Fig. 2e), and exhibited a static contact angle of only 95.2 (Fig. 5a), even though they were formed by the same chemical structure. Similarly, the static contact angle of water on the thin film of chloroform xerogel showing the formation of spherical structure was measured to be 113.5° (Fig. 5b). Roughness enhancement of the surface is also an important factor for hydrophobicity according to the Wenzel’s equation (33). SEM images suggest a much rougher, more corrugated surface of the 1,2-dichloroethane xerogel than that of the dichloromethane xerogel. In the case of a rough surface, air trapped in the cracks of the ball also prevents the contact of water droplet with the layer surface. The fraction of air/water interface is calculated to be as high as 0.87 according to modified Cassie’s equation (34), which indicates that high hydrophobic xerogel film is mainly a result of specific surface topographic structure.

It is noteworthy, compared with other methods used for preparation of superhydrophobic surface, that the unique structure and property in this work were readily obtained by means of an easy gelation process. In addition, the wettability could be switched by simple immersion of a given xerogel film in a different solvent and drying again. For AOB-t8, after the 1,2-dichloroethane xerogel was dipped in dichloromethane and heated above 40°C, the xerogel first converted to a sol; subsequently, the sol turned to a dichloromethane gel, which finally turned to a dichloromethane xerogel after cooling to room temperature and drying. Furthermore, the reversible switching was repeated several times from the 1,2-dichloroethane xerogel to the dichloromethane one, and a good reversibility of surface wettability was observed (Fig. 6) in our experiments. This observation clearly demonstrated that the surface wettability of the xerogel can be easily modified by changing the gelling solvent.

The aforementioned results indicate that the cooperation of hydrogen bonds, \(\pi-\pi\) interaction, and Van der Waals force plays an important role in self-assembly. The gelation of AOB-t8 provides an easy method for preparation of multidimensional structure and manipulation of morphology ranging from particle fiber to even 3D net-like structure in different solvents. The great super-hydrophobicity and wettability of AOB-t8 gel can be tuned effectively simply by changing solvents in the gelation process, which is of significance for potential applications of this molecule.

Conclusion

We have successfully prepared a gelator (AOB-t8) containing hydroxyl, azobenzene, and 1,3,4-oxadiazole moieties, and three alkyl chains. AOB-t8 showed strong gelation ability in a wide variety of organic solvents, such as 1,2-dichloroethane, dichloromethane, ethanol, chloroform, benzene, acetone, and so on. The minimum gel concentration of AOB-t8 in benzene is as low as 5.0 × 10^{-4} M, which can be considered as a supergelator. It has also been demonstrated that the morphology of the xerogels strongly depends on the nature of the gelling solvents, which in turn affects the wettability of the gel surface. Based on these observations, the cooperation of hydrogen bonds, \(\pi-\pi\) interaction, and Van der Waals force might be crucially involved in the process of self-assembly. Lastly, the unique and tunable surface properties demonstrated by AOB-t8 distinguish organogel as a novel class of functional materials.

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Fig. 5. Photographs of the water droplet on silicon plate drop-casting with xerogel of AOB-t8 from (a) dichloromethane \((3.5 \times 10^{-3} \text{ M})\), (b) chloroform \((9.8 \times 10^{-3} \text{ M})\) and (c) 1,2-dichloroethane \((2.5 \times 10^{-3} \text{ M})\).

Fig. 6. Reversible wettability transition between dichloromethane and 1,2-dichloroethane gel by the sol-gel process.

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Supplemental Material

AFM image of AOB-t8 xerogel prepared from dichloromethane \((3.5 \times 10^{-3} \text{ M})\); original larger SEM images of Fig. 2a–f. Supplemental data for this article can be accessed on the publisher’s website.

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