

Research Article

Characterization of Binary Organogels Based on Some Azobenzene Compounds and Alkyloxybenzoic Acids with Different Chain Lengths

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Received 12 May 2014; Accepted 4 June 2014; Published 10 August 2014

Academic Editor: Xinqing Chen

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In this work the gelation behaviors of binary organogels composed of azobenzene amino derivatives and alkyloxybenzoic acids with different lengths of alkyl chains in various organic solvents were investigated and characterized. The corresponding gelation behaviors in 20 solvents were characterized and shown as new binary organic systems. It showed that the lengths of substituent alkyl chains in compounds have played an important role in the gelation formation of gelator mixtures in present tested organic solvents. Longer methylene chains in molecular skeletons in these gelators seem more suitable for the gelation of present solvents. Morphological characterization showed that these gelator molecules have the tendency to self-assemble into various aggregates from lamella, wrinkle, and belt to dot with change of solvents and gelator mixtures. Spectral characterization demonstrated different H-bond formation and hydrophobic force existing in gels, depending on different substituent chains in molecular skeletons. Meanwhile, these organogels can self-assemble to form monomolecular or multilayer nanostructures owing to the different lengths of due to alkyl substituent chains. Possible assembly modes for present xerogels were proposed. The present investigation is perspective to provide new clues for the design of new nanomaterials and functional textile materials with special microstructures.

1. Introduction

In recent years, organogels have been attracting more attention as one kind of special functional materials, in which various organic solvents are immobilized by different gelators [1–4]. Previously gels are widely found in polymer systems; however, there has recently been more increasing attention in low-molecular-mass organic gelators (LMOGs) [5–9]. In recent years, organized gelation of organic solvents by LMOGs has been reported as one of the hot areas in the soft matter and organized materials fields due to their scientific values and many potential applications in the biomedical field, including drug delivery, tissue engineering, and medical implants [10–14]. The gels based on LMOGs are commonly regarded as supramolecular gels, in which the

gelator molecules can self-assemble in an orderly manner and form three-dimensional nanostructures in which the solvent is captured via various noncovalent interactions, such as π - π stacking, hydrogen bonding, van der Waals interaction, coordination, solvophobic interaction, dipole-dipole interaction, and host-guest interaction [15–17]. LMOGs have some advantages over polymer gels: the molecular structure of the gelator is defined and the gel process is usually reversible. Such properties make it possible and reasonable to design various functionalized assembly systems and produce more complicated and/or controllable nanocomposites and nanostructures [18–20].

In our previous reported research work, the gelation properties of some cholesterol imide compounds with cholesteryl units and azobenzene substituent headgroups

have been characterized and investigated [21]. We found that the change in azobenzene headgroup can produce an obvious change in the gelation formation and assembly modes of these compounds. Furthermore, another gels system based on some bolaform and trigonal cholesteryl compounds with different molecular skeletons have been characterized [22]. In this paper, we have characterized the spacer effect on the gel formation and nanostructures of such organogels and found that different kinds of hydrogen bond interactions among the molecules play a crucial role in the self-assembly process.

As a subsequent research work, now, we have prepared new binary organogels composed of aminoazobenzene derivatives and alkyloxybenzoic acids with different lengths of alkyl chains. We have found that some of present mixtures could form various organogels in different organic solvents. Morphological investigation of present organogels indicated the formation of different nanostructures of the aggregates in the gels. In addition, we have characterized the effect of substituent methylene chains in gelators on the nanostructures of present organogel systems and found various kinds of self-assembly modes. This report may afford a specific approach to understanding the soft matter and design of new nanomaterials and functional textile materials.

2. Experiments

2.1. Materials and Reagents. 4-Aminoazobenzene and 2-aminoazotoluene were purchased from Alfa Aesar Tianjin Chemicals and TCI Shanghai Chemicals, respectively. Other used reagents with analysis purity were obtained from Beijing Chemicals and distilled before use. Deionized water was used in all process. 4-n-Alkyloxybenzoic acids with different substituent chain lengths (carbon numbers 18, 16, 14, and 12) were prepared in similar method according to previous paper [23] and confirmed by ^1H NMR.

2.2. Gelation Test. All mixed organogels were prepared according to a simple procedure. Firstly, these alkyloxybenzoic Acids and amine compounds were mixed with 1:1 molar ratio according to the number matching of intermolecular carboxylic acid and amine group, respectively. Then, a fixed amount of binary mixtures and a volume of organic solvent were taken into a sealed glass bottle and ultrasonically dispersed evenly. Then the solution was heated in a water bath at 70°C for 20 min. After that, the solution was cooled to room temperature in air and the test bottle was inversed to see if a gel was formed. If the binary mixture formed a gel by immobilizing the solvent at this stage, it was designated as a gel system "G." For the systems in which only solution obtained until the end of the tests, they were denoted by solution (S). When the binary mixtures formed into a few precipitates in some solvent, it was denoted as a "PS." Critical gelation concentration (CGC) refers to the minimum concentration of the gelator for gel formation.

2.3. Characterization Techniques. Firstly, these xerogels were obtained by a vacuum pump for 12–24 h from the as-formed gels under the critical gelation concentration. Then the dried samples were attached to different substrates, such as copper

plates, glass slices, and CaF_2 slice for morphological and spectral investigation, respectively. Before SEM measurement, the xerogels were coated on copper plates fixed by conductive adhesive tape and shielded by gold. SEM images of the xerogels were obtained from a Hitachi S-4800 field emission scanning electron microscopy with the accelerating voltage of 5–15 kV. Transmission FT-IR spectra of the xerogels were measured by Nicolet is/10 FT-IR spectrophotometer from Thermo Fisher Scientific Inc. by average 32 scans with a resolution of 4 cm^{-1} . The XRD patterns were measured by a Rigaku D/max 2550PC diffractometer (Rigaku Inc., Tokyo, Japan). The curves were obtained by $\text{CuK}\alpha$ radiation with an incident wavelength of 0.1542 nm under a voltage of 40 kV and a current of 200 mA with scan rate of $0.5^\circ/\text{min}$.

3. Results and Discussions

3.1. Gelation Behaviors. Firstly, the gelation properties of all binary mixtures in 20 solvents are investigated. The experimental data showed that the binary mixtures of alkyloxybenzoic acids with different alkyl chains and 4-aminoazobenzene/2-aminoazotoluene could form organogels in special organic solvents, as listed in Table 1. The binary mixtures of alkyloxybenzoic acids with different carbon numbers (18, 16, 14, and 12) and 4-aminoazobenzene are denoted as C18-Azo, C16-Azo, C14-Azo, and C12-Azo, respectively. Similarly, the binary mixtures of these acids and 2-aminoazotoluene are denoted as C18-Azo-Me, C16-Azo-Me, C14-Azo-Me, and C12-Azo-Me, respectively. Firstly, for the mixtures containing longer alkyl chains with carbon numbers of 18 and 16, 4 and 6 kinds of organogels can be formed in different solvents, respectively. For example, C18-Azo can form gels in toluene, nitrobenzene, ethanolamine, and benzene, respectively. In addition, for the cases with shorter alkyl chains, only two and one kinds of organogels can form organogels in present solvents for mixtures with carbon numbers of 14 and 12, respectively. The organogels have been only prepared in ethanolamine for C12-Azo and C12-Azo-Me systems, respectively. Their photographs of all as-made organogels in various solvents were shown in Figure 1. The present research results indicated that length change of alkyl substituent chains can have an obvious effect upon the gel formation of present studied mixtures. This phenomenon indicated that longer methylene chains in molecular skeletons in present gelators systems are more suitable for the present mixtures, which was similar to the recent reports [23, 24]. Furthermore, it should be noted that the difference of methyl groups in azobenzene segment seemed to have no obvious effect on the regulation of gelator behaviors in present cases, which is different from previous report [21].

3.2. Morphological Investigation. It has been reported that various gelator molecules can construct nanoscale superstructures such as fibers, ribbons, and sheets in a supramolecular gel [25–27]. In order to obtain more insight into the gel nanostructures, the organized structures of these organogels were characterized by SEM technique, as shown in

TABLE 1: Gelation behaviors of these binary organogels at room temperature.

Solvents	C18-Azo	C18-Azo-Me	C16-Azo	C16-Azo-Me	C14-Azo	C14-Azo-Me	C12-Azo	C12-Azo-Me
Acetone	PS	PS	PS	PS	PS	PS	PS	PS
Aniline	PS	PS	PS	PS	PS	PS	PS	PS
n-Hexane	S	S	S	S	S	S	S	S
Toluene	G (3.0)	G (3.0)	G (2.8)	G (2.8)	S	S	S	S
Pyridine	S	S	S	S	S	S	S	S
Isopropanol	S	S	S	S	S	S	S	S
Cyclopentanone	PS	PS	PS	PS	PS	PS	PS	PS
Cyclohexanone	PS	PS	PS	PS	PS	PS	PS	PS
Nitrobenzene	G (3.0)	G (3.0)	G (2.5)	G (2.5)	G (2.5)	G (2.5)	S	S
n-Butanol	S	S	S	S	S	S	S	S
Ethanolamine	G (3.0)	G (3.0)	G (2.8)	G (2.8)	G (2.5)	G (2.5)	G (2.5)	G (2.5)
n-Butyl acrylate	PS	PS	G (2.8)	G (2.8)	PS	PS	PS	PS
1,4-Dioxane	S	S	S	S	S	S	S	S
Petroleum ether	PS	PS	PS	PS	PS	PS	PS	PS
Ethyl acetate	PS	PS	PS	PS	PS	PS	PS	PS
Chloroform	PS	PS	G (2.5)	G (2.5)	PS	PS	PS	PS
THF	S	S	S	S	S	S	S	S
DMF	S	S	S	S	S	S	S	S
DMSO	S	S	S	S	S	S	S	S
Benzene	G (3.0)	G (3.0)	G (2.8)	G (2.8)	PS	PS	S	S

DMF, dimethylformamide; THF, tetrahydrofuran; DMSO, dimethyl sulfoxide; S: solution; PS: partially soluble; and G: gel; for gels, the critical gelation concentrations at room temperature are shown in parentheses (% w/v).

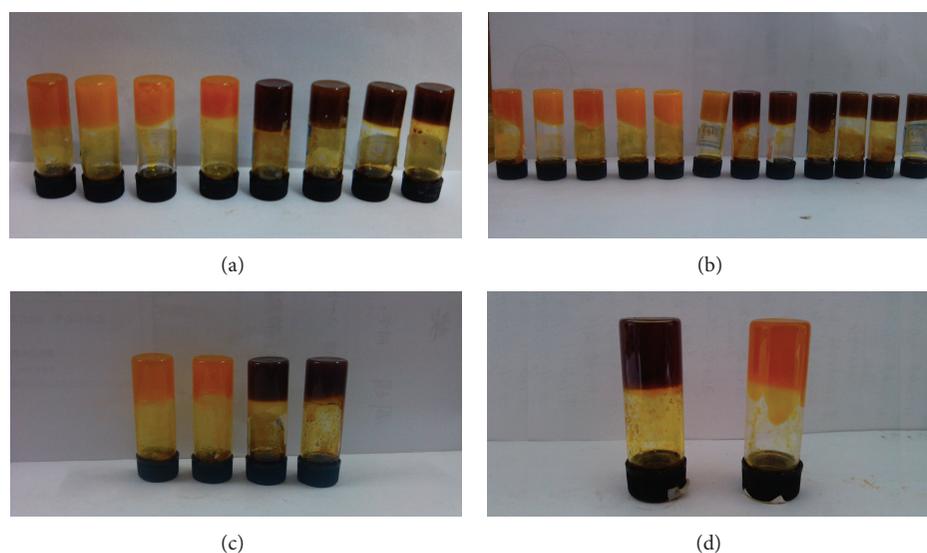


FIGURE 1: Photographs of as-made organogels: (a), C18-Azo and C18-Azo-Me; (b), C16-Azo and C16-Azo-Me; (c), C14-Azo and C14-Azo-Me; and (d), C12-Azo and C12-Azo-Me, respectively.

Figures 2, 3, and 4. From the present diverse images, it can be obviously observed that the nanostructures of all xerogels from various solvents are obviously different from each other, and the nanostructures of the aggregates change from lamella, wrinkle, and belt to dot with change of solvents and mixtures. Furthermore, more nanorod or nanobelt aggregates with different sizes were obtained in gels with longer alkyl substituent

chains. In addition, it should be noted that these belt-like aggregates showed a tendency to aggregate together due to highly directional intermolecular interactions and/or solvent evaporation. The difference of morphologies can be mainly attributed to the different strengths of the intermolecular forces, such as hydrophobic force between alkyl methylene chains and π - π stacking of azobenzene segments, which

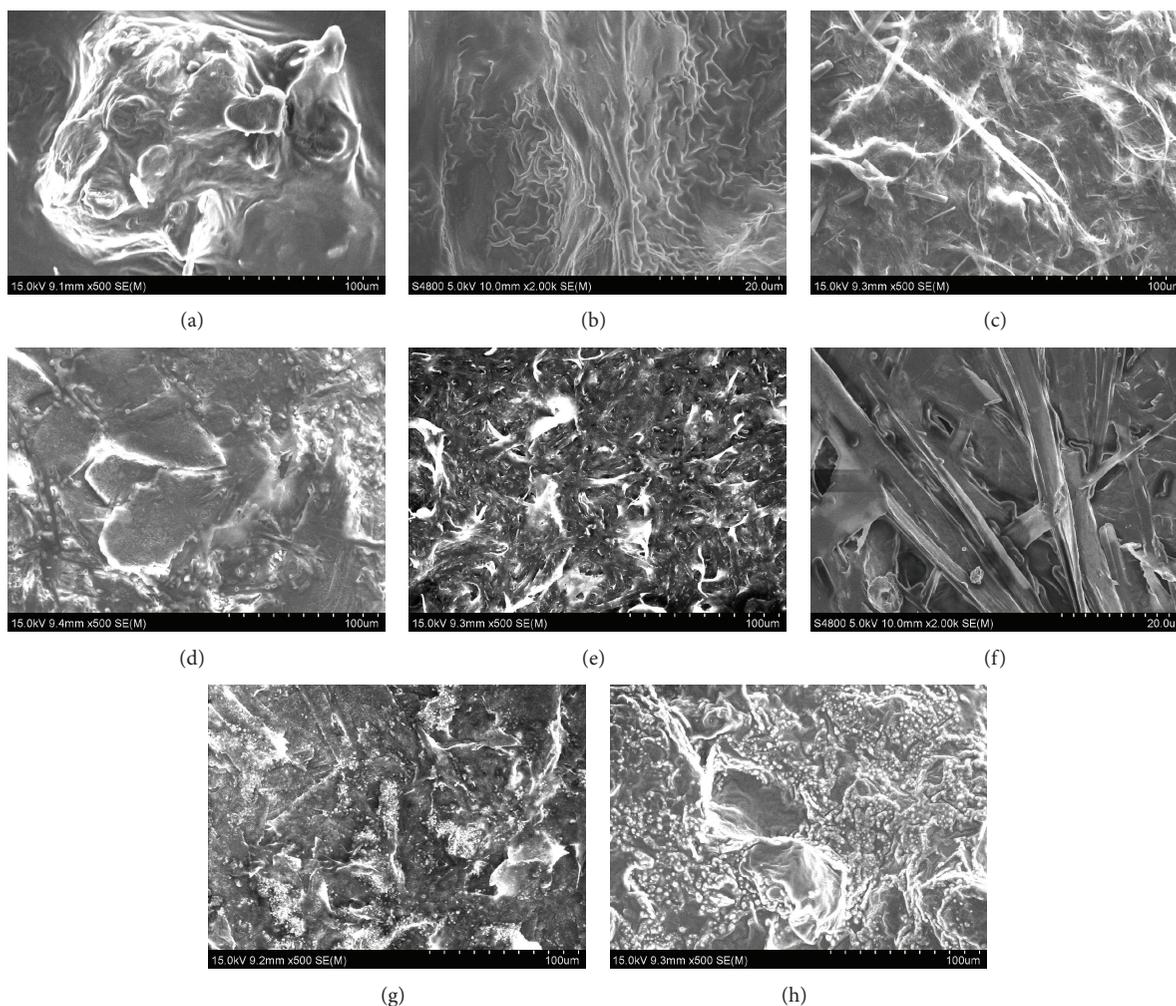


FIGURE 2: SEM images of xerogels. C18-Azo (a, b, c, and d) and C18-Azo-Me (e, f, g, and h) in toluene, nitrobenzene, ethanolamine, and benzene, respectively.

can regulate the orderly stacking and formation of organized aggregates in gel formation.

3.3. Spectral Investigation. In addition, in order to further character the organized stacking unit of xerogels nanostructures, XRD patterns of all xerogels were measured, as shown in Figure 5. Firstly, the curves of C18-Azo and C18-Azo-Me xerogels from various solvents show similar strong peaks in the angle region (2θ values, 5.44, 6.02, 9.02, 16.78, and 19.38°) corresponding to d values of 1.63, 1.47, 0.98, 0.53, and 0.46 nm, respectively. In addition, for the curves of C16-Azo and C16-Azo-Me xerogels, the minimum peaks appeared at 2θ value of 5.92°, corresponding to d value of 1.49 nm. However, as for the curves of C14-Azo and C14-Azo-Me from nitrobenzene and ethanolamine, the small 2θ values are 2.18 and 3.44°, corresponding to d values of 4.06 and 2.57 nm, respectively. Meanwhile, as for the curves of C12-Azo and C12-Azo-Me from ethanolamine, the small 2θ values are 2.50 and 3.76°, corresponding to d values of 3.54 and 2.35 nm, respectively. The difference of values between C18-Azo and C16-Azo with longer alkyl methylene chains can be mainly

attributed to the change of length in substituent groups linked to azobenzene segment in the molecular skeleton, which affected the assembly modes in the 3D stacking of organogels [28]. At the same time, for these xerogels from gelators with shorter alkyl chains, the changed d values indicated organized multilayer formation in the self-assembly of organogels. The XRD results mentioned above demonstrated that the length of substituent alkyl chain in molecular skeletons had obvious effect on the assembly modes of these gelator mixtures.

It is reported that hydrogen bonding can be used to monitor the self-assembly process of organogels [29]. In present case, in order to investigate the effect of many factors on assembly, the FT-IR spectra of all xerogels were measured, as shown in Figure 6. Firstly, C18-Azo xerogel was taken as an example, as shown in Figure 6(a). Some main characteristic peaks were observed at 3374, 2917, 2848, 1705, 1602, and 1471 cm^{-1} , respectively, which could be assigned to the N-H and O-H stretching, methylene stretching, carbonyl group band, amide I band, and methylene scissoring, respectively [30, 31]. These bands indicated that the formation of hydrogen bonding interactions between intermolecular amino and

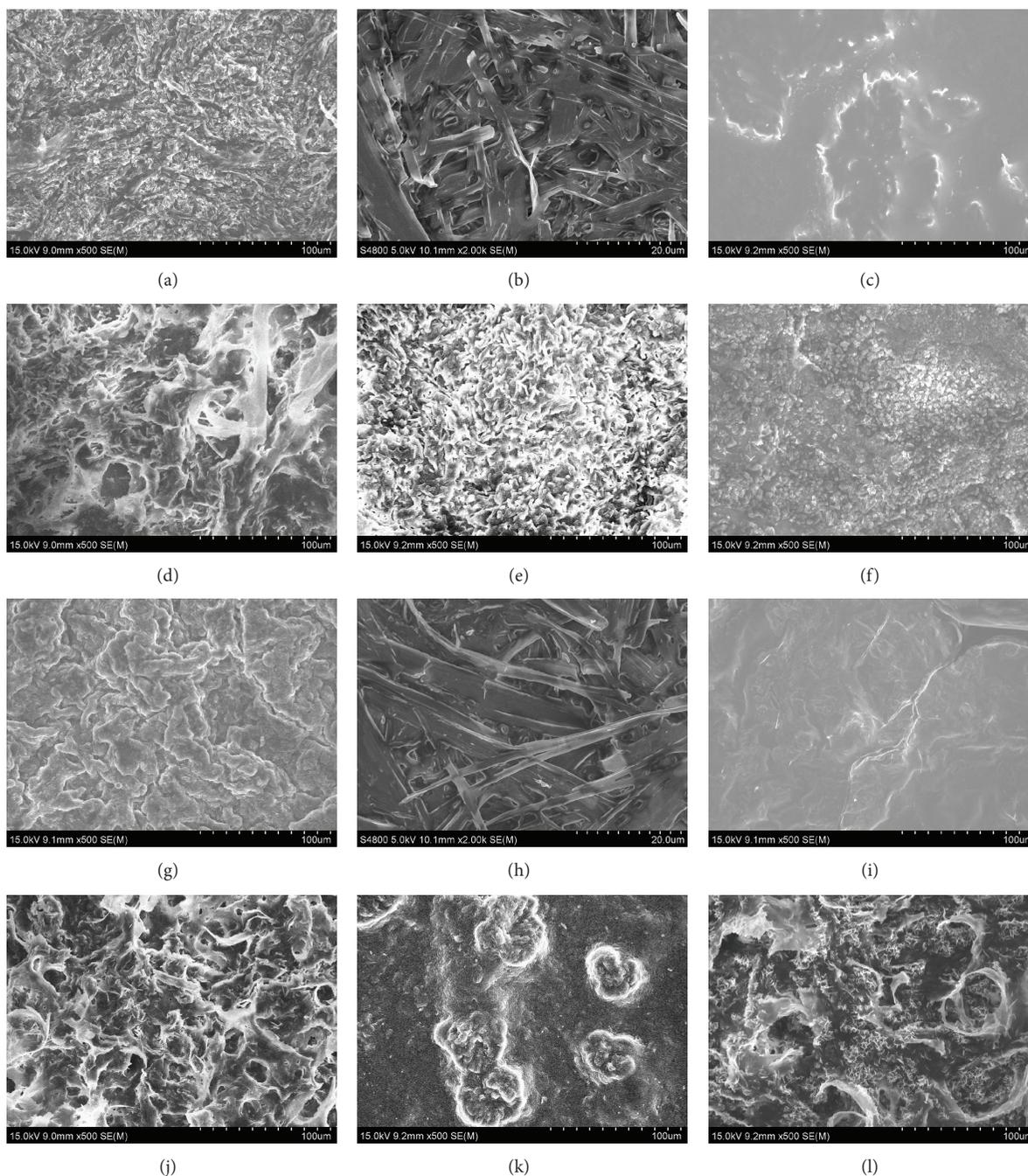


FIGURE 3: SEM images of xerogels. Cl6-Azo (a, b, c, d, e, and f) and Cl6-Azo-Me (g, h, i, j, k, and l) in toluene, nitrobenzene, ethanolamine, n-butyl acrylate, chloroform, and benzene, respectively.

carboxylic acid groups in the gel state can regulate the self-assembly modes of the gelator molecules to stack in ordered nanostructures. Similar spectra were observed for other xerogels.

3.4. Discussion of Assembly Modes. Considering the XRD results described above and the hydrogen bonding nature of these binary mixtures as confirmed by FT-IR measurements, the possible assembly modes for present xerogels were proposed and schematically shown in Figure 7. As for xerogel of Cl8-Azo and Cl6-Azo containing longer alkyl

chains, due to the strong intermolecular hydrophobic force between substituent alkyl chains and hydrogen bonding interaction between amino and carboxylic acid groups, these gelators can self-assemble to form orderly nanostructures. The calculated repeating unit with length of about 1.6~1.7 nm was obtained. The obtained experimental values were 1.63 and 1.49 nm for gels of Cl8-Azo and Cl6-Azo, respectively, which was in well accordance with the calculation result. In addition, for the xerogels of Cl4-Azo and Cl2-Azo with shorter alkyl chains, with the decrement of alkyl substituent chains, the weaker intermolecular hydrophobic

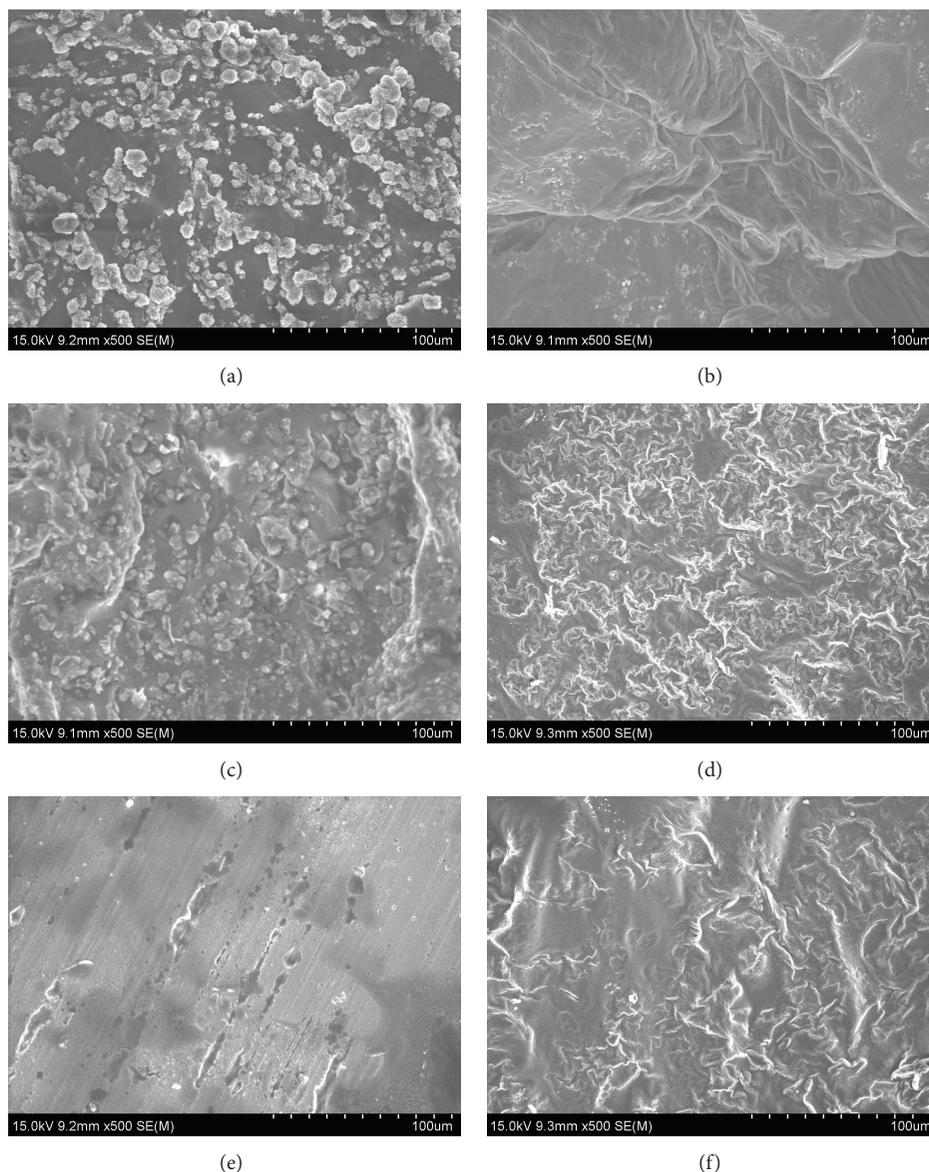


FIGURE 4: SEM images of xerogels. C14-Azo (a and b) and C14-Azo-Me (c and d) in nitrobenzene and ethanolamine, respectively; C12-Azo (e) and C12-Azo-Me (f) in ethanolamine, respectively.

force between the alkyl chains of the neighboring molecules will not enable present gelators to orderly assemble as those cases with longer chains and show a tendency to self-assemble to form multilayer structures and more disorderly stacking unit. Thus, the obtained experimental values were double or multilayer values compared to monomolecular lengths of C14-Azo and C12-Azo. Meanwhile, it should be noted that this phenomena is similar to the results of recent reports [21, 24, 30]. Therein, the substituent groups in azobenzene residue, luminol segment, or benzimidazole/benzothiazole imide compounds can have an obvious effect upon the gel formation, self-assembly, and the as-formed nanostructures of the studied compounds. For the present system, the experimental results also demonstrated that the substituent methylene chains had played a very crucial role in changing the assembly modes and nanostructures in these organogels.

4. Conclusion

The gelation behaviors of binary organogels composed of azobenzene amino derivatives and alkyloxybenzoic acids with different lengths of alkyl chains in various organic solvents were investigated. The experimental results indicated that their gelation behaviors could be changed by varying the lengths of substituent methylene chains. Longer alkyl methylene chains in molecular skeletons in present gelation systems are suitable for the gel formation and self-assembly of organic solvents. For the mixtures containing longer alkyl chains with carbon numbers of 18 and 16, 4 and 6 kinds of organogels can be formed in different solvents, respectively. In addition, for the cases with shorter alkyl chains, only two and one kinds of organogels can form organogels in present solvents for mixtures with carbon numbers of 14 and 12,

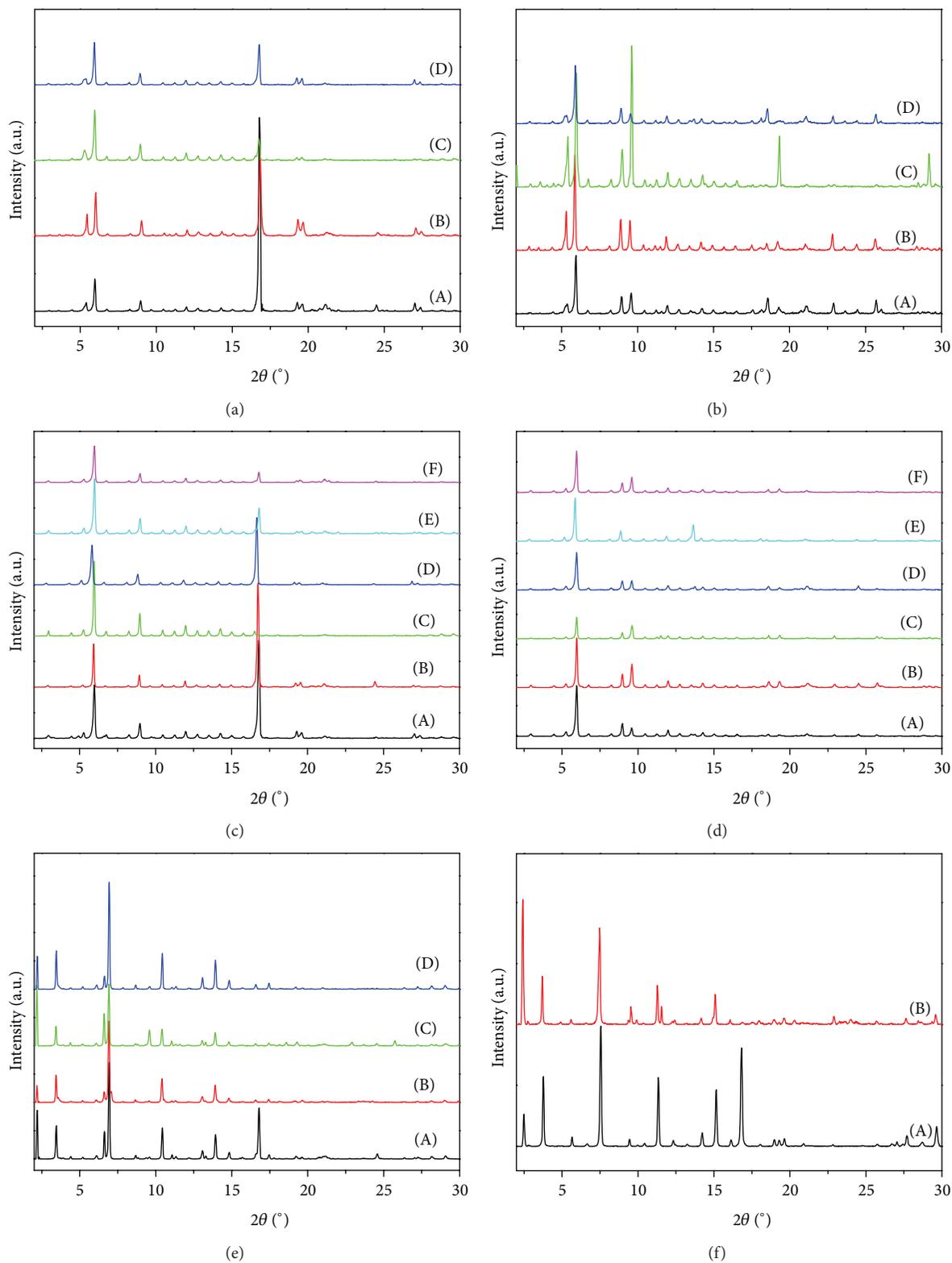


FIGURE 5: X-ray diffraction patterns of xerogels. C18-Azo (A) and C18-Azo-Me (B) in toluene (a), nitrobenzene (b), ethanolamine (c), and benzene (d), respectively; C16-Azo (C) and C16-Azo-Me (D) in toluene (a), nitrobenzene (b), ethanolamine (c), n-butyl acrylate (d), chloroform (e), and benzene (f), respectively; C14-Azo and C14-Azo-Me (E) in nitrobenzene (a and c) and ethanolamine (b and d), respectively; C12-Azo and C12-Azo-Me (F) in ethanolamine (a and b), respectively.

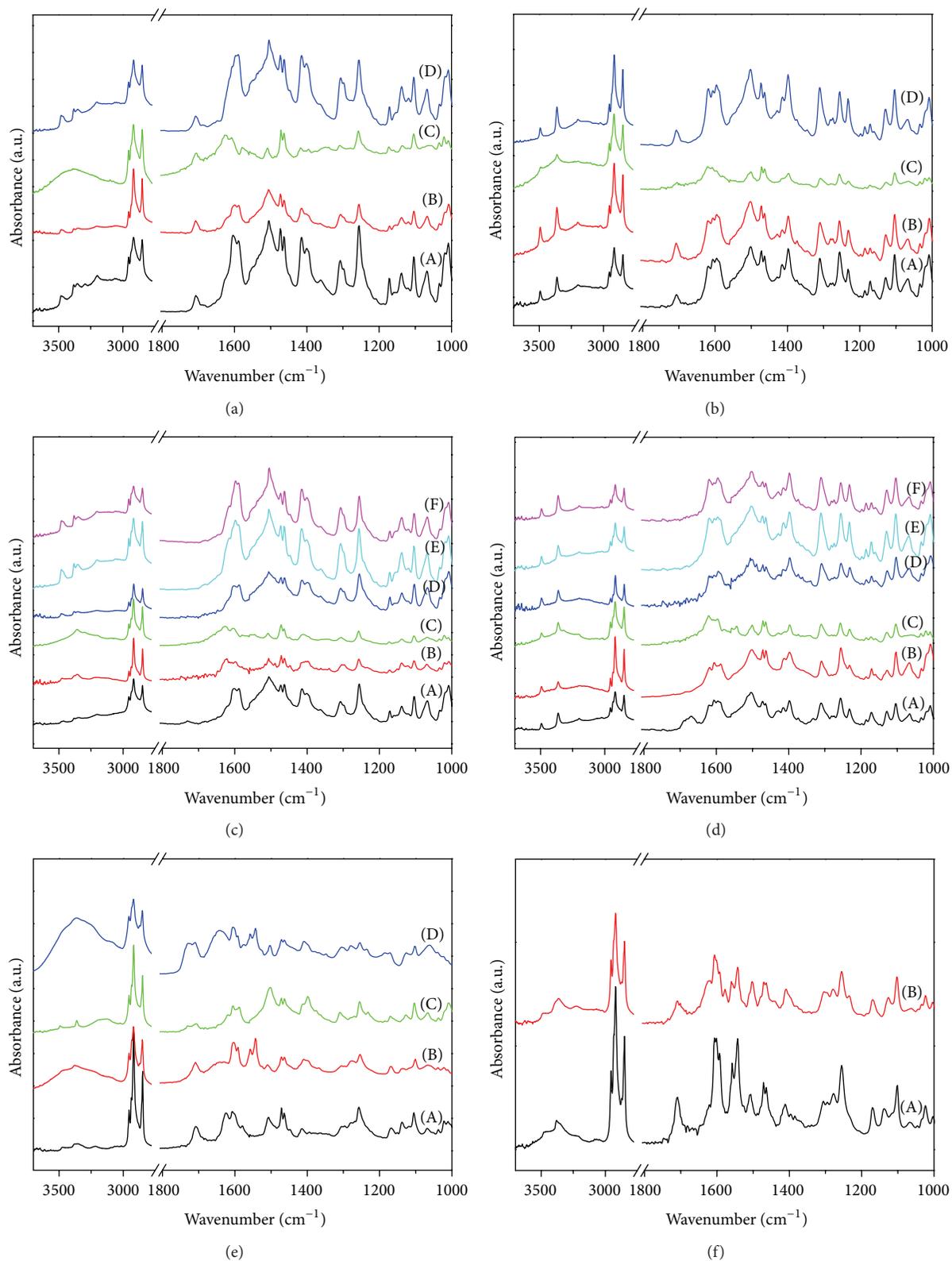


FIGURE 6: FT-IR spectra of xerogels. C18-Azo (A) and C18-Azo-Me (B) in toluene (a), nitrobenzene (b), ethanolamine (c), and benzene (d), respectively; C16-Azo (C) and C16-Azo-Me (D) in toluene (a), nitrobenzene (b), ethanolamine (c), n-butyl acrylate (d), chloroform (e), and benzene (f), respectively; C14-Azo and C14-Azo-Me (E) in nitrobenzene (a and c) and ethanolamine (b and d), respectively; C12-Azo and C12-Azo-Me (F) in ethanolamine (a and b), respectively.

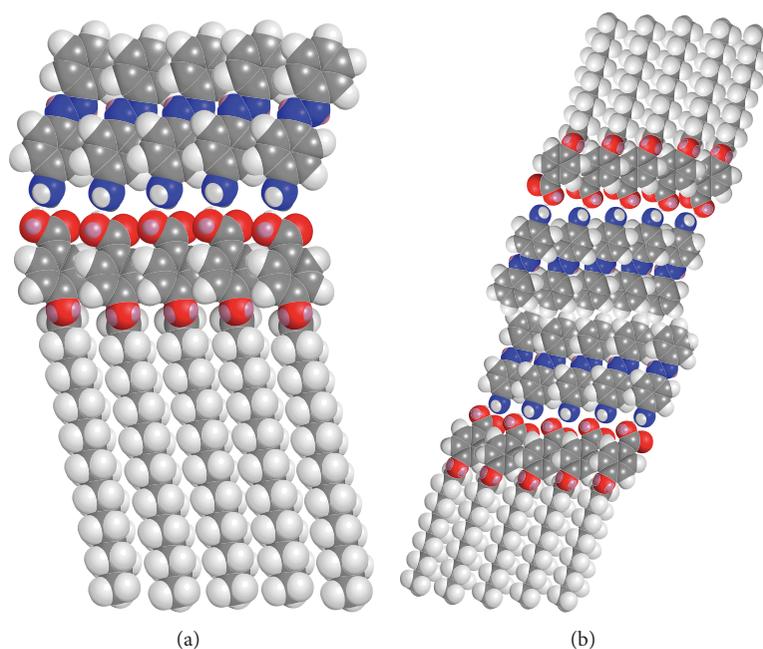


FIGURE 7: Schematic assembly modes for organogels of C18-Azo (a) and C12-Azo (b).

respectively. Morphological characterization indicated that the gelator molecules could organize in orderly stacking and self-assemble into different nanostructures from lamella, wrinkle, and belt to dot with change of solvents and gelator mixtures. Spectral characterization indicated that different H-bond formation and hydrophobic force could be obtained depending on different substituent chains in molecular skeletons. Meanwhile, these organogels can self-assemble to form monomolecular or multilayer nanostructures owing to the different lengths of due to alkyl substituent chains. Possible assembly modes for present xerogels were proposed. The prepared nanostructured materials have wide perspectives and many potential applications in nanoscience and material fields due to their scientific values. The present investigation is perspective to provide new clues for the design of new nanomaterials and functional textile materials with special microstructures.

Conflict of Interests

The authors declare that they have no direct financial relations with the commercial identities mentioned in this paper that might lead to a conflict of interests for any of the authors.

Acknowledgments

This work was financially supported by the Natural Science Foundation of Hebei Province (no. B2013203108), the Science Foundation for the Excellent Youth Scholars from Universities and Colleges of Hebei Province (no. YQ2013026), and the Support Program for the Top Young Talents of Hebei Province.

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