



# Sonication induced morphological transformation between 3D gel network and globular structure in a two-component gelation system



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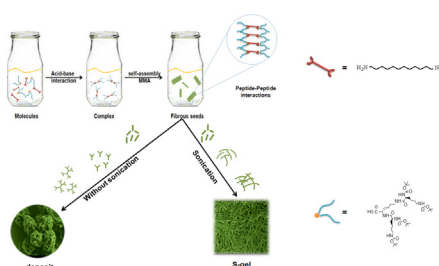
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## HIGHLIGHTS

- Two-component gelator based on the interaction between dendritic branch and diaminododecane.
- The exchange of morphology between a 3D gel network and globular structure.
- Self-assembly mechanism of two-component gelator.

## GRAPHICAL ABSTRACT

Schematic diagram of the ultrasonic effects on the gelation process.



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## ABSTRACT

A two-component gelator (B1...C12...B1) based on interaction between dendritic branch (Branch1 (COOH)) and diaminododecane is designed and synthesized, and its self-assembly behavior in methyl methacrylate (MMA) is firstly investigated. It is interesting to find that the gel formation depends on the concentration and sonication during cooling of the B1...C12...B1/MMA solution. Sonication plays as a switch simulator for exchanging morphology between a 3D gel network and globular structure in a certain concentration range. Thermal stability, rheological properties, morphology and self-assembly mechanism of the gels were investigated with tube inversion methodology, Dynamic oscillatory measurement, SEM and FTIR, respectively. The results indicated that Sonication-induced-gel (S-gel) exhibits much higher gel-sol temperature, storage modulus and lower critical gelator concentration compared with Temperature-induced-gel (T-gel), while sonication could partially destroy the hydrogen bond network and benefit the formation of the organogels.

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## 1. Introduction

There has been a surge of interest in low-molecular-weight gelators (LMWGs), a family of organic molecules that can self-assemble into a 3D gel network in organic solvent through intra or intermolecular non-covalent interaction including hydrogen bonding, Vanderwaals force,  $\pi$ - $\pi$  stacking and charge transfer interaction [1–6]. The subject of gels have attracted considerable attention

because of their special properties and potential applications in field such as drug delivery, catalysis, cosmetics, food, tissue engineering, textile, the recovery of spilled crude oil and lubrication [7–12]. In order to obtain suitable organogels, numerous studies have been carried out over the past several decades [13–15]. Among those studies, development of smart or adaptive gels which can be manipulated or switched by external responsive stimulus is very still a challenge.

Stimuli-responsive gels have been attracted widespread attention in are as ranging from chemistry and biology to materials science because of their potential applications in sensors, actuators, shape memories, drug delivery, hydrophilicity/hydrophobicity

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modulation and so on [16–20]. It is generally accepted that external stimuli such as light, electric, temperature and PH have a great effect on switching of molecular aggregations involved in the formation of gel, micelles, vesicles and membranes [21–24]. However, a simple physical method of controlling that provides not only a reversible switching of original molecular aggregation but also the kinetic control of the hierarchical assembly of molecules remains a challenge. Recently, sonication as one of the external stimulations has begun to play a significant role in the organogel field [25–27]. In general, sonication is usually used to disrupt the weak non-covalent interactions or to disintegrate aggregated particles but seldom to be in favor of the assembly formation [33–37]. But recent reports showed that sonication could act as a stimulus to induce gelation of organic liquids with LMWGs. The Naota and Zhang groups independently observed sonication-induced gelation in hydrogen-bonded gelators for the first time in 2005 [28,29]. More recently, Sijbesma and associates reported that sonication could cause rheology switching and conformational changes, subsequently induced supramolecular self-assembly leading to gelation of the solvent [30,31]. Furthermore, Bardelang and associates have shown that sonication may reshape sheet-like dipeptide particles into elongated molecular assemblies and that sonocrystallisation is at the origin of gelation [32]. Although the studies on the sonication-induced reversible gelation have been reported stage by stage, effective manipulation of the morphology change in a gel system by sonication still remains rarely.

In our present work, our research group has designed and synthesized a two-component gelator (B1...C12...B1) based on interaction between dendritic branch (Branch1 (COOH)) and diaminododecane and firstly investigated the influence of ultrasonic stimulation for their self-assembly behavior in MMA. It is interesting to find that, during cooling of the B1...C12...B1/MMA solution in a certain concentration range, gelation is observed exclusively when sonication is used as an external stimulus, while

deposit is obtained without sonication. The SEM results confirmed that B1...C12...B1 deposit from MMA resulting in 25–45  $\mu\text{m}$  globular structure, while B1...C12...B1 xerogel from MMA under sonication leads to the gel network among 50–150 nm formation. The results of tube inversion methodology, Dynamic oscillatory measurement and Fourier transform infrared (FTIR) indicated that sonication acting as a simple physical stimulus could significant changes the self-assembly properties of gels. This paper provides a new way for the exchanging morphology between a 3D gel network and globular structure, which are consequently able to achieve the supramolecular functional materials with controllable macroscopic properties.

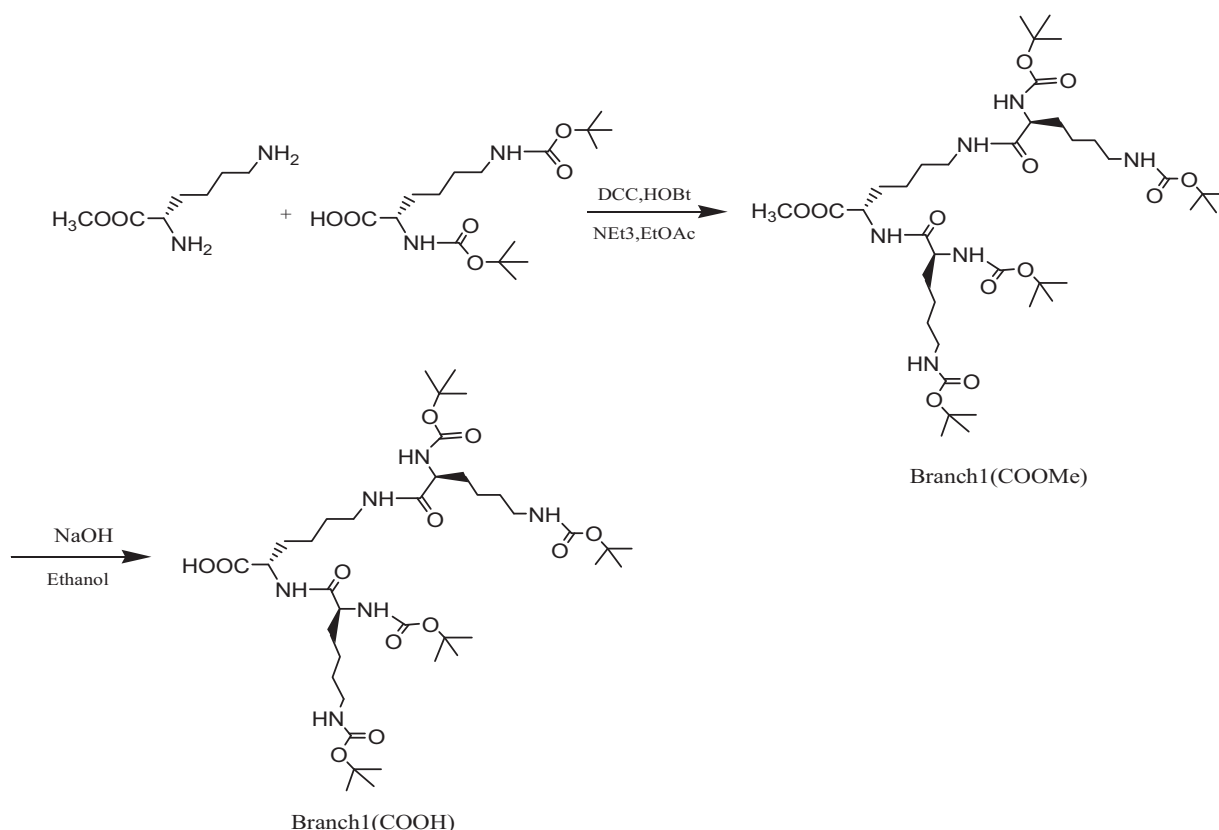
## 2. Materials and methods

### 2.1. Materials

L-Lysine methyl ester dihydrochloride (98%, Aladdin, China), Boc-protected L-lysine (99%, Aladdin, China), 1,3-dicyclohexyl carbodiimide (DCC, 99%, Shanghai Covalent Chemical Technology Co., Ltd., China), 1,12-diaminododecane (98%, Aladdin, China), 1-hydroxybenzo-triazole (HOBt, 99%, Shanghai Covalent Chemical Technology Co., Ltd., China), triethylamine ( $\text{NEt}_3$ , Aladdin, China), sodium hydroxide ( $\text{NaOH}$ , 96%, Aladdin, China) were used as received. Solvents including dichloromethane, methanol, ethanol, petroleum ether (PE) and ethyl acetate ( $\text{EtOAc}$ ) (AR grade, Aladdin China) were used as received. MMA (99%, Aladdin, China).

### 2.2. Syntheses of compounds

The L-lysine-based dendritic branch about Branch1 (COOH) was synthesized using standard amide coupling methodology (Scheme 1) according to Smith and co-workers [33]. Boc-protected



**Scheme 1.** Synthetic route and molecular structure of Branch1 (COOH).

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