

Design and Characterization of a Novel Arabinose-Derived Organogelator: A Study on Gelation Behavior and Structural Properties

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Abstract

Organogels formed by low molecular weight gelators (LMWGs) offer immense potential for biomedical and material science applications. Carbohydrate-based gelators are of special interest due to their biocompatibility and structural versatility. This study aims to synthesize a novel triazole-modified pentose sugar derivative and evaluate its gelation behavior, microstructure, and mechanical properties. The synthesis involved acetylation, bromination, azidation, and click chemistry (CuAAC) steps starting from D-arabinose. Structural characterization was performed using FT-IR, ¹H-NMR, ¹³C-NMR, and XRD. Gelation properties were evaluated in various organic solvents, and the resulting gels were studied using field emission scanning electron microscopy (FESEM) and atomic force microscopy (AFM). Rheological analysis was performed to assess the mechanical strength. The synthesized triazole derivative displayed efficient gelation in aromatic solvents and oils at low concentrations. FESEM and AFM revealed a dense fibrillar network. XRD analysis confirmed lamellar molecular packing, and FT-IR suggested extensive hydrogen bonding and π - π stacking interactions in the gel phase. The novel arabinose-derived organogelator demonstrated excellent gel-forming capability and robust mechanical properties, opening avenues for its application in biomedical formulations and soft material engineering.

Keywords: Organogelator, Pentose sugar, Self-assembly, Triazole, Gelation, Carbohydrate chemistry

1. Introduction

Organogels are semi-solid materials formed through the immobilization of organic solvents within a three-dimensional network created by the self-assembly of low molecular weight gelators (LMWGs). Carbohydrate-based gelators are highly sought after due to their natural abundance, eco-friendliness, and ease of chemical functionalization. Organogels are versatile materials with a wide range of physical properties, making them suitable for diverse applications such as drug delivery, tissue engineering, sensors, and nanotechnology. These gels are formed when solvent molecules become entrapped within a three-dimensional network created by the self-assembly of organogelators. This network structure effectively immobilizes the solvent, endowing the material with several advantageous characteristics. Notably, organogels exhibit viscoelastic properties, enabling them to maintain their shape under mechanical stress and, in certain cases, respond to external stimuli. Organogelators themselves are amphiphilic in nature, possessing both hydrophilic and hydrophobic moieties, which facilitate their self-assembly. Depending on the chemical structure and functional groups of the gelators, organogels can be classified into several common types.

Low-molecular-weight gelators (LMWGs), also referred to as small organic compounds, play a key role in organogel formation. Examples of LMWGs include fatty acids, short peptides, and various low-molecular-weight organic molecules. In addition to small molecules, polymeric materials can also serve as effective gelators for organogel formation. Polymers such as block copolymers, dendrimers, and other macromolecular structures offer unique properties to the resulting gels, often enhancing their mechanical strength, responsiveness, and functionality. The introduction of rigid groups such as triazoles into carbohydrate frameworks can enhance the ability of molecules to form ordered assemblies via hydrogen bonding, π - π stacking, and van der Waals interactions. Despite extensive research on hexose-based gelators, pentose sugars like arabinose have not been widely explored for organogelator applications. This study presents the design, synthesis, and detailed physicochemical characterization of a novel arabinose-derived triazole gelator. The aim is to investigate its gelation behavior in various solvents and understand the underlying structural organization using advanced analytical techniques. Agar-agar is a natural gelling agent extracted from the cell walls of red algae species, primarily *Gelidium* and *Gracilaria*. It dissolves when heated in water and forms a gel upon cooling. Agar appears as a translucent white substance with no flavor or odor. Rich in dietary fiber, agar can aid digestion and is widely used in culinary and pharmaceutical applications. Notably, agar has a higher melting temperature than gelatin, around 37°C (99°F).

Alginate, another naturally occurring polysaccharide, is extracted from the cell walls of brown algae (family *Phaeophyceae*). Alginate forms strong gels when combined with specific cations, particularly calcium ions. Even without gelation, alginate solutions can thicken liquids effectively. It is biodegradable, considered safe for human consumption, and is utilized to create edible capsules for dietary supplements and controlled drug release systems. Additionally, alginate dressings are employed in wound care to absorb exudates and promote healing. In agriculture, alginate helps improve soil moisture retention. Pectin is a heteropolysaccharide composed of multiple sugar units and functions as a natural adhesive in plant cell walls. It is predominantly extracted from fruits, vegetables, and other plant tissues. Pectin offers several health benefits as a soluble fiber, including aiding in blood sugar regulation and digestion. It possesses the unique ability to form gels when heated with acid and sugar, making it an essential ingredient in the production of jams, jellies, and preserves. Even without full gelation, pectin solutions can thicken various food products, such as yogurts, sauces, and soups, enhancing their texture and consistency.

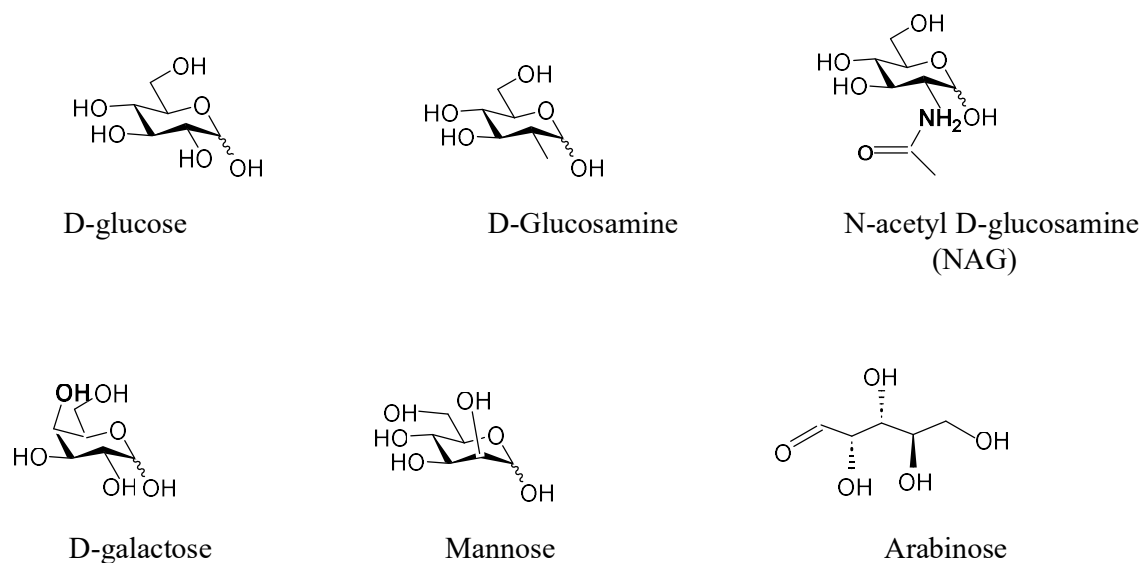


Figure 1. Structures of the sugar starting materials used often for designing low molecular weight gelators(LMWGs).

2. Materials and Methods

2.1 Chemicals and Reagents

All chemicals and reagents used in this study were of analytical grade and were employed without further purification unless otherwise stated. D-arabinose was procured from Sigma-Aldrich and used as the starting sugar for subsequent derivatization reactions. Acetic anhydride and pyridine (Merck) served as reagents for acetylation processes to protect the hydroxyl groups of D-arabinose. Phosphorus tribromide (Merck) was employed for the preparation of glycosyl bromide intermediates via halogenation. Sodium azide (Sigma-Aldrich) was utilized for azidation reactions to introduce the azide functional group necessary for click chemistry applications, while phenylacetylene (Sigma-Aldrich) acted as the alkyne counterpart in the azide-alkyne cycloaddition reaction. Copper sulfate pentahydrate and sodium ascorbate were used as catalytic agents to facilitate the generation of the active Cu(I) species required for the CuAAC (Copper(I)-Catalyzed Azide-Alkyne Cycloaddition) reaction. Solvents including benzene, toluene, and xylene (Merck, analytical grade) were used during reaction and purification procedures. Natural oils such as coconut oil and sesame oil were also used as gelation media during gelation experiments. All solvents were either distilled or used as received, and reactions were performed under controlled atmospheric conditions where necessary.

2.2 Synthesis of the Organogelator

2.2.1 Synthesis of 2,3,4-tri-O-acetyl-1-azido- β -D-arabinopyranoside (Compound 3)

The synthetic route to Compound 3 is outlined in Scheme 1 (Insert Reaction Scheme). D-arabinose (1 equivalent) was dissolved in pyridine under ice-cold conditions (0°C) and subsequently reacted with excess acetic anhydride under constant magnetic stirring to ensure complete acetylation of the hydroxyl groups. The reaction mixture was allowed to warm to room temperature gradually and stirred for additional hours until TLC monitoring indicated complete conversion to the acetylated sugar. After completion, the reaction mixture was poured into ice-cold water and extracted with an organic solvent to isolate the fully acetylated product. This acetylated derivative was then subjected to bromination using phosphorus tribromide in glacial acetic acid under anhydrous conditions to yield the corresponding glycosyl bromide intermediate. Subsequently, the brominated intermediate was treated with an excess of sodium azide in dry DMF at 80°C under nitrogen atmosphere to facilitate nucleophilic substitution, leading to the formation of 2,3,4-tri-O-acetyl-1-azido- β -D-arabinopyranoside (Compound 3).

The product was purified using column chromatography and characterized using spectroscopic techniques.

2.2.2 Synthesis of 1-(2,3,4-tri-O-acetyl- β -D-arabinopyranosyl)-4-(phenyl)-1H-1,2,3-triazole (Compound 4)

The preparation of the triazole-functionalized sugar derivative (Compound 4) was carried out via a Cu(I)-catalyzed azide-alkyne cycloaddition (CuAAC) reaction, as illustrated in Scheme 2 (Insert Reaction Scheme). Compound 3 was reacted with phenylacetylene in a mixed solvent system of tert-butanol and water (1:1, v/v). Copper sulfate pentahydrate and sodium ascorbate were added to the reaction mixture to generate the active Cu(I) catalyst in situ. The reaction was carried out at 60°C for 24 hours under stirring to ensure complete conversion. Upon completion, the reaction mixture was extracted with organic solvents, and the crude product was subjected to purification by silica gel column chromatography, yielding the desired triazole-linked sugar derivative (Compound 4). Structural confirmation of the product was performed using FT-IR and NMR spectroscopic techniques.

2.3 Characterization Techniques

The synthesized compounds were characterized using a suite of analytical techniques. Fourier Transform Infrared (FT-IR) Spectroscopy was conducted on a Bruker Alpha spectrometer in the spectral range of 4000–400 cm^{-1} to identify characteristic functional groups. Proton (^1H) and Carbon (^{13}C) Nuclear Magnetic Resonance (NMR) spectra were recorded at 400 MHz in DMSO- d_6 to confirm structural integrity and stereochemistry. Powder X-ray Diffraction (XRD) analysis was carried out using a Rigaku Miniflex II diffractometer to investigate the crystallinity and molecular packing behavior of the xerogel. Surface morphology and microstructural features of the xerogel were examined using Field Emission Scanning Electron Microscopy (FESEM, ZEISS Sigma). In addition, surface topography was analyzed via Atomic Force Microscopy (AFM, Bruker Dimension Icon) operating in tapping mode.

2.4 Gelation Studies

The gelation behavior of Compound 4 was investigated by dissolving different concentrations (0.1–2% w/v) of the compound in various solvents such as benzene, toluene, xylene, coconut oil, and sesame oil. The solutions were subjected to heating until complete dissolution was achieved, followed by cooling to room temperature or refrigeration to induce gel formation. Gelation was evaluated using the inverted vial method, wherein a gel was deemed to have

formed if no flow was observed upon inverting the vial after the cooling cycle. Critical gelation concentration (CGC) was recorded as the minimum concentration required to achieve stable gelation.

2.5 Rheological Studies

The mechanical properties and viscoelastic behavior of the gels were studied using a rheometer (Anton Paar MCR302). Dynamic oscillatory measurements were performed to determine the storage modulus (G') and loss modulus (G'') over a range of frequencies and strains. The measurements provided insights into the elastic and viscous nature of the gels, as well as the stability and robustness of the gel networks formed by Compound 4 in various solvent systems.

3. Results and Discussion

3.1 Structural Characterization

FT-IR Analysis

The structural transformation from azide to triazole was confirmed through Fourier Transform Infrared (FT-IR) spectroscopy. The FT-IR spectrum of Compound 4 exhibited a distinct disappearance of the azide stretching vibration ($\sim 2100\text{ cm}^{-1}$), accompanied by the emergence of a characteristic triazole C=N stretching band around 1650 cm^{-1} , indicating successful cycloaddition. Additionally, a broad absorption band around 3400 cm^{-1} was observed, corresponding to O–H stretching vibrations, suggesting the presence of intermolecular hydrogen bonding interactions among gelator molecules, which play a crucial role in the self-assembly and gelation process.

^1H and ^{13}C NMR Spectroscopic Analysis

The ^1H NMR spectrum recorded in DMSO- d_6 revealed key signals that corroborated the formation of the triazole ring. A singlet at approximately $\delta \sim 8.0\text{ ppm}$ was attributed to the proton of the triazole moiety, confirming the formation of the 1,2,3-triazole linkage. Additionally, signals appearing around $\delta \sim 2.0\text{ ppm}$ were assigned to the methyl groups of the acetyl protecting groups. The ^{13}C NMR spectrum further supported the structure, displaying signals corresponding to carbonyl carbons (C=O) of the acetyl groups around $\delta \sim 170\text{ ppm}$ and aromatic carbons of the phenyl ring between $\delta \sim 120\text{--}140\text{ ppm}$. Together, the NMR data provided strong evidence for the successful synthesis of Compound 4.

XRD Analysis

The X-ray Powder Diffraction (XRD) pattern of the xerogel of Compound 4 (Figure 1) displayed sharp and intense diffraction peaks, indicative of high crystallinity and well-organized molecular packing. The d-spacing values, calculated using Bragg's Law, suggested the presence of lamellar stacking structures. Such ordered molecular arrangements are essential for robust gel formation, as they provide the physical framework necessary to entrap solvents within the self-assembled network.

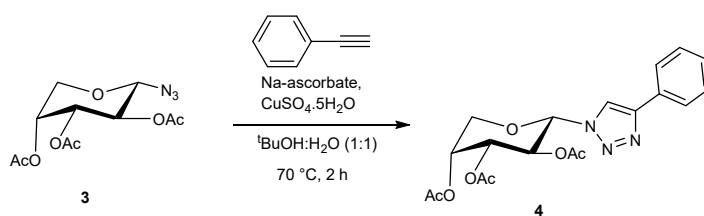


Figure 1. synthesis of 2,3,4-tri-o-acetyl- β -D-arabinopyranosyl)-4-(phenyl)-1H-1,2,3-triazole).

3.2 Gelation Properties

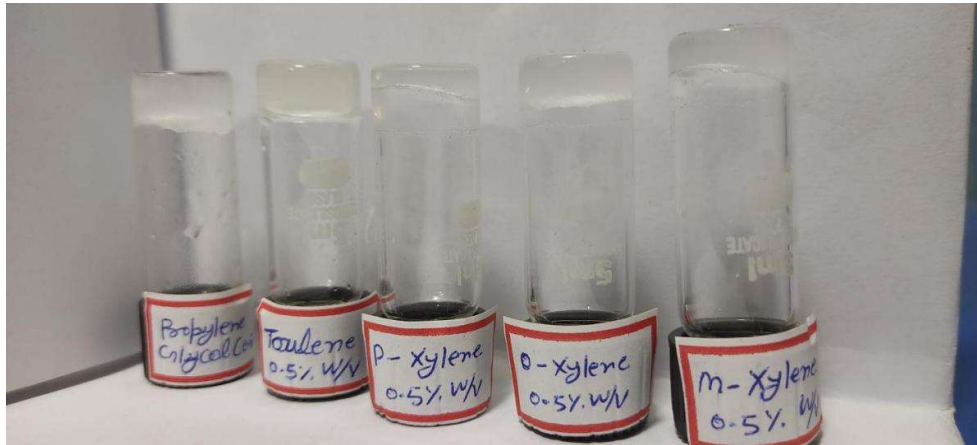
The gelation ability of Compound 4 was systematically evaluated across a range of organic solvents and oils. The compound demonstrated excellent gelation behavior in nonpolar aromatic solvents such as benzene, toluene, and all isomers of xylene (o-, m-, p-), as well as in natural oils like coconut oil and sesame oil. The minimum gelation concentration (MGC) was determined to be as low as 0.5% w/v in several cases, highlighting the high efficiency of the gelator.

Table 1. Gelation test, I= Insoluble, S = Soluble, G = Gel, PP =Partial Precipitate, PG =Partial Gel, MGC =Minimum gelation concentration %w/v.

Sr. N.	Solvent	Appearance (MGCs)
1.	Coconut oil	G(0.5%, >100 ⁰ c)
2.	Water	I
3.	Alsi oil	PG
4.	Mustard oil	PG
5.	Sesame oil	G(0.5%, 100-110 ⁰ c)
6.	Linseed oil	PP
7.	Propylene Glycol	G(0.5%, 90-95 ⁰ C)
8.	Chloroform	S
9.	o-xylene	G(0.5%, 52-55 ⁰ c)
10.	m-xylene	G(0.5%, 52-58 ⁰ c)
11.	p-xylene	G(0.5%, 50-52 ⁰ c)
12.	Ethanol	PG
13.	Methanol	S
14.	Toluene	G(0.5%, 50-55 ⁰ c)
15.	Benzene	G(0.5%, 47-50 ⁰ c)
16.	Chlorobenzene	G(0.5%, 45-54 ⁰ c)

Photographic documentation (Figure 2) clearly shows the formation of stable gels under ambient conditions. It was observed that the strength and robustness of the gels varied with solvent polarity, suggesting that solvent–gelator interactions, including π – π stacking, van der

Waals forces, and hydrogen bonding, significantly influence the gelation efficiency and network stability.

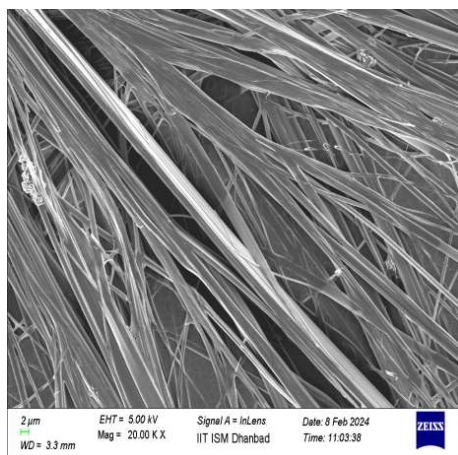


(a) propylene Glycol (0.5%w/v), (b) Toulene (0.5%w/v), (c) p-xylene (0.5%w/v), (d) o-xylene (0.5%w/v), (e) m-xylene (0.5%w/v).

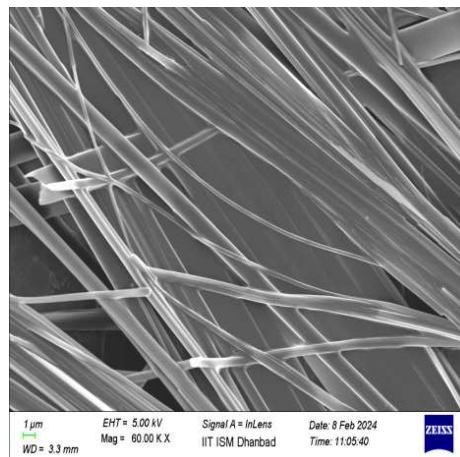
3.3 Morphology and Self-Assembly Behavior

FESEM Analysis

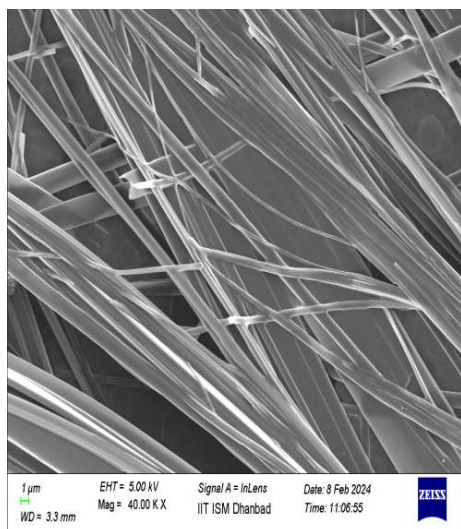
Field Emission Scanning Electron Microscopy (FESEM) was employed to study the xerogel morphology. The FESEM images (Figure 3) revealed the formation of highly interconnected fibrillar networks with fiber diameters ranging from 100–300 nm. These fibrous architectures are characteristic of low molecular weight gelators and are essential for the physical entrapment of solvent molecules within the gel matrix.



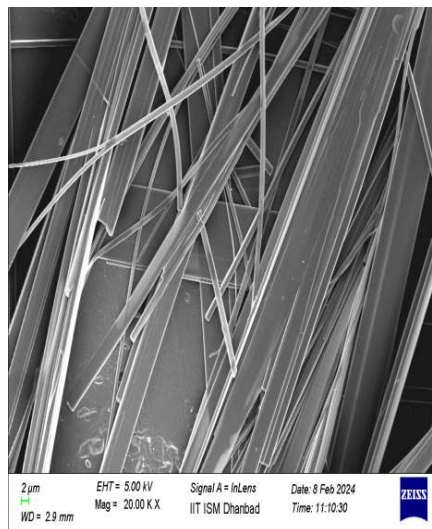
(a) FESEM image of xerogels in propylene glycol solvent and width of fibre 3.3 mm.



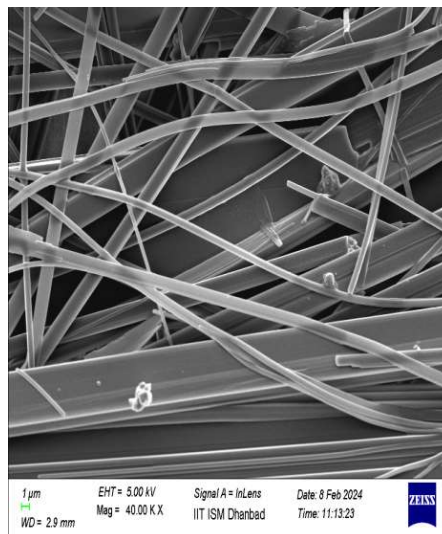
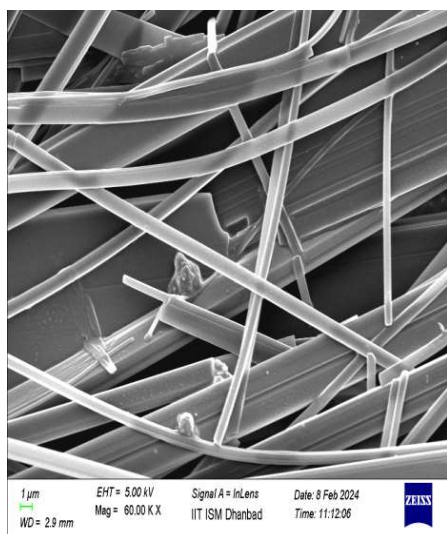
(b) FESEM image of xerogels in propylene glycol solvent and width of fibre 3.3 mm.



(d) FESEM image of xerogels in propylene glycol solvent and width of fibre 3.3 mm.



(c) FESEM image of xerogels with drug in propylene glycol solvent and width of fibre 2.9 mm.



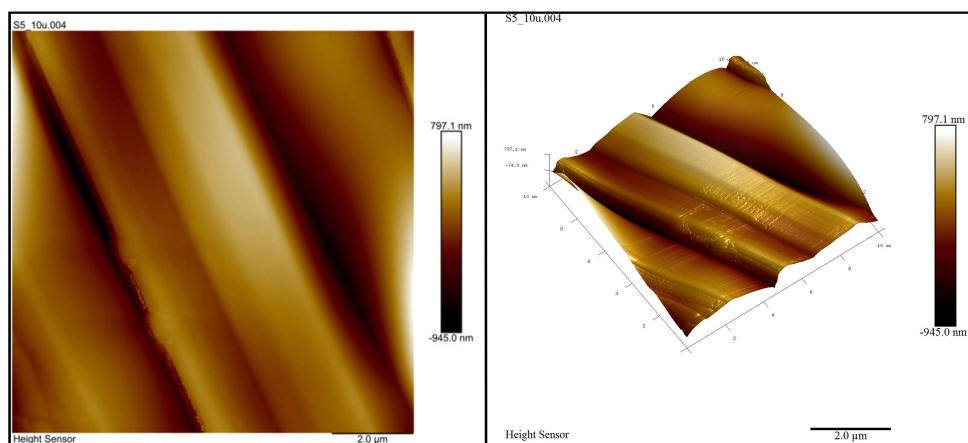
(f) FESEM image of xerogels with drug in propylene glycol solvent and width of fibre 2.9 μm .

(e) FESEM image of xerogels with drug in propylene glycol solvent and width of fibre 2.9 μm .

Figure 2 . Field emission scanning electron microscopy (FESEM) of image.

AFM Analysis

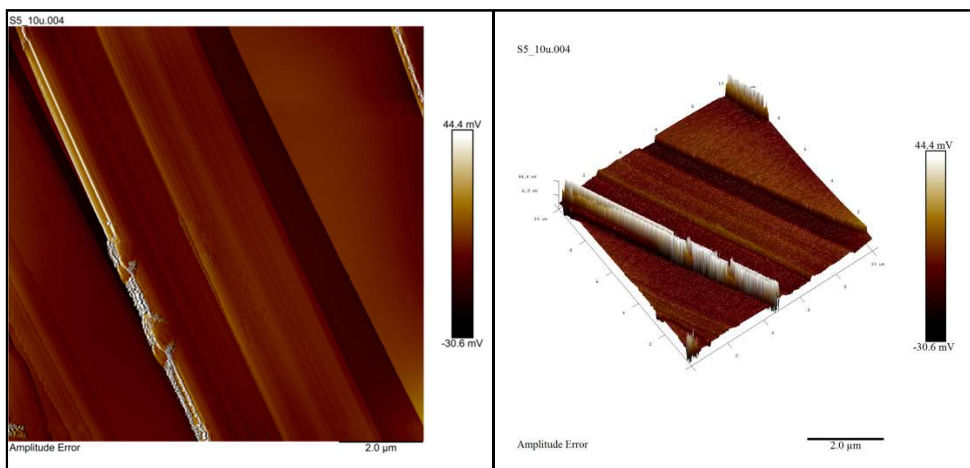
Atomic Force Microscopy (AFM) provided complementary insights into the surface topography at the nanoscale. AFM images (Figure 4) revealed well-organized, bundled nanofibers with uniform diameters, further confirming the nanoscale self-assembly behavior of Compound 4. Such finely structured networks are critical for the mechanical integrity and long-term stability of the gels.



(a)

(b)

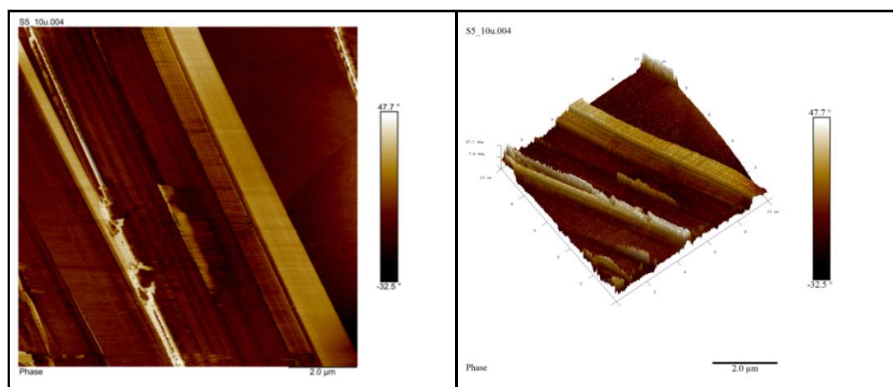
AFM image of gelator (4) in propylene glycol solvent, (a) 2-D image and (b) 3-D image of height sensor.



(c)

(d)

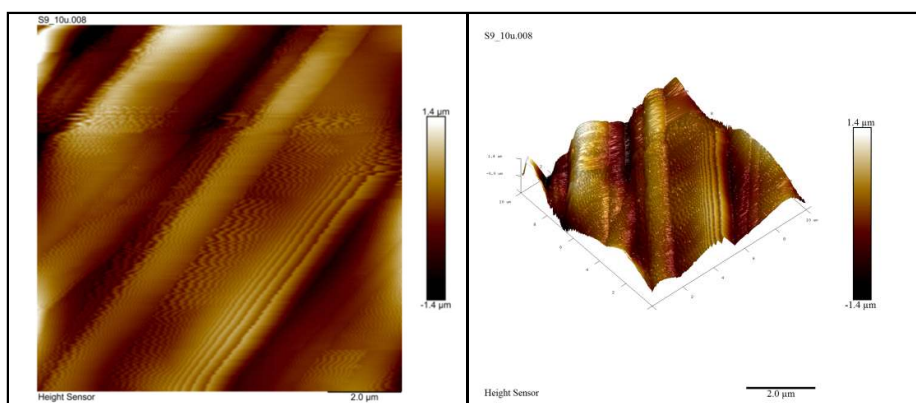
AFM image of gelator (4) in propylene glycol solvent, (c) 2-D image and (d) 3-D image of amplitude error.



(e)

(f)

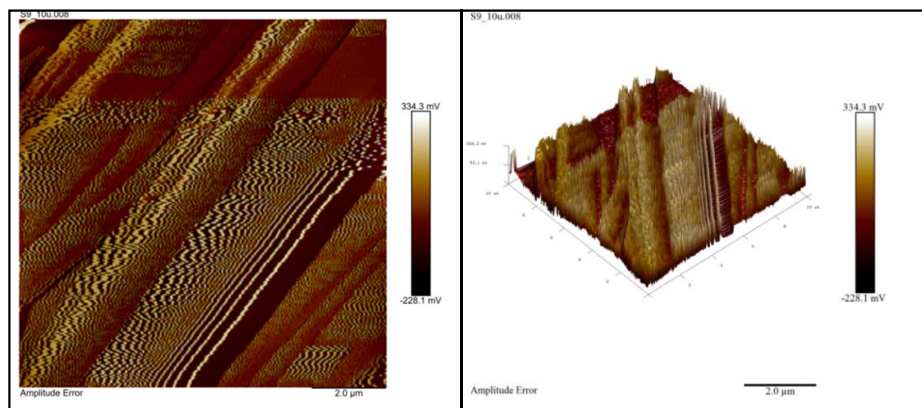
AFM image of gelator (4) in propylene glycol solvent, (e) 2-D image and (f) 3-D image of phase.



(g)

(h)

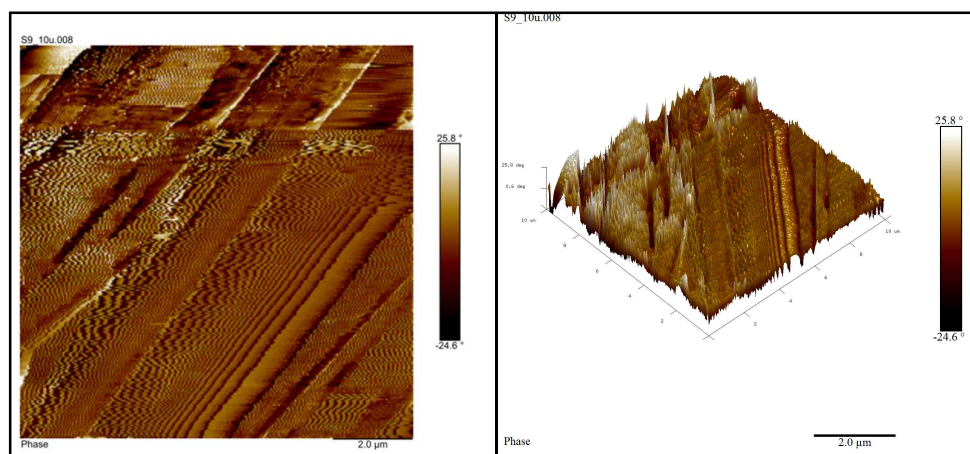
AFM image of gelator (4) with drug in propylene glycol solvent, (g) 2-D image and (h) 3-D image of height sensor.



(i)

(j)

AFM image of gelator (4) with drug in propylene glycol solvent, (i) 2-D image and (j) 3-D image of amplitude error.



(k)

(l)

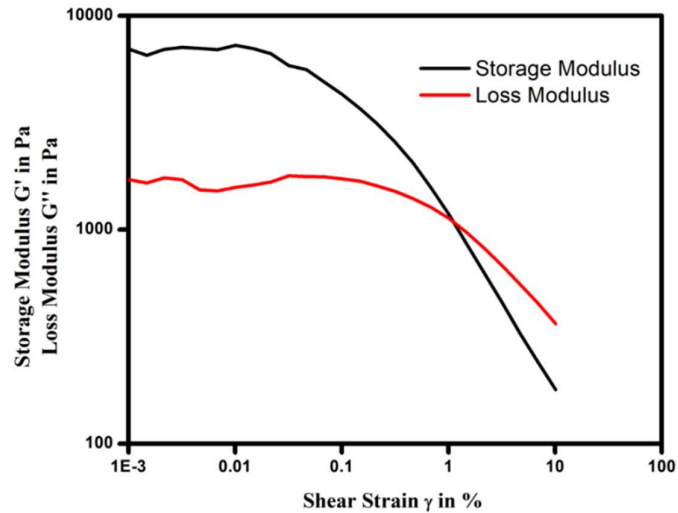
AFM image of gelator (4) with drug in propylene glycol solvent, (k) 2-D image and (l) 3-D image of phase.

Figure 3. Atomic force microscopy Image.

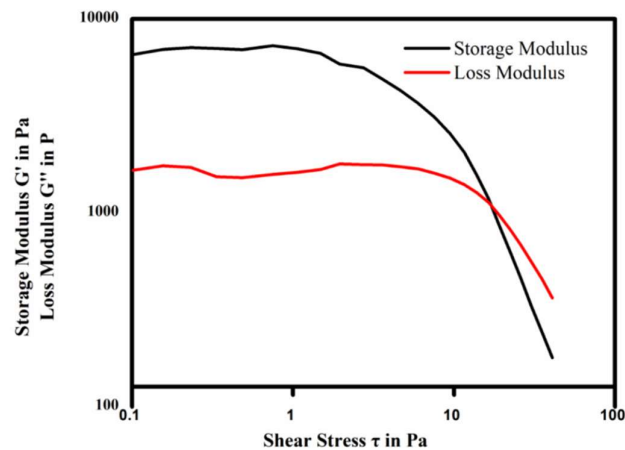
3.4 Rheological Behavior

The mechanical properties of the gels were probed through oscillatory rheological measurements. The frequency sweep experiments demonstrated that the storage modulus (G')

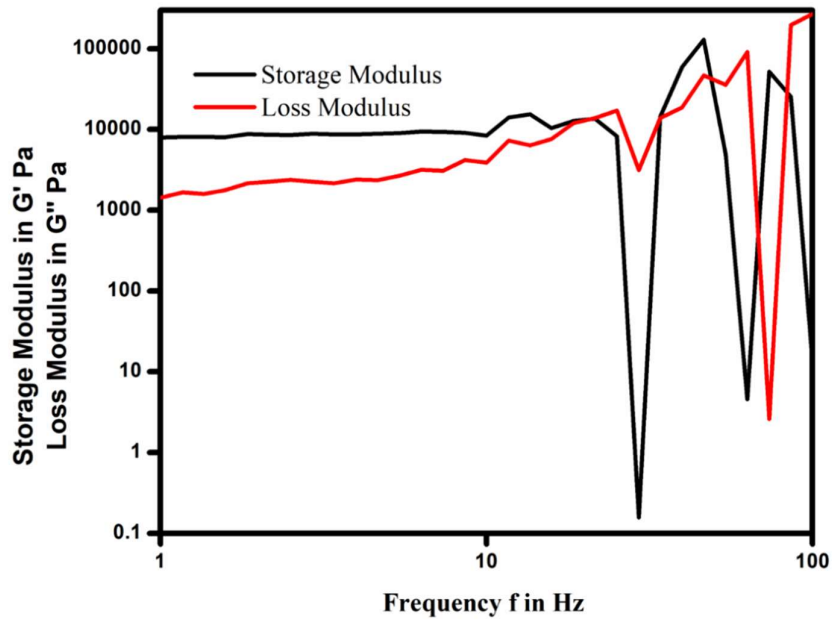
was consistently higher than the loss modulus (G''), confirming the predominantly elastic behavior of the gels (Figure 5). This elastic dominance is a key requirement for practical applications, as it indicates the ability of the gel to maintain its shape under external stress. Moreover, the gels displayed good mechanical resilience, withstanding moderate shear stresses without structural breakdown, thereby highlighting their mechanical robustness and suitability for applications such as soft materials, drug delivery matrices, and tissue engineering scaffolds.



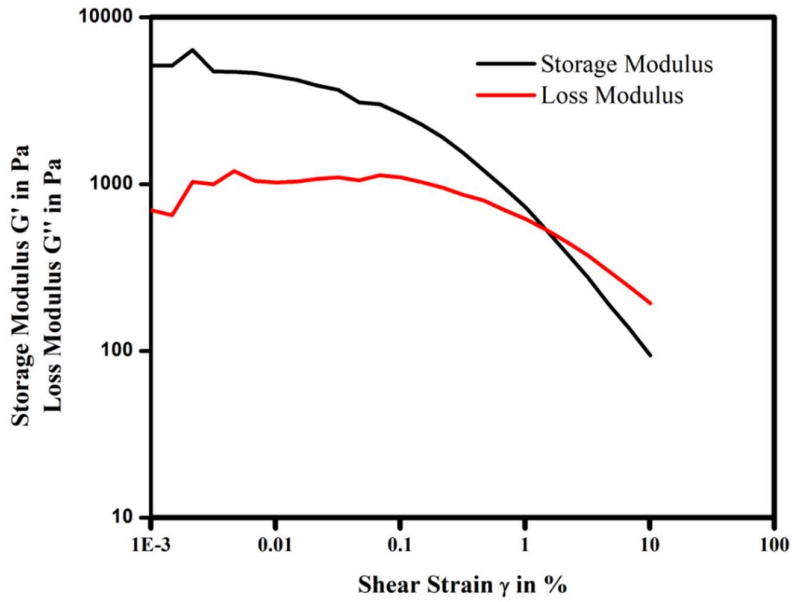
(a) Graph between Shear Strain and Storage modulus, Loss modulus of the gelator.



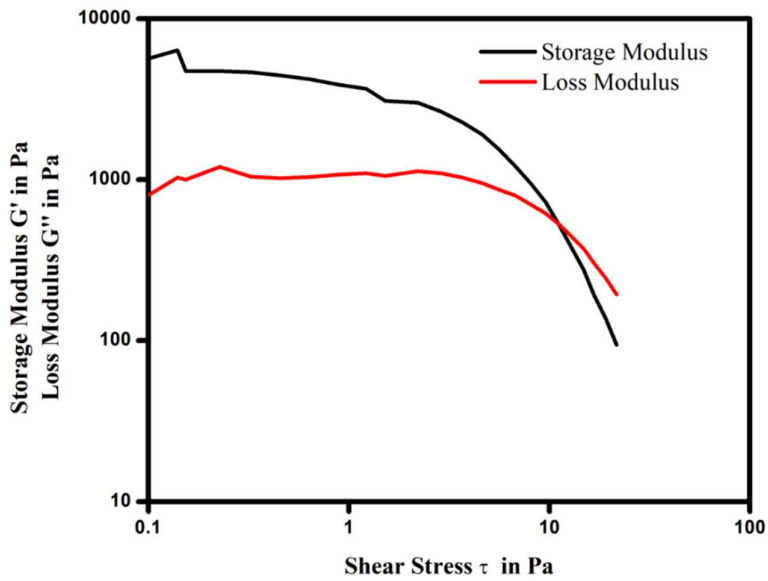
(b) Graph between Shear Stress and Storage modulus, Loss modulus of the gelator.



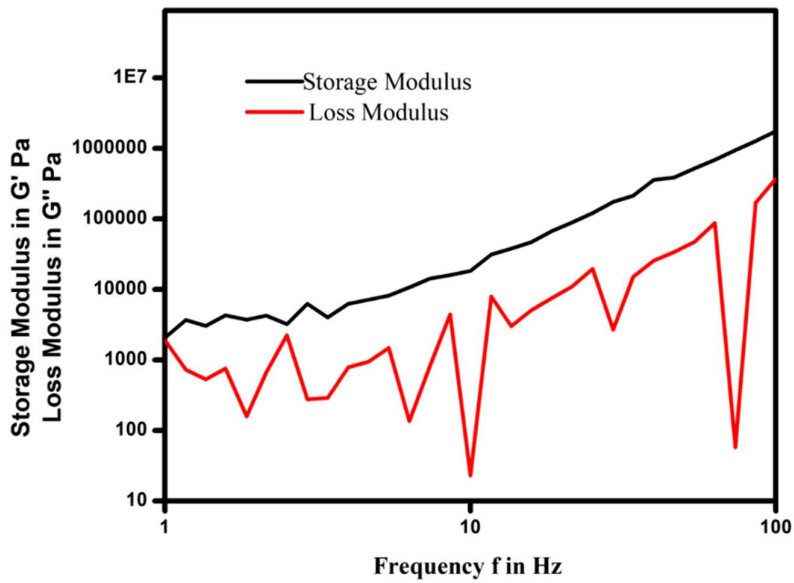
(c) Graph between Frequency and Storage modulus, Loss modulus of the gelator.



(d) Graph between Shear Strain and Storage modulus, Loss modulus of the gelator with drug.



(e) Graph between Shear Stress and Storage modulus, Loss modulus of the gelator with drug.



(f) Graph between frequency and Storage modulus, Loss modulus of the gelator with drug.

Figure 4. Rheology Studies graph.

4. Conclusion

In this study, a pentose-based triazole derivative was successfully synthesized and evaluated for its organogelation properties. The compound exhibited excellent gelation ability, forming stable and self-supporting gels across a variety of organic solvents at low critical concentrations. Structural characterization confirmed that the ordered molecular packing was primarily governed by strong non-covalent interactions, including hydrogen bonding and π - π stacking, which facilitated the formation of a robust supramolecular network. The organogels demonstrated superior mechanical strength and viscoelastic properties, as evidenced by rheological analysis, and exhibited a characteristic fibrillar morphology under microscopic examination. These combined attributes underscore the potential of the synthesized organogelator for a wide range of applications in the biomedical, pharmaceutical, and soft materials fields. Future studies focusing on functional modifications and application-specific evaluations may further expand the utility of this promising class of supramolecular materials.

Ethics Approval and Consent to Participate

Not applicable.

Human and Animal Rights

No animals or humans were involved in this study.

Consent for Publication

Not applicable.

Availability of Data and Materials

All data generated or analyzed during this study are available from the corresponding author upon reasonable request.

Funding

No external funding was received for this study.

Conflict of Interest

The authors declare no conflict of interest.

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