



The Effect of Vacuum Drying vs. Ambient Drying on the Nanostructural, Mechanical, and Rheological Properties of PEGDA Hydrogels

Ahmad Akid Zulkifli¹, Syazwani Mohd Zaki^{1,*}, Flora Serati²

¹ Department of Manufacturing and Materials Engineering, Kulliyah of Engineering, International Islamic University Malaysia, PO Box, 50728 Kuala Lumpur, Malaysia

² Department of Electrical and Computer Engineering, Kulliyah of Engineering, International Islamic University Malaysia, PO Box, 50728 Kuala Lumpur, Malaysia

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ABSTRACT

Drying is a critical yet often overlooked step in the synthesis of poly(ethylene glycol) diacrylate (PEGDA), as it governs solvent removal, polymer chain organization, and ultimately the structural and mechanical properties of the resulting hydrogel. In this study, PEGDA was synthesized through an acylation reaction, purified via sequential washing and filtration, and subsequently subjected to two distinct drying methods: ambient drying and vacuum drying at 35 °C. The dried PEGDA powders were then photo-crosslinked using IRGACURE 1173 to form hydrogels for comparative analysis. Results revealed that the vacuum-dried PEGDA exhibited superior structural uniformity and mechanical strength, producing nanoparticles with an average diameter of 4.6 nm as measured by DLS. The corresponding hydrogel displayed a Young's modulus of 7.1 kPa, nearly double that of the ambient-dried sample (3.9 kPa). Rheological analysis further confirmed a fourfold increase in crossover stress ($G' = G''$) for vacuum-dried PEGDA (34.5 kPa) compared to the ambient-dried counterpart (8.7 kPa), signifying enhanced elasticity and network integrity. These findings highlight that controlled vacuum drying significantly improves polymer uniformity and crosslinking efficiency, providing a simple yet effective strategy for optimizing PEGDA hydrogels in advanced applications such as solar vapor generation and water purification.

Keywords:

Polymer Hydrogel; Solar Vapor Generation; Polyaniline

1. Introduction

Solar vapor generation (SVG) has gained increasing attention as an emerging and sustainable approach to address global freshwater scarcity by utilizing renewable solar energy for efficient interfacial evaporation. Unlike traditional desalination or purification technologies, which require high energy consumption, SVG systems utilise localized photothermal conversion processes, making them more energy-efficient and environmentally friendly [1]. Their scalability and adaptability to various water sources position SVG as a key technology for sustainable water management. Recent advances have focused on bio-inspired and conductive hydrogels for enhancing interfacial

* Corresponding Author

E-mail address: syazwani.mohdzaki@iium.edu.my

evaporation and photothermal conversion efficiency [2]. However, most studies prioritize compositional modification rather than processing control.

Poly(ethylene glycol) diacrylate (PEGDA) based hydrogels have recently attracted significant interest as advanced materials for SVG due to their tunable crosslinking density, biocompatibility, and strong water affinity. These hydrogels possess three dimensional porous networks that facilitate continuous water diffusion and evaporation, providing excellent interfacial contact for solar-driven processes [3]. Previous studies have explored various compositional modifications of PEGDA hydrogels, including copolymerization and the incorporation of nanofillers, to enhance their mechanical integrity and light absorption[4]. However, the influence of post-synthesis processing steps, particularly drying, on the structural and functional behaviour of PEGDA hydrogels remains largely underexplored.

Drying is a crucial yet often overlooked step that dictates solvent removal, polymer chain packing, and pore structure formation. Improper drying can lead to structural collapse, incomplete solvent evaporation, or non-uniform crosslinking, which significantly alter the physical and mechanical performance of hydrogels [5]. Emerging studies have shown that controlled drying conditions, such as vacuum or freeze-drying, can enhance polymer homogeneity, porosity, and mechanical resilience[6]. This vacuum drying accelerates solvent removal and promotes uniform polymer network formation, while the drying under controlled conditions improves pore architecture and elasticity in PEG-based systems. These studies underscore that drying acts as a powerful parameter for tuning hydrogel properties yet its systematic role in PEGDA-based hydrogels designed for solar vapor generation remains unclear.

Therefore, this study aims to systematically investigate the effect of two distinct drying conditions of ambient drying and vacuum drying on the nanostructural, mechanical, and rheological properties of PEGDA hydrogels. By correlating the drying technique with the resulting polymer morphology and viscoelastic performance, this work addresses a key knowledge gap in hydrogel engineering and provides fundamental insight into optimizing PEGDA hydrogels for efficient solar-driven water purification applications. The insights derived provide a foundation for tailoring drying strategies toward scalable fabrication of high-efficiency hydrogel-based photothermal materials for sustainable water purification.

2. Methodology

2.1 Materials

PEG- 8000 powders, anhydrous benzene (99.8%), triethylamine (0.012moles), acryloyl chloride (97%) (0.012 moles) and hexane were purchased from PLT scientific. All chemicals were used without further purification.

2.2 Method

In this study, 12 g of PEG ($M_n = 8000$) was dissolved in 150 mL of benzene and stirred at 45 °C for 1 h at 300 rpm. After the solution was cooled to room temperature under a controlled atmosphere, triethylamine (1.67 mL, 0.012 mol) was added first, followed by the dropwise addition of acryloyl chloride (0.97 mL, 0.012 mol, 97%; Sigma-Aldrich) under continuous stirring. The reactant quantities were set to four times the stoichiometric ratio to ensure complete acylation. The reaction was conducted under a nitrogen atmosphere to prevent unwanted side reactions with moisture or oxygen. The mixture was then stirred at 80 °C for 3 h at 300 rpm to facilitate complete bonding of acrylate groups to the PEG chains. Upon completion, the synthesized PEGDA was precipitated and

thoroughly washed with hexane to remove residual by-products, such as triethylamine hydrochloride, and any unreacted monomers (e.g., acryloyl chloride) or excess triethylamine, ensuring the purity of the final PEGDA powder. For room temperature drying (RT Drying), the precipitated PEGDA obtained after the washing process was transferred into a 400 mL beaker, covered with aluminium foil, and kept in a dark environment at room temperature for 48 hours. After drying, the solidified PEGDA aggregates were gently crushed using a spatula to obtain fine white PEGDA powder. For vacuum oven drying, the washed PEGDA precipitate was placed in a 400 mL beaker, covered with aluminium foil, and dried in a vacuum oven at 35 °C for 48 hours to ensure complete removal of residual solvents and moisture. The resulting dried PEGDA aggregates were ground into fine PEGDA powder. For hydrogel fabrication, PEGDA hydrogels for testing were prepared by dissolving 1 g of the PEGDA powder in deionized water and 1 μ l of photoinitiator IRGACURE 1173 was added to the solution. The solution was vortexed, poured into a cylindrical mould (1.0 cm diameter x 0.5 cm height), and photo-crosslinked under a 365 nm of UV lamp for 60 mins.

3. Results

The comparative analysis of ambient and vacuum-dried PEGDA hydrogels reveals how drying conditions critically shape the polymer's nanostructure and mechanical performance.

3.1 Dynamic Light Scattering Analysis of PEGDA

The particle size distribution of PEGDA powders obtained from different drying methods is shown in Fig. 1. The vacuum-dried PEGDA exhibited a sharp and narrow size distribution centred at 4.58 nm with a polydispersity index (PDI) of 0.230, indicating a uniform and monodisperse nanoparticle population. In contrast, the RT-dried PEGDA displayed a broad distribution with an average particle size of 323.2 nm and a PDI of 0.658, reflecting significant heterogeneity and particle agglomeration.

This remarkable contrast in particle size can be attributed to the pressure and temperature differences inherent in the two drying methods. Under vacuum conditions, the reduced ambient pressure lowers the boiling point of the solvent, enhancing the rate of evaporation even at low temperatures. This rapid solvent removal minimizes the opportunity for polymer chains to aggregate, thereby preventing particle growth and promoting the formation of smaller, well-dispersed PEGDA nanoparticles[7, 8]. Furthermore, the vacuum environment suppresses oxidation and contamination, thereby stabilizing the polymer structure and maintaining its nanoscale dimensions. Conversely, the RT drying process occurs under atmospheric pressure and without thermal assistance, leading to a slower solvent evaporation rate. This prolonged drying duration facilitates moisture absorption and chain entanglement, allowing PEGDA particles to coalesce into larger aggregates. Additionally, slow evaporation promotes solvent migration toward the particle surface before vaporization, contributes to secondary agglomeration and uneven particle morphology. The TEM image in Fig. 1(b) confirms the presence of nanosized PEGDA particles in the vacuum-dried sample, consistent with the DLS data. The agreement between TEM and DLS results verifies that the vacuum drying technique effectively suppresses agglomeration, yielding stable nanoscale PEGDA suitable for forming homogeneous hydrogels with improved mechanical and photothermal properties.

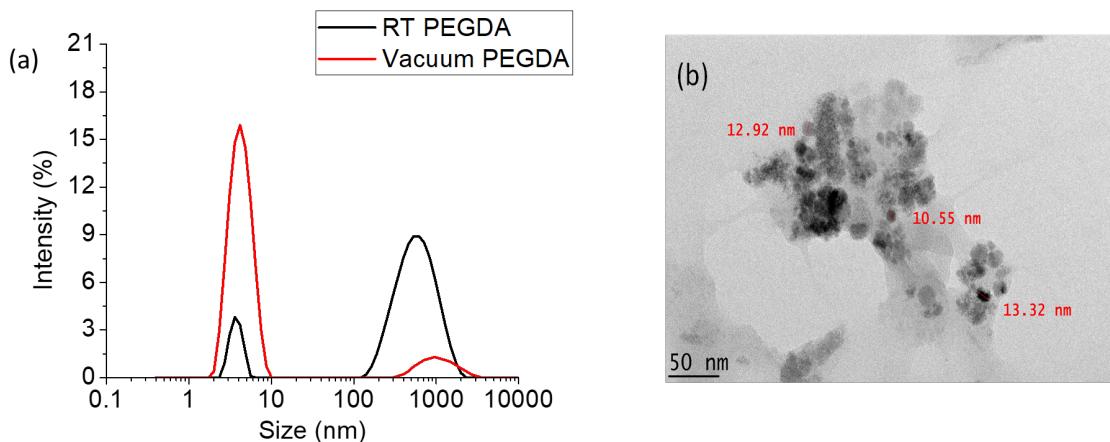


Fig. 1. The particle size of PEGDA powder. (a) particle size measured using DLS. (b) Imaging of the particle size of vacuum PEGDA measured using TEM

3.2 Compression Analysis of RT Drying PEGDA and Vacuum Drying PEGDA

A compression test was performed to evaluate the mechanical properties of PEGDA hydrogels prepared using two methods: room temperature (RT) drying (RT PEGDA) and vacuum oven drying (Vacuum PEGDA). The hydrogels were fabricated in cylindrical form with dimensions of 1.5 cm in height and 1.0 cm in diameter. The corresponding stress-strain curves for both samples are presented in Fig. 2, where Fig. 2(a) represents the vacuum-dried PEGDA hydrogel and Fig. 2(b) represents the RT-dried hydrogel. As observed in Fig. 2, the RT-dried PEGDA hydrogel exhibited greater ductility (breaking point at 0.80 of strain), reflected by its higher strain at failure and lower Young's modulus (E), as quantified in Fig. 2(a) and Fig. 2(b). The calculated Young's modulus of the vacuum-dried PEGDA hydrogel was 7.1 kPa, whereas the RT-dried PEGDA hydrogel demonstrated a significantly lower modulus of 3.9 kPa. Vacuum PEGDA exhibited a higher Young's modulus, indicating a denser and more elastic network [5]. This improved stiffness and viscoelastic stability stem from reduced free volume and stronger interchain packing. Such enhancement supports durability under cyclic wet dry conditions relevant to SVG. Furthermore, Vacuum drying facilitates the efficient removal of residual solvent and unreacted monomers, resulting in a denser polymer network with increased crosslinking sites and reduced free volume [9, 10]. In contrast, RT PEGDA, which involves slower solvent evaporation under ambient conditions, tends to produce a more loosely cross-linked structure with greater chain mobility and water retention [11]. This structural difference contributes to the enhanced ductility and lower modulus observed in the RT-dried hydrogel. These findings are consistent with previous reports, which show that the mechanical strength of PEGDA-based hydrogels strongly depends on crosslinking density and drying conditions, influencing polymer chain entanglement, porosity, and elasticity [12, 13]. Therefore, the vacuum drying process is more effective in achieving mechanically robust PEGDA hydrogels, whereas RT drying yields softer and more deformable networks, which are suitable for applications requiring flexibility and high-water content.

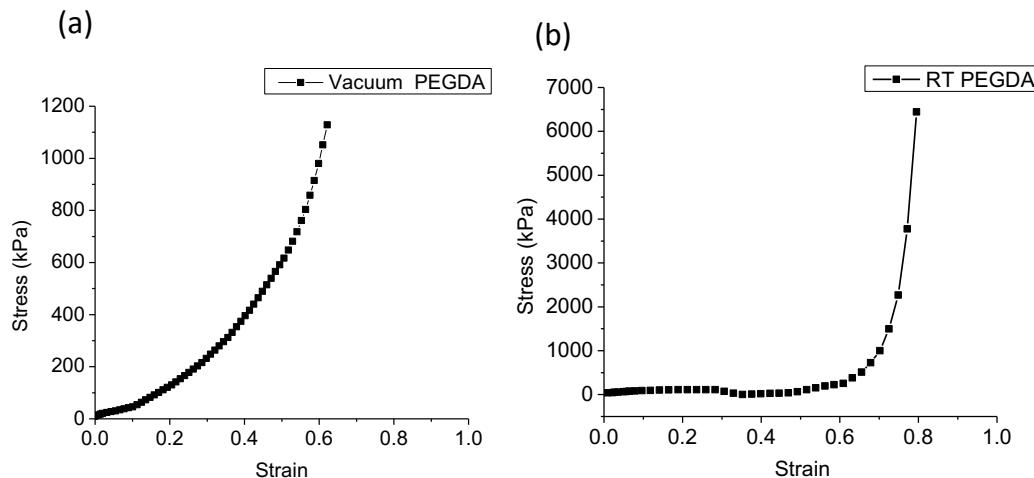


Fig. 2. Stress-strain curve of PEGDA hydrogel (a) vacuum PEGDA hydrogel (PEGDA Vac) (b) RT PEGDA hydrogel (PEGDA RT)

3.3 UV-VIS and FTIR Analysis of PEGDA

The UV–Vis spectra of PEGDA polymers (Fig. 3) revealed strong absorbance peaks in the ultraviolet region (200–400 nm), with maxima observed at 215 nm and 210 nm for the RT-dried and vacuum-dried samples, respectively. These peaks are attributed to the ether (C–O–C) bonds present in the ethylene glycol backbone of PEGDA. The slightly higher absorbance intensity of the RT-dried PEGDA suggests a greater concentration or stronger transition of ether bonds compared to the vacuum-dried sample, possibly due to a less compact network structure and residual solvent retention[14]. The FTIR spectra of both RT-dried and vacuum-dried PEGDA samples (Fig. 1) exhibited characteristic peaks at 1110 cm^{-1} , 1722 cm^{-1} , and 1636 cm^{-1} , corresponding to C–O–C stretching, C=O stretching, and C=C stretching vibrations, respectively [4]. These peaks confirm the successful acrylation of PEG, indicating the presence of acrylate functional groups within the PEGDA chain. Notably, the absence of a broad O–H stretching band around 3400 cm^{-1} in the vacuum-dried PEGDA indicates that this method effectively removed water and residual hydroxyl groups, yielding a purer product. Additional peaks were observed at 2901 cm^{-1} (CH₂ vibrations), 2917 cm^{-1} (symmetric C–H stretching), 1112 cm^{-1} (symmetric C–O–C vibrations), 954 and 841 cm^{-1} (C–O–C vibrational modes), and 523 cm^{-1} (CH deformation). The peak at 683 cm^{-1} corresponds to the COO[−] group, further supporting the successful synthesis of PEGDA. Collectively, the UV–Vis and FTIR results confirm that PEGDA was successfully synthesized, with vacuum drying producing a cleaner and more structurally defined polymer network compared to RT drying.

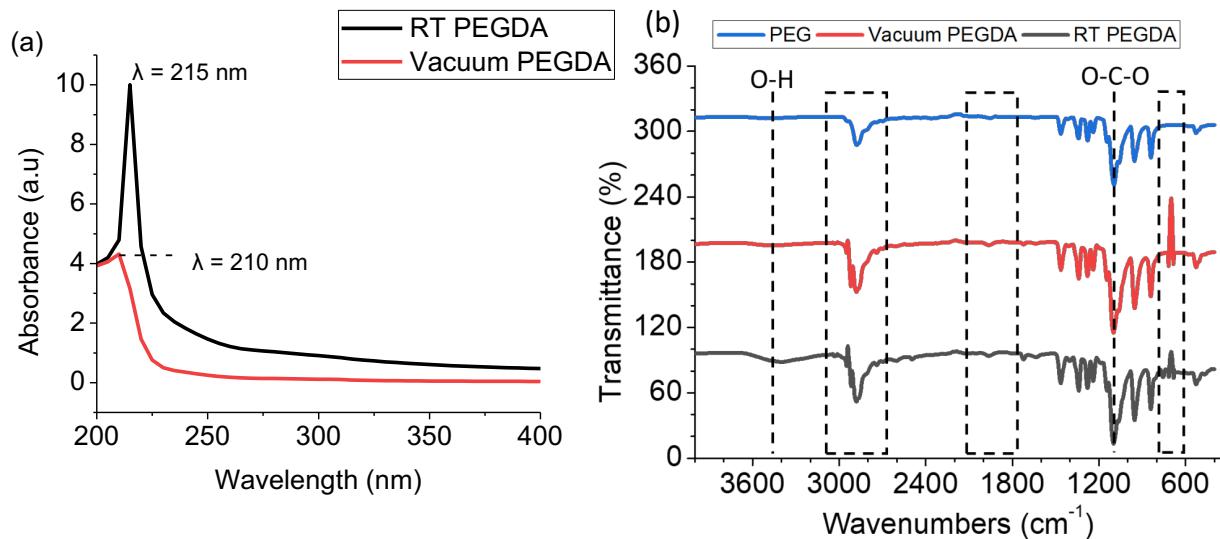


Fig. 3. UV-Vis of PEGDA powder (b) FTIR spectra of vacuum dry PEGDA powder, RT dry PEGDA powder and PEG powder

3.4. Analysis of Surface Morphology of PEGDA

The surface morphologies of the PEGDA hydrogels were examined using scanning electron microscopy (SEM) to evaluate the presence and distribution of pores within the polymer matrix. The pore size, distribution, and density significantly influence the hydrogel's physical and mechanical properties, as well as its ability to store and release water or chemical compounds. As shown in Fig. 4, the SEM images of PEGDA hydrogels prepared via RT drying (Fig. 4a, c, e) and vacuum drying (Fig. 4b, d, f) reveal notable differences in surface texture and uniformity. Both samples exhibited relatively smooth, non-porous surfaces without visible mesh structures or voids. This absence of porous morphology can be attributed to the relatively low molecular weight of the PEG precursor (PEG-8000) used in the hydrogel formulation, which limits chain entanglement and pore formation within the crosslinked matrix[15]. At higher magnification (1000 \times), distinct textural differences were observed between the two samples. The RT-dried PEGDA hydrogel exhibited a rougher and more irregular surface with pronounced valleys and peaks compared to the smoother surface of the vacuum-dried PEGDA hydrogel (Fig. 4c and 4f). The surface roughness in the RT-dried sample may arise from a non-uniform particle size distribution and partial aggregation that occurred during solvent evaporation at ambient conditions. The slower and less controlled drying at room temperature likely caused heterogeneous shrinkage and uneven polymer chain packing, leading to localized stress concentration regions that manifest as surface irregularities. In contrast, vacuum drying produced a more homogeneous and compact structure due to efficient solvent removal and reduced capillary stress, which promotes uniform polymer chain alignment and minimises surface defects. Such morphological uniformity is expected to enhance the mechanical integrity and structural stability of the vacuum-dried hydrogel, aligning with the mechanical results in the next subtopic.

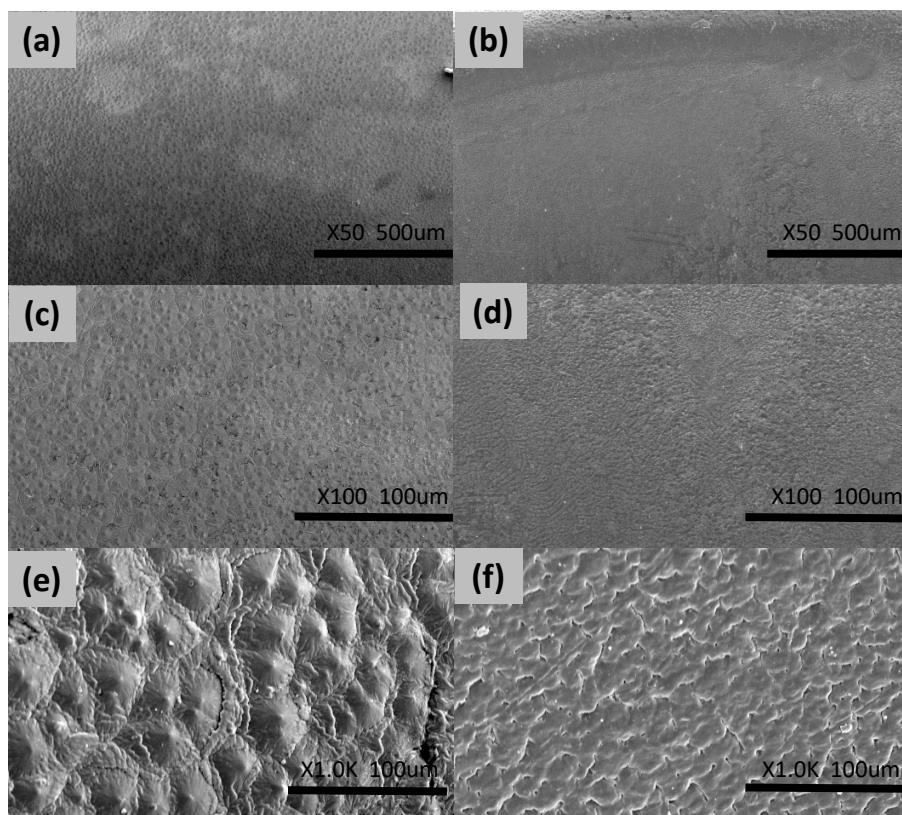


Fig. 4. SEM image of RT PEGDA hydrogel (a,c,e) and Vacuum PEGDA hydrogel (b,d,f)

3.5. Dynamic Rheology Analysis of PVA-CS hydrogel and PVA-CS/PANI hydrogel

The rheological behavior of the PEGDA hydrogels was examined using dynamic frequency and amplitude sweep tests to evaluate their viscoelastic properties (Fig. 5). Both samples exhibited a broad linear viscoelastic region (LVR), indicating structural stability under small oscillatory deformations. In this region, the storage modulus (G') was consistently higher than the loss modulus (G'') for both RT-dried and vacuum-dried PEGDA hydrogels, signifying that the elastic (solid-like) response dominated over the viscous component. A lower G'' value reflects effective restriction of polymer chain slippage, suggesting a stable and well-crosslinked polymeric network [16]. The vacuum-dried PEGDA hydrogel demonstrated a significantly higher storage modulus (G') compared to the RT dried sample, indicating greater stiffness and improved elastic recovery. This enhancement can be attributed to the smaller particle size and higher surface area-to-volume ratio of the vacuum dried PEGDA powder, which promotes better interparticle contact and more efficient crosslinking during the formation of the hydrogel. The resulting dense network structure provides superior load-bearing capability and enhanced mechanical integrity. The dominance of G' over G'' across the frequency range further confirms that both hydrogels exhibit typical elastic gel behaviour, although the vacuum-dried sample shows stronger solid-like characteristics.

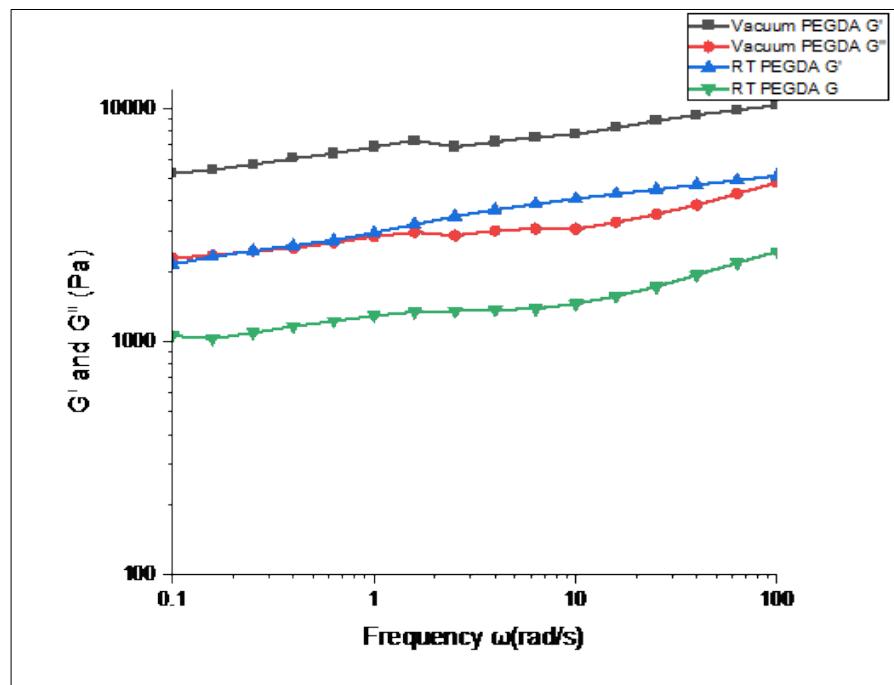


Fig. 5. Storage modulus (G') and loss modulus (G'') of PEGDA hydrogel.
(a) RT PEGDA hydrogel. (b) Vacuum PEGDA hydrogel

The amplitude sweep results (Fig. 6) further highlight the distinction between the two samples. The crossover point where G' equals G'' , indicating structural breakdown occurred at a strain nearly five times higher for the vacuum-dried PEGDA hydrogel (34529 Pa) compared to the RT-PEGDA (7424 Pa). This suggests that the vacuum-dried hydrogel possesses a higher degree of crosslinking and structural resilience before yielding. The improved viscoelastic stability is likely derived from the uniform nanostructure formed under vacuum drying, which minimizes residual stresses and voids within the polymer matrix. These findings are consistent with previous studies, which report that denser crosslinked networks and smaller particle dimensions enhance hydrogel elasticity, resistance to deformation, and overall mechanical robustness. Consequently, vacuum drying can be considered a more effective technique for fabricating mechanically stable PEGDA hydrogels suitable for solar vapor generation applications, where elasticity and structural durability are critical for repeated wet dry cycles.

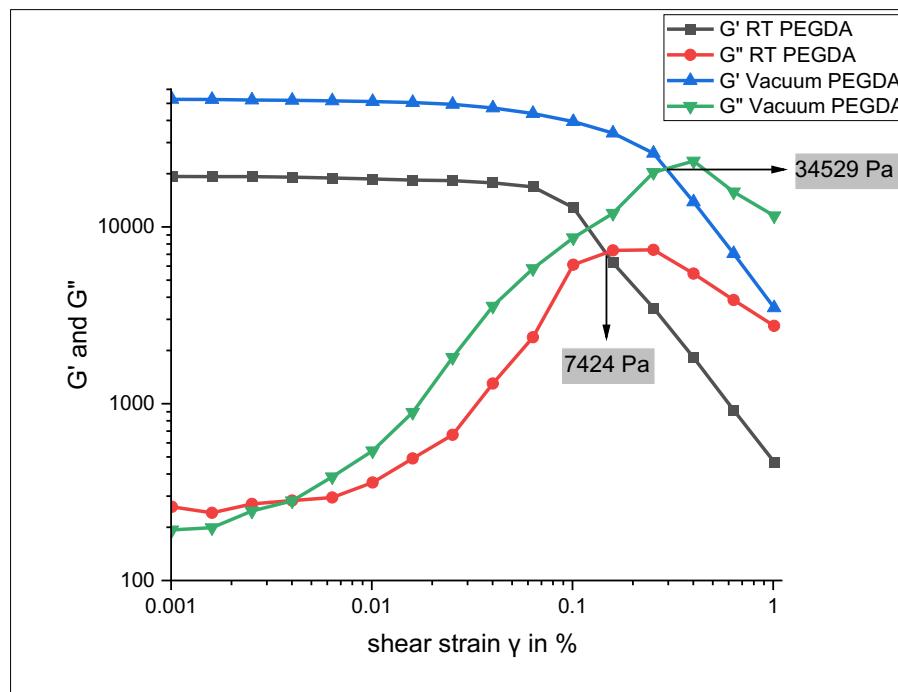


Fig. 6. Rheology studies for the amplitude sweep of PEGDA RT hydrogel and PEGDA vacuum hydrogel

4. Conclusions

This study demonstrates that the post-synthesis drying method is a critical and decisive parameter in fabricating PEGDA hydrogels with tailored properties. We found that vacuum drying is vastly superior to ambient temperature drying, as it effectively suppresses nanoparticle agglomeration and removes residual impurities. The low-pressure environment not only accelerated solvent evaporation but also minimized oxidation and residual contamination, resulting in PEGDA with well-preserved acrylate functionality. This improvement in nanostructural uniformity directly translated into denser crosslinking within the hydrogel matrix, indicating superior elastic recovery and mechanical robustness. Morphological analysis further confirmed that vacuum-dried PEGDA hydrogels exhibited smoother, defect-free surfaces compared to the rough, irregular topography observed in RT-dried samples. Such homogeneity reduces stress concentration and improves structural stability during cyclic hydration and evaporation, which are crucial for long-term operational durability in solar vapor generation systems. The implication of this finding is a direct, fundamental link between processing, nanostructure, and function. The nanoscale particles produced via vacuum drying result in a more homogeneous and densely cross-linked hydrogel network. This enhanced network structure translates directly into demonstrably superior mechanical and rheological performance, including a substantially higher Young's modulus and improved viscoelastic stability. These findings provide a clear processing guideline for researchers. By optimizing the drying step, it is possible to fabricate mechanically robust PEGDA hydrogels suitable for demanding applications, such as solar vapor generation, where structural integrity and long-term durability are paramount.

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