

Enhanced Physical Properties of TGS/GO-Infused Polyurethane Nanocomposites for Advanced Aerospace Applications

Mona A. Ahmed^{1*}, Hamdy M. Naguib¹, Ashraf M. El-Saeed¹, Emad Mousa², Eman O. Taha¹

¹Petroleum Applications Department, Egyptian Petroleum Research Institute (EPRI), Cairo, Egypt. ²Physics Department, Faculty of Science, Cairo University, Giza, Egypt

* Corresponding author: mona_chemist17@yahoo.com

Received 13 August 2024; revised 17 October 2024; accepted 01 November 2024

Abstract

In this study, polyurethane nanocomposites filled with varying ratios of triglycine sulfate (TGS) and graphene oxide (GO) were synthesized and evaluated for their potential use in aerospace applications. Thermogravimetric analysis (TGA) was employed to assess the thermal decomposition and stability of the nanocomposites. The key findings revealed that the inclusion of TGS and GO significantly enhanced the thermal stability, mechanical properties, and shape memory behavior of the nanocomposites. The sample with 1.5% TGS and 0.5% GO demonstrated the highest thermal stability, while the 1% TGS and 1% GO sample exhibited the fastest shape recovery time. These results highlight the potential of optimizing TGS and GO ratios for advanced aerospace materials, particularly in applications like satellite deployment.

Keywords: Polyurethane, Triglycine Sulfate, Graphene Oxide, Shape Memory Properties, Aerospace Applications

1. Introduction

Shape memory polymers (SMPs) are materials capable of "remembering" a permanent shape through covalent bonds or physical crosslinking. A temporary shape, in a non-equilibrium state, can be stabilized through mechanisms like crystallization, vitrification, reversible crosslinking, or other processes (Lendlein & Kelch, 2002a). SMPs retain this temporary shape until triggered by external stimuli, such as heat, light, pH changes, interaction with specific cells, or other environmental factors.

Heat is one of the most widely used stimuli in SMP applications, where the process involves heating the polymer above a transition temperature, such as the melting temperature or glass transition

temperature, deforming it into a desired shape, and then cooling it below this transition temperature (C. Liu et al., 2007). The polymer holds its temporary shape due to switching units, which prevent immediate recovery of the original form, but when reheated above transition temperature, the material returns to its permanent shape due to stable network points (C. Liu et al., 2007).

Recently, SMPs have emerged as a transformative material in aerospace engineering, offering a unique combination of lightweight properties, adaptability, and mechanical robustness(Tandon et al., 2015). These polymers possess the remarkable ability to undergo programmed deformations and then recover their original shape when exposed to external stimuli, such as temperature changes or light. This capability has opened new avenues for innovation in aerospace applications, including deployable structures, morphing airframes, and self-healing components. SMPs provide significant advantages over traditional materials, reducing the overall weight of aerospace systems while enhancing their functionality and resilience(Y. Liu et al., 2014; Tandon et al., 2015).

Additionally, the tunable nature of SMPs allows engineers to optimize their performance under extreme environmental conditions, such as varying atmospheric pressures and temperatures, making them highly suitable for space exploration, satellites, and next-generation aircraft(Keihl et al., 2005).

In recent studies, it has been observed that some polyurethanes demonstrate intriguing shapememory characteristics(Ahmed et al., 2015; Gupta et al., 2019). Polyurethane (PU) represents a significant category of polymers, characterized by alternating hard and soft segments. Over the past decade, extensive research has been conducted on the morphology, crystalline structure, thermal characteristics, deformation mechanisms, and rheological properties of various types of PU. Polyurethane-based nanocomposites, particularly those infused with novel fillers, have garnered significant attention due to their versatility and wide range of applications in sectors like aerospace, automotive, and electronics. In recent years, the incorporation of carbonaceous nanomaterials into polyurethane matrices has been shown to significantly improve the thermal stability, mechanical strength, and dynamic mechanical properties of these materials, making them suitable candidates for aerospace applications(Huang et al., 2010, 2012). Fonseca et al. (L. Fonseca et al., 2018)studied polyurethane (PU) nanocomposites filled with carbon nanotubes (CNTs) and graphene nanosheets. Morphological, thermal, and mechanical characterizations were performed, showing that the addition of carbon-based nanofillers improved the thermal conductivity, heat transfer, and mechanical strength of the nanocomposites, Graphene, in particular, demonstrated the most promising mechanical reinforcement, while non-treated CNTs showed better overall performance compared to treated CNTs. The study concluded that these fillers, especially graphene, significantly enhance the shape-memory capabilities of PU. Thiyagu et al.(Thiyagu & NarendraKumar, 2022) studied the effect of graphene (Gr) on the thermal, mechanical, and shape memory properties of polyurethane (PU) nanocomposites. The incorporation of graphene significantly enhanced the thermal stability and mechanical strength of the nanocomposites, with a 59% improvement in tensile modulus and a 12% increase in tensile strength observed. Thermogravimetric analysis revealed that graphene improved the degradation temperature of the nanocomposites. The shape memory properties were maintained despite the improved mechanical properties, although a slight reduction in the shape recovery ratio was noted with increasing graphene content. The study concluded that the addition of graphene to PU enhanced the thermal and mechanical properties without adversely affecting its shape memory behavior. Dehaghani et al(Zarghami Dehaghani et al., 2020) studied shape memory thin films of polyurethane (PU) nanocomposites incorporating graphene nanoplatelets (GNPs) with varying GNP content (0.1 to 1 wt.%). The addition of GNPs was shown to improve the storage modulus and shape memory

properties, particularly at 0.25 wt.% GNP content, which exhibited the highest shape recovery ratio of 92.4%.

Triglycine sulfate (TGS) is well-known for its ferroelectric properties, which have been widely studied and exploited in various applications such as infrared detectors and memory devices (Bose et al., 2012). When incorporated into polymer matrices, TGS can introduce pyroelectric and piezoelectric characteristics, which are desirable for aerospace materials requiring high sensitivity to temperature changes and the ability to convert mechanical energy into electrical energy (Plyushch et al., 2019). However, despite its functional benefits, TGS tends to exhibit limitations in thermal stability, which necessitates further optimization when used in high-temperature environments.

In this study, we explore the development and characterization of polyurethane nanocomposites infused with varying ratios of TGS and GO. The focus is on evaluating their thermal stability, dynamic mechanical properties, and potential applications as shape-memory materials for aerospace structures. By optimizing the TGS/GO ratio, we aim to achieve a balance between thermal performance and mechanical flexibility, ultimately enhancing the functionality and reliability of materials used in satellite deployment mechanisms and other critical aerospace components. In Egypt, the aerospace sector is experiencing significant growth, with an increasing demand for advanced materials that offer lightweight properties and the ability to withstand harsh space conditions. The development of nanocomposites, such as polyurethane infused with triglycine sulfate and graphene oxide, is crucial for supporting Egypt's emerging space projects. Utilizing these materials in local aerospace applications not only enhances technological capabilities but also contributes to the country's ambition to participate in global space exploration initiatives.

2. Experimental:

2.1. Methods:

1- Synthesis of Graphene Oxide (GO) and Triglycine Sulfate (TGS):

Graphene oxide (GO) was synthesized using an improved Hummer's method (Taha et al., 2022). Graphite was mixed with a 3:1 ratio of sulfuric acid and phosphoric acid, followed by the gradual addition of potassium permanganate. After stirring for 24 hours at elevated temperatures, the reaction was stopped with hydrogen peroxide, and the resulting GO was washed with hydrochloric acid and distilled water until the pH reached three.

For the synthesis of TGS, glycine was mixed with concentrated sulfuric acid (Analar grade) in a 3:1 molar ratio and dissolved in deionized water at 50°C to create a saturated solution. TGS crystals were subsequently formed through natural evaporation at room temperature.

2- Synthesis of TGS/GO-Infused Polyurethane Nanocomposites:

In-situ polymerization was used to prepare the TGS/GO-infused polyurethane nanocomposites. The appropriate amount of TGS/GO was first dispersed in xylene using ultrasonic treatment at 22 kHz (UZD-22/44, Ukrrospribor, Ukraine) for 5 minutes. This dispersion was then incorporated into the polyurethane (PU) matrix for an additional 15 minutes. A hardener was added at a base-to-hardener ratio of 9:1. TGS and GO were introduced into the PU matrix in ratios of 2%:0, 1%:1% and 1.5%:0.5%. The nanocomposite films were cast on Teflon substrates, with initial drying conducted at 25°C for 48 hours, followed by further drying for three days under the same conditions.

2.2. Characterizations:

Thermogravimetric analysis (TGA, Mettler Toledo) was performed from room temperature up to 700°C under a nitrogen atmosphere, utilizing a heating rate of 10°C per minute. A mechanical analyzer (MA, Triton Instruments) was employed to assess the mechanical properties in tension mode. Dynamic properties were examined at a frequency of 1 Hz, with the test conducted over a temperature range of 30 to 140°C, using a scanning rate of 5°C per minute.

To examine the shape memory behavior of TGS/GO-infused polyurethane nanocomposites, the analysis followed a bending test protocol based on the methodology by Lendlein and Kelch(Lendlein & Kelch, 2002b), where the polyurethane nanocomposites samples were twisted above their glass transition temperature (T_g) to an initial angle, denoted as θ_0 . The samples were then cooled to 0°C while maintaining the deformation under an external force to achieve a deformed angle, θ_i . Once cooled, the samples were immersed in water at a temperature above T_g , triggering the recovery of the material's original shape. Throughout this process, the variation in the recovery angle (θ_f) was recorded over time to monitor the shape recovery.

Two key ratios were used to quantify the shape memory behavior:

- The recovery ratio (R_r) , defined as $(\theta_i$ $\theta_f)$ / θ_i , measured the material's ability to return to its original shape.
- The fixity ratio (R_f), evaluated the extent to which the material retained the deformed shape after cooling.

3. Results and Discussion

3.1. Thermal Gravimetric Analysis (TGA)

Figure 1 (a, b) represents thermogravimetric analysis (TGA) and derivative thermogravimetric analysis (DTG), which provide critical insights into the thermal stability and decomposition behavior of polyurethane (PU) nanocomposites infused with varying ratios of Triglycine Sulfate (TGS) and Graphene Oxide (GO). Figure 1 (a), the TGA curve shows three distinct stages of thermal behavior. Initial stability (1st stage): All samples maintain 100% of their weight up to approximately 170°C, reflecting no significant degradation during this phase. Primary Decomposition (2nd stage): Between 170°C and 500°C, a sharp weight loss is observed, corresponding to the thermal degradation of the polymer matrix and additives. The TGA results indicate that the PU-1.5%TGS-0.5%GO sample demonstrates the highest thermal resistance, with an onset temperature (T_{onset}) of 189.51°C compared to 172.81°C for PU-2% TGS and 172.39 °C for PU-1% TGS-1% GO. The delayed onset of degradation suggests that the inclusion of GO enhances the thermal stability of the composite. Residual weight (3rd stage): After 500°C, minimal additional weight loss occurs. At 700°C, the PU-1.5%TGS-0.5%GO sample retains the highest residual weight (0.477%), indicating the formation of a more stable residue compared to PU-2% TGS (0.094%) and PU-1% TGS-1% GO (0.303%). This further underscores the positive effect of GO on the composite's thermal performance, as GO likely contributes to the formation of a thermally stable char residue.

Figure 1 (b), the DTG curve provides additional clarity by plotting the rate of weight loss (d(Weight)/d(Temperature)) as a function of temperature, identifying specific points of rapid thermal degradation. The peaks in the DTG curves correspond to different stages of thermal decomposition, allowing for a more detailed understanding of the degradation mechanisms. First decomposition peak:

The initial degradation, associated with the breakdown of low-molecular-weight components, occurs at around 246-248°C for all samples. The similarity in the first peak positions suggests that the initial thermal degradation mechanisms are not significantly influenced by the presence of GO. Main decomposition peak: The second peak, representing the major degradation of the polyurethane matrix, occurs between 430°C and 435°C. The GO-containing samples (PU-1%TGS-1%GO and PU-1.5%TGS-0.5%GO) exhibit slightly higher peak temperatures compared to PU-2%TGS, indicating improved thermal stability. This result aligns with previous research, which has shown that GO enhances the thermal resistance of polymer composites by acting as a thermal barrier and promoting the formation of a protective char layer(Suthar et al., 2022; Wang et al., 2011). Final decomposition peak: The third peak, representing the final stage of decomposition, occurs between 469°C and 487°C. The PU-1%TGS-1%GO sample demonstrates the highest final peak temperature (486.53°C), while the PU-1.5%TGS-0.5%GO sample exhibits a lower peak (469.24°C). These results suggest that different TGS and GO ratios influence the degradation pathways and the thermal stability of the residual material.

Both TGA and DTG analyses confirm that the inclusion of GO in the nanocomposite formulation enhances thermal stability. The PU-1.5% TGS-0.5% GO sample demonstrates the highest overall thermal resistance, with delayed onset of degradation, higher peak decomposition temperatures, and a larger residual weight. The synergistic effects of TGS and GO provide improved thermal stability by reinforcing the polymer matrix and promoting the formation of a stable char residue during decomposition.

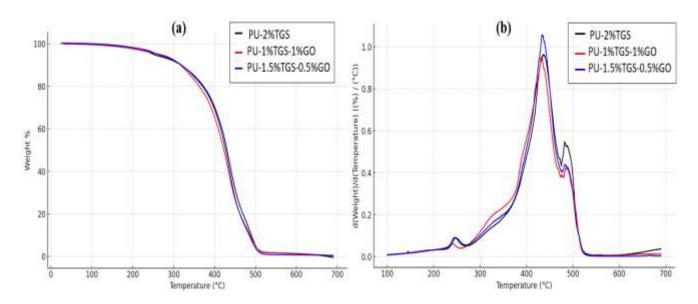


Figure 1 (a, b): thermogravimetric analysis (TGA) and derivative thermogravimetric analysis (DTG) of TGS/GO-Polyurethane nanocomposites.

3.2. Dynamic Mechanical Analysis (DMA)

Figure 2 and 3 represent Dynamic Mechanical Analysis (DMA) results of polyurethane (PU) nanocomposites infused with varying ratios of Triglycine Sulfate (TGS) and Graphene Oxide (GO).

The DMA is a powerful technique for understanding the viscoelastic behavior of materials as a function of temperature(AbdelKader et al., 2024; El-Deeb et al., 2022).

Figure 2 shows the storage modulus (GPa) of polyurethane (PU) nanocomposite samples as a function of temperature, which represents the stiffness of a material and its ability to store mechanical energy. The storage modulus shows three regions as the temperature increases. Initial high modulus region (Below 40°C): All samples exhibit a high storage modulus in the glassy region (low temperature), indicating that the materials are stiff and rigid at lower temperatures. The PU-1.5% TGS-0.5% GO sample shows the highest modulus, suggesting superior stiffness, while PU-2% TGS exhibits the lowest modulus. The inclusion of GO in the nanocomposite matrix improves the stiffness, as seen with the samples containing GO. Modulus drop region (40°C to 80°C): As the temperature increases, a sharp decline in the storage modulus is observed, marking the transition from the glassy to the rubbery state. This reduction in modulus is associated with increased molecular mobility as the material approaches its glass transition temperature (T_g). The PU-1.5% TGS-0.5% GO sample retains a higher modulus throughout this transition compared to the other samples, indicating that the addition of GO enhances the material's mechanical integrity even as it softens. Rubbery plateau region (Above 80°C): Beyond 80°C, the modulus stabilizes, reflecting the material's behavior in the rubbery state. Here, the PU-1%TGS-1%GO sample shows a higher modulus than the other two samples, suggesting that it maintains better structural integrity at higher temperatures, likely due to the balanced ratio of TGS and GO.

Figure 3 shows the Tan δ (damping factor) of polyurethane (PU) nanocomposite samples as a function of temperature, which provides insights into the damping behavior and the glass transition temperature (Tg) of the material. Tan δ is the ratio of the loss modulus (energy dissipated as heat) to the storage modulus (energy stored), and peaks in the tan δ curve correspond to the Tg of the material(Taha et al., 2023; Taha & Nasr, 2021). The PU-1.5% TGS-0.5% GO sample exhibits the highest Tg (52.8°C), indicating that the incorporation of 0.5% GO raises the temperature at which the polymer transitions from a glassy to a rubbery state. The PU-1% TGS-1% GO sample has the lowest Tg (49.4°C) compering to PU-2% TGS (52.4°C), suggesting that the equal ratio of TGS and GO decreases the Tg slightly, likely due to enhanced molecular mobility facilitated by the presence of GO. The intensity of the tan δ peak correlates with the material's damping ability. A higher tan δ value suggests more energy dissipation (damping). The PU-1.5% TGS0.5% GO sample exhibits the highest tan δ peak, reflecting greater damping capacity. This indicates that this sample can better dissipate mechanical energy, which could be beneficial for applications requiring high energy absorption.

The addition of GO significantly enhances the mechanical properties of the nanocomposites, as seen by the higher storage modulus and increased T_g for the GO-containing samples. GO acts as a reinforcing agent, improving the stiffness and thermal stability of the material. The T_g values suggest that varying the TGS/GO ratio has a significant effect on the material's thermal behavior, with the highest T_g observed in the PU-1.5%TGS-0.5%GO sample. Also, the damping behavior of the materials, indicated by the tan δ plot, reveals that the inclusion of GO enhances the material's ability to dissipate mechanical energy, which could be beneficial in applications requiring high impact resistance or vibration damping.

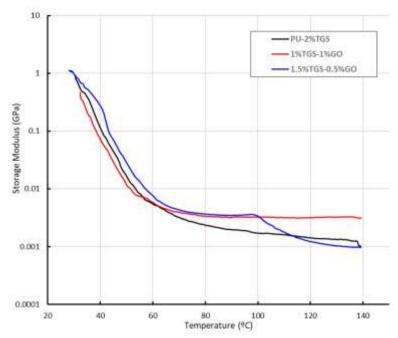


Figure 2: Storage modulus of TGS/GO-Polyurethane nanocomposites.

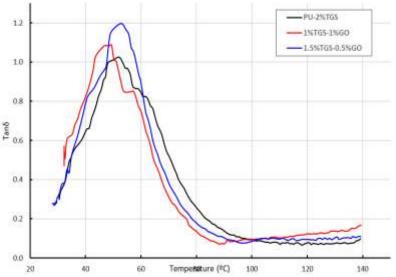


Figure 3: Tan δ (damping factor) of TGS/GO-Polyurethane nanocomposites.

3.3. Shape Memory Behavior Analysis:

Figure 4 represents the shape recovery ratio (%) as a function of time for polyurethane (PU) nanocomposites infused with varying ratios of Triglycine Sulfate (TGS) and Graphene Oxide (GO). Shape memory polymers (SMPs) like these are capable of returning to their original shape after deformation, triggered by external stimuli such as temperature(Lotfy & Basfar, 2023). The shape recovery ratio quantifies the percentage of deformation recovery over time. The PU-1%TGS-1%GO

sample achieves 100% shape recovery in approximately 9 sec., indicating that an equal distribution of TGS and GO enhances the activation and recovery process of the polymer matrix. The PU-1.5%TGS-0.5%GO sample exhibits the slowest recovery, reaching 100% recovery after 13 sec. The reduced GO content in this sample slowing down the shape recovery process. GO significantly improves the recovery speed, as seen in the PU-1%TGS-1%GO sample. GO enhances the thermal conductivity of the polymer matrix, enabling a faster response to heat stimuli and increasing recovery efficiency. The superior mechanical performance is also linked to GO's reinforcement of the matrix, making the recovery process smoother and more efficient. The PU-1.5%TGS-0.5%GO sample, despite having a slower recovery, still maintains a smooth and consistent shape recovery curve. The lower GO content results in less thermal reinforcement, but the material still demonstrates effective shape memory characteristics, just at a slower rate.

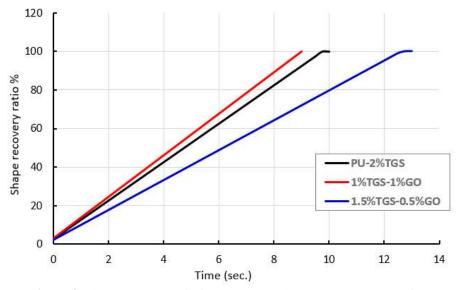


Figure 4: Shape recovery ratio for TGS/GO-Polyurethane nanocomposites.

Figure 5 displays the fixity ratios of polyurethane (PU) nanocomposites infused with varying amounts of Triglycine Sulfate (TGS) and Graphene Oxide (GO). The fixity ratio across all samples consistently remains at 100%, demonstrating the materials' ability to effectively retain their deformed shapes during cooling. This consistency suggests that the incorporation of TGS and GO enhances the shape memory properties without compromising the deformation retention capability of the PU matrix. These findings highlight the importance of optimizing the TGS and GO concentrations to achieve superior shape memory performance, especially for applications requiring rapid response, such as in aerospace engineering.

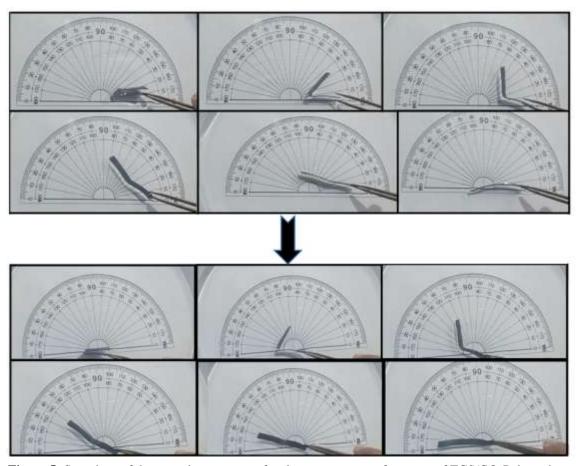


Figure 5: Snapshots of the repeating processes for shape recovery performance of TGS/GO-Polyurethane nanocomposites.

Ultimately, we conclude that this study's findings on the incorporation of triglycine sulfate (TGS) and graphene oxide (GO) into polyurethane nanocomposites demonstrate significant improvements in both thermal, mechanical and shape memory properties. These results align with previous research on the effectiveness of GO as a reinforcing agent in polymer matrices.

To further contextualize our findings, we compare them with existing literature. For example, Kausar et al. (Kausar, 2020) reported enhancements in mechanical and thermal properties with the addition of graphene oxide to polyurethane composites, which is consistent with the increased thermal stability observed in our study, particularly with the 1.5% TGS and 0.5% GO samples. Additionally, the shape memory behavior observed here aligns with the findings of Luo et al. (Luo et al., 2021), who showed similar improvements in shape recovery ratios through GO incorporation. The dynamic mechanical analysis (DMA) results from our study, which highlighted improved stiffness and a higher T_g in GO-containing samples, are supported by the work of Sofla et al. (Lotfi Mayan Sofla et al., 2019). Our research, however, offers new insights by optimizing the TGS and GO ratios for aerospace applications, thus contributing to the further development of advanced nanocomposites for specific industrial uses.

Future work will focus on expanding the current research by exploring the performance of these nanocomposites under extreme environmental conditions typical of aerospace applications. This will include investigating the long-term stability, durability, and adaptability of the materials in varying temperatures and pressures, with the aim of further optimizing the TGS/GO ratios for enhanced performance. Additionally, there will be a focus on integrating these nanocomposites into real-world aerospace systems to evaluate their practical utility in satellite deployment mechanisms and other structural components.

4. Conclusion

The incorporation of Triglycine Sulfate (TGS) and Graphene Oxide (GO) into polyurethane (PU) nanocomposites has proven to enhance the thermal and mechanical performance of these materials, making them suitable candidates for aerospace applications. The TGA results confirmed improved thermal stability, with the PU-1.5%TGS-0.5%GO sample exhibiting the highest residual weight and delayed onset of decomposition. Similarly, the DMA results demonstrated increased stiffness and thermal resistance in GO-containing samples, with the PU-1.5%TGS-0.5%GO sample showing the highest storage modulus and glass transition temperature. The shape memory analysis highlighted the ability of these nanocomposites to achieve efficient shape recovery, with the PU-1%TGS-1%GO sample achieving full recovery in the shortest time. Overall, this research underscores the importance of optimizing the TGS and GO ratios to maximize shape memory properties and thermal stability, further extending the potential of these materials for use in advanced aerospace systems.

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