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Research Article

Mechanical Robust and Conductive Polyurea Nanocomposites Using Graphene Platelets

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Abstract

Graphene, renowned for its exceptional surface area, electrical and thermal conductivity, and gas permeation resistance, serves as an excellent filler for enhancing the properties of polyurea (PUA). In this study, graphene platelets (GNPs) were mass-produced via thermal expansion of graphite intercalation compound followed by ultrasonic exfoliation. These GNPs were then incorporated into PUA using a straightforward mixing method to create PUA/GNPs composites. Characterization using SEM and a high resistivity meter revealed strong interfacial bonding between GNPs and the PUA matrix, facilitated by isophorone diisocyanate (IPDI) and Jeffamine D2000 (D2000). This robust interaction significantly improved the composites' performance. Notable enhancements in mechanical properties were observed: tensile strength increased by approximately 79% at 0.5 vol%, impact strength by 15.7% at 0.2 vol%, and tear strength by 30.6% at 0.5 vol%,. These improvements underscore the effectiveness of GNPs as reinforcing fillers, significantly boosting the durability and robustness of the PUA composites. Additionally, the study examined the effect of varying graphene content on the electrical properties of the composites, revealing substantial improvements in electrical conductivity. This research presents a practical strategy for developing high-performance PUA/GNPs composites, leveraging GNP's unique properties to enhance both mechanical and electrical characteristics. The study contributes valuable insights into the synthesis and property enhancement of GNPs nanocomposites, paving the way for further advancements in the field of multifunctional materials.

Keywords

High-Performance Polymer, Graphene, Nanocomposite, Polyurea

1. Introduction

Polyurea (PUA) is a widely utilized polymeric matrix [1-3], prized for its exceptional energy absorption and mechanical properties, making it suitable for various applications [4, 5]. Despite its inherent strengths, polyurea, like many other polymers, often requires reinforcement to further enhance its mechanical and physical properties, flexibility, processability, and cost-effectiveness [6-8]. To address these needs, a variety of fillers have been employed, including carbon-based materials, layered

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silicates, and fibers. The integration of fillers into polyurea matrices can significantly improve their performance [9, 10].

However, achieving the desired enhancements often necessitates high filler loadings. This results in composites that are not only dense and costly but also exhibit limited mechanical properties, which can counteract the benefits of reinforcement [11-14]. For instance, while carbon-based fillers like carbon black are commonly used due to their availability and reinforcing capabilities, their incorporation at high levels can lead to diminished flexibility and increased brittleness in the final composite. Moreover, the production and use of traditional carbon black fillers present environmental concerns. The manufacturing process of carbon black is associated with substantial CO_2 emissions and the generation of pollutant waste, posing a significant environmental burden. This highlights the need for more sustainable alternatives that can provide comparable or superior reinforcement while minimizing ecological impact.

To this end, innovative materials such as graphene platelets (GNPs) have emerged as promising alternatives [15-17]. Graphene, a two-dimensional material with remarkable chemical and physical properties [13, 18], has found numerous applications in electronic devices, energy storage, sensors, adsorption, and composites. Graphene has emerged as a promising nanofiller for improving the mechanical properties, thermal and electrical conductivity, and gas impermeability of composites [19]. Their high aspect ratio and unique structure enable significant enhancements in the composite's performance with minimal environmental footprint. Additionally, the utilization of advanced production techniques for GNPs, such as ultrasonic exfoliation of natural graphite, further mitigates environmental impact by reducing waste and energy consumption.

In recent years, graphene/elastomer composites have been prepared using various methods and graphene sources to enhance mechanical, electrical, and thermal properties. For instance, Kang et al. [20] reported the preparation of graphene/natural rubber composites with excellent mechanical properties by coagulating natural rubber latex in the presence of graphene. Smith et al. [21] dispersed reduced graphene oxide in rubber latex using a simple mixing method, leading to enhanced mechanical, electrical, and thermal properties of the rubber material. However, most of these composites were prepared by dispersing GO into natural rubber latex, followed by reduction, which introduced structural defects, unreduced oxygen functionalities, and aggregation during the in-situ reduction process, thereby reducing compatibility between natural rubber and graphene. Recently, Joseph et al. [22] successfully fabricated graphene dispersions through planetary ball milling and prepared graphene/natural rubber latex nanocomposites, demonstrating significant improvements in thermal and electrical conductivity.

In this study, we employ a previously developed method for the large-scale production of graphene platelets (GNPs) through thermal expansion of graphite intercalation compound followed by ultrasonic exfoliation. This technique enables the efficient and scalable production of high-quality GNPs, which are subsequently incorporated into a polyurea matrix using a straightforward mixing method. The resulting PUA/GNPs nanocomposites are then subjected to thorough characterization to evaluate their mechanical behavior and compare it to that of neat polyurea.

The incorporation of GNPs into the polyurea matrix is expected to enhance the composite's mechanical properties due to the unique characteristics of GNPs, such as its high surface area, exceptional mechanical strength, and excellent thermal and electrical conductivity. By varying the GNP loadings within the polyurea matrix, we aim to investigate how different concentrations of GNPs influence the curing characteristics and mechanical properties of the composites. This involves analyzing parameters such as tensile strength, elongation at break, impact strength, and tear strength to understand the reinforcement effects of GNPs.

To achieve this, we utilize scanning electron microscopy (SEM) and a high resistivity meter to characterize the structural and electrical properties of the PUA/GNPs composites. SEM imaging provides insights into the dispersion and distribution of GNPs within the polyurea matrix, while the high resistivity meter measures the electrical conductivity of the composites. These characterization techniques are crucial for understanding the interfacial interactions between GNPs and the polyurea matrix, which play a significant role in determining the overall performance of the nanocomposites.

The findings from this study indicate that the inclusion of GNPs significantly enhances the mechanical properties of the polyurea composites. For instance, the tensile strength of the PUA/GNPs composites increases by approximately 79%, while the elongation at break improves by 188%. Additionally, the impact strength and tear strength of the composites are enhanced by 15.7% and 30.6%, respectively.

Moreover, the study explores the electrical properties of the PUA/GNPs composites, revealing that the incorporation of GNPs leads to a marked improvement in electrical conductivity. This enhancement is particularly significant for applications requiring materials with both superior mechanical and electrical properties. The ability to tailor the properties of the composites by adjusting the GNP loadings offers a versatile approach to developing advanced materials for various applications, including electronics, structural materials, and protective coatings [23-25].

Overall, this research provides valuable insights into the potential of GNPs as a reinforcement filler in polyurea composites. By elucidating the structure-property relationships of PUA/GNPs nanocomposites, the study contributes to the development of high-performance materials with enhanced mechanical and electrical properties. The findings highlight the advantages of using GNPs over traditional fillers, such as carbon black, particularly in terms of environmental sustainability and performance efficiency. The successful integration of GNPs into polyurea matrices opens new avenues for the design and application of advanced composite materials, paving the way for innovations in various industries.

2. Experiment

2.1. Materials

Isophorone diisocyanate (IPDI) was sourced from Bayer, China, and Jeff-amine D2000 (D2000) was obtained from Huntsman, China. Diethyl toluene diamine (E-100) was acquired from Jinan NUOSHI New Materials Co., Ltd. All other chemicals were purchased from Sinopharm Chemical Reagent Co., Ltd. These chemicals were of analytical grade and used as received without any further purification.

2.2. Preparation

The preparation of PUA composites was carried out as follows: Initially, 1 g of GNPs was dispersed in DMF using sonication at room temperature. Subsequently, 5.55 g of IPDI was added to the mixture and stirred for 2 hours in a three-necked flask under a nitrogen atmosphere. After this, 20.00 g of D2000 was introduced into the flask, and the mixture was stirred at room temperature for another 2 hours. Next, 2.67 g of E-100 was added as a chain extender, and the stirring continued for an additional 3 hours. The resulting products were then dried at 80 ℃ for 24 hours. Figure 1 shows the illustration for the preparation of PUA nanocomposites.
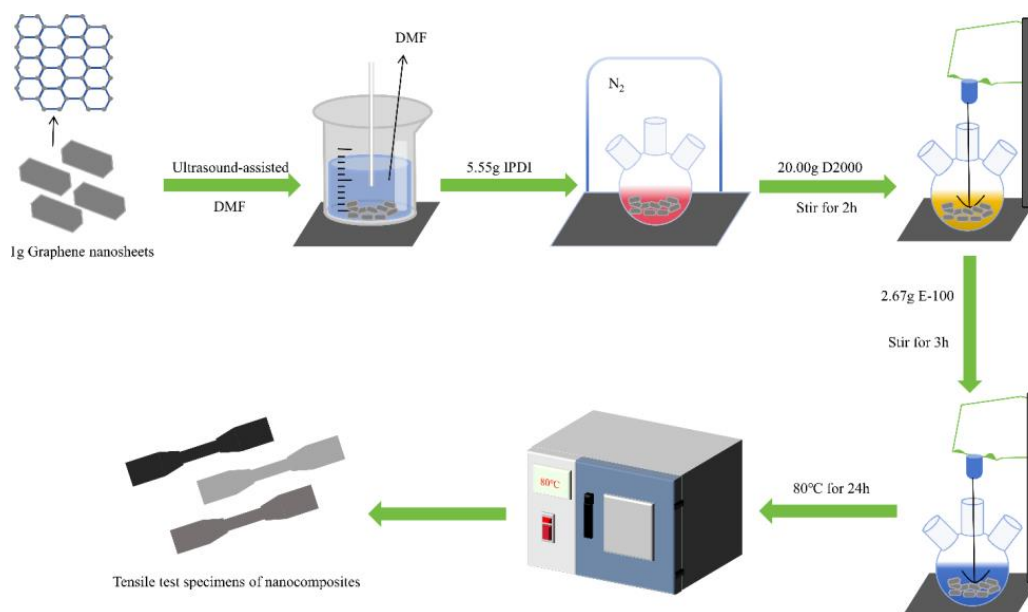
Figure 1. Illustration for the preparation of PUA nanocomposites.

2.3. Characterization

Transmission electron microscopy (TEM) using a Philips CM200 operating at 200 kV was utilized to investigate the morphology of GNPs. Prior to imaging, GNPs were dispersed in acetone via sonication and diluted to a concentration of 0.0004 wt%. A small volume of this dispersion was drop-cast onto 200-mesh copper grids and dried, ensuring a uniform dispersion for TEM analysis.

X-ray photoelectron spectroscopy (XPS) was performed using an ESCALAB 250 spectrometer equipped with Al Kα excitation radiation. This technique provided detailed information about the chemical composition and bonding states present within the GNPs and their interaction with the PUA matrix.

The microstructure of the composites was examined using scanning electron microscopy (SEM), specifically a JEOL JSM-7800F operating at 5 kV. To enhance imaging quality,

the sample surfaces were coated with a thin layer of platinum. SEM analysis allowed for visual inspection of the distribution and alignment of GNPs within the PUA matrix, providing insights into the structural integrity and interfacial interactions within the nanocomposites.

Mechanical properties of the nanocomposites were evaluated through several standardized tests. Tensile testing was performed using a dumbbell-shaped specimen at a speed of 200 mm/min according to ISO 37:2011, assessing parameters such as tensile strength and elongation at break. Tear strength was measured using a trouser test piece at a speed of 100 mm/min following ISO 34-1:2004, gauging the material's resistance to tearing forces. Impact resistance was assessed using a Charpy impact testing machine (KEBIAO) per ISO 179-1:2000, where PUA-coated samples on an aluminum substrate underwent impact testing with a 15 J pendulum at a velocity of 3.8 m/s.

Additionally, the electrical conductivity of the nanocomposites was characterized using a Keithley 6517B high resis-

tivity meter equipped with a model 8009 resistivity fixture. Measurements were conducted at room temperature on samples 2 ± 0.5 mm thick and 50 mm in diameter. This assessment provided crucial data on how the incorporation of GNPs influenced the electrical properties of the PUA matrix, highlighting potential applications in conductive coatings or electronic devices.

3. Result and Discussion

3.1. Microstructure of Graphene Platelets

Figure 2(a) illustrates a TEM micrograph depicting the morphology of multilayered graphene platelets, observed on a lacey carbon support. The image distinctly reveals the plate-like structure of the GNPs, highlighting their characteristic folded and wrinkled surfaces, indicative of 2 to 3 layers stacked together. This structural configuration is crucial as it impacts the mechanical and interfacial properties of GNPs when incorporated into polyurea matrices.

X-ray photoelectron spectroscopy (XPS) is a powerful analytical technique used to investigate the surface chemistry of materials. In the context of this study, XPS was employed to analyze the chemical composition of pristine GNPs and elucidate their interaction with polyurea. Figure 2(b) presents the XPS spectra of GNPs, showing characteristic peaks at binding energies of approximately 284 eV for C1s and 533 eV for O1s. These peaks correspond to the carbon and oxygen species present on the surface of GNPs, providing qualitative and quantitative insights into their chemical environment. The TEM and XPS analyses collectively contribute to a comprehensive characterization of GNPs for their application in polyurea nanocomposites. TEM micrographs offer visual confirmation of the morphology and layering of GNPs, cru-

cial for understanding their dispersion and alignment within the polyurea matrix. The folded and wrinkled surfaces observed in TEM suggest increased surface area and potential sites for interfacial interactions, which can influence mechanical reinforcement and interface bonding in the nanocomposites.

On the other hand, XPS spectra provide detailed chemical information about the surface composition of GNPs. The presence of carbon and oxygen species identified in the XPS spectra indicates the nature of functional groups and potential contaminants on the GNPs' surfaces. These surface characteristics are essential as they affect the compatibility and adhesion between GNPs and the polyurea matrix, thereby influencing the overall performance of the nanocomposites.

The integration of GNPs into polyurea matrices aims to enhance mechanical, electrical, and barrier properties for various applications. Mechanical tests, such as tensile, tear, and impact testing, evaluate the strength, elasticity, and toughness of the nanocomposites. Electrical conductivity measurements assess the suitability of GNPs for applications requiring conductive materials. Barrier properties are examined to understand the potential for GNPs to improve gas and moisture resistance in polyurea coatings and films.

Overall, the combined use of TEM and XPS provides a comprehensive understanding of GNPs' morphology, structure, and surface chemistry. This knowledge is pivotal for optimizing the fabrication process of PUA/GNPs nanocomposites and tailoring their properties for specific applications. Future research could focus on further enhancing the dispersion and alignment of GNPs within polyurea matrices, exploring novel functionalization techniques, and investigating the long-term durability and stability of these advanced materials in real-world conditions.
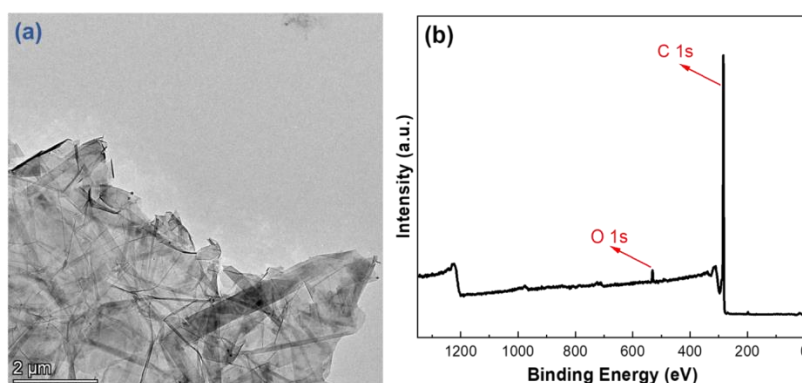

Figure 2. (a) TEM, (b) XPS of graphene platelets.

3.2. Morphologies of the PUA Nanocomposite

To investigate the interfacial interactions between

nano-additives and the PUA matrix, a detailed fractographic analysis was conducted using scanning electron microscopy (SEM). This analysis aimed to elucidate the impact of graphene nanoplatelets (GNPs) on the fracture characteristics of

PUA composites. The resulting SEM micrographs are presented in Figure 3.

The fractured surfaces of both the neat PUA and its nanocomposite variants were scrutinized. The neat polyurea film generally exhibited a smooth, featureless cross-sectional surface, which did not reveal significant structural details and thus, is not included in the analysis. In stark contrast, the nanocomposite films, especially those with higher filler fractions, displayed distinctive structural features on their cross-sectional SEM images, as shown in Figure 3. In Figure 3(a), the incorporation of 0.5 vol% GNPs into the PUA matrix notably increases the fracture roughness compared to the neat polyurea. The introduction of GNPs at this concentration enhances the roughness of the fracture surface, indicating a more complex and disrupted fracture path. This increased roughness is indicative of improved interfacial interactions between the GNPs and the PUA matrix, which likely contributes to better energy dissipation during fracture.

Figure 3(c) illustrates that increasing the GNP content to 1.0 vol% does not significantly alter the fracture roughness compared to the 0.5 vol% GNPs. This suggests that while 0.5 vol% GNPs effectively modify the fracture characteristics, further increases in GNP concentration up to 1.0 vol% do not produce additional significant changes in fracture surface morphology. The lack of significant improvement with higher GNP content might be attributed to the fact that at 1.0 vol%,

the GNPs are already well-dispersed and interacting effectively with the PUA matrix, and additional GNPs may lead to agglomeration rather than further enhancement.

The fracture surface at this concentration reveals a substantial number of voids and layer breakage, as shown in Figure 3(d). These features are characteristic of toughened PUA systems and indicate that the increased GNP content contributes to the formation of a more complex fracture network. The voids and layer breakage suggest that the GNPs are effectively engaged in energy absorption during fracture, thereby enhancing the toughness of the composite.

The observed fracture phenomena, including the presence of voids and layer breakage, are directly related to the dispersion and distribution of GNPs within the PUA matrix. The GNPs facilitate increased energy dissipation during fracture by creating a more intricate and disrupted fracture path. This results in improved toughness and mechanical performance of the nanocomposites.

In summary, the SEM analysis reveals that the incorporation of GNPs into the PUA matrix significantly affects the fracture characteristics of the composites. A GNP concentration of 0.5 vol% improves fracture roughness, indicative of enhanced interfacial interactions. While increasing the GNP content to 1.0 vol% does not further enhance fracture roughness, it maintains effective dispersion and interaction.
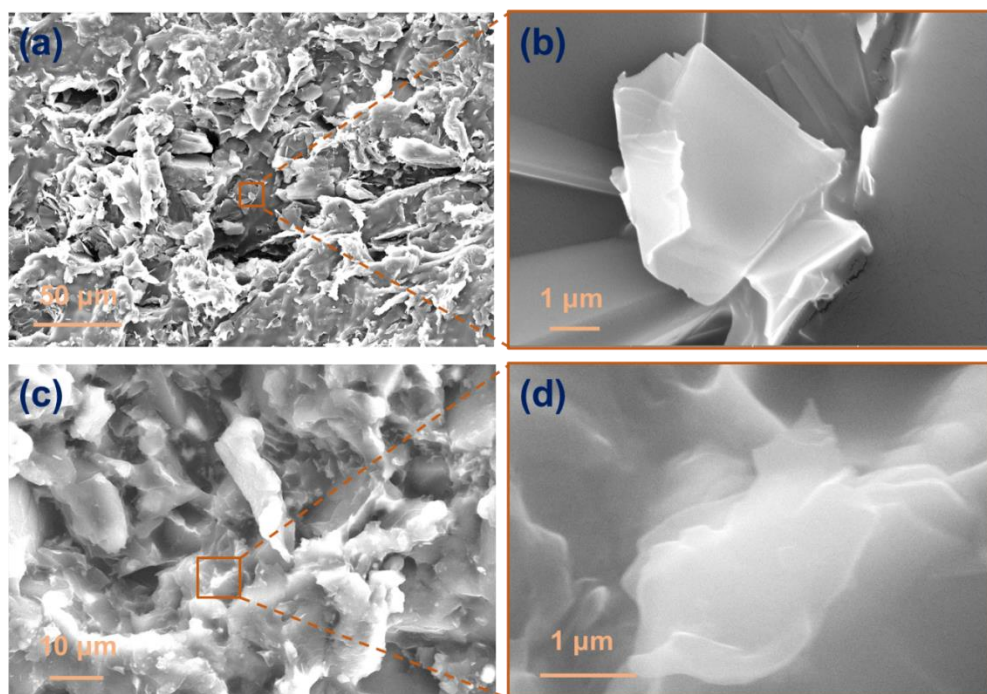
Figure 3. *SEM images of fracture surface for PUA/GNP nanocomposites: (a-b) 0.5 vol% and (c-d) 1 vol%.*

3.3. Mechanical Properties

The mechanical properties are crucial for evaluating elas-

tomer materials in various applications [23, 26, 27]. The mechanical properties of the PUA and its nanocomposites are depicted in Figure 4.

Figure 4(a) illustrates the changes in tensile strength of polyurea nanocomposites as the GNPs filler fractions increase from 0 vol% to 1 vol%. The maximum tensile strength of the polyurea nanocomposites reaches 32.4 MPa at a 0.5 vol% filler fraction, reflecting a 79% increase compared to pure polyurea. This enhancement is attributed to the GNPs' ability to offer a larger specific surface area and improved interfacial structure, which effectively mitigates stress concentrations and promotes stress transfer across the interface under loading. However, at a 1 vol% filler fraction, the maximum tensile strength is 8.33% lower than at 0.5 vol%. This reduction may result from the aggregation of graphene platelets at higher filler fractions, which diminishes the structural integrity and mechanical properties of the polyurea nanocomposites, thereby reducing their maximum tensile strength.

As shown in Figure 4(b), the elongation at break of the polyurea nanocomposites is shown with increasing graphene filler fractions. At a 0.2 vol% GNPs, the elongation at break increases to 589%. This significant improvement is due to the reinforcing effect of graphene on the polyurea matrix, which undergoes hardening and toughening as the nano-fillers ac-cumulate.

Comparing to neat PUA coatings on Aluminium alloy plate having an impact strength of 350 kJ/m^2, an improvement (15.7%) was seen when 0.2 vol% GNP was incorporated, as observed in Figure 4(c). This enhancement suggests that the GNPs are well dispersed within the polyurea matrix, providing a sufficient interface for effective load transfer from the matrix to the filler. In this scenario, GNPs impart a positive reinforcing effect, improving the low stiffness and poor impact toughness of polyurea at filler fractions less than 0.2vol%. However, further increasing the GNP content to 1.0vol% negatively impacts mechanical performance, resulting in an 8.1% reduction in impact strength compared to the 0.2 vol% GNP-filled nanocomposites. This decline in performance is attributed to poorer interfacial interactions between the GNPs and the polyurea matrix at higher filler fractions. These weaker interactions hinder effective stress transfer at the interfaces, leading to a decrease in impact strength. Additionally, excessive GNP content can immobilize the macromolecular chains, increasing the brittleness of the polyurea matrix and further reducing its impact strength.
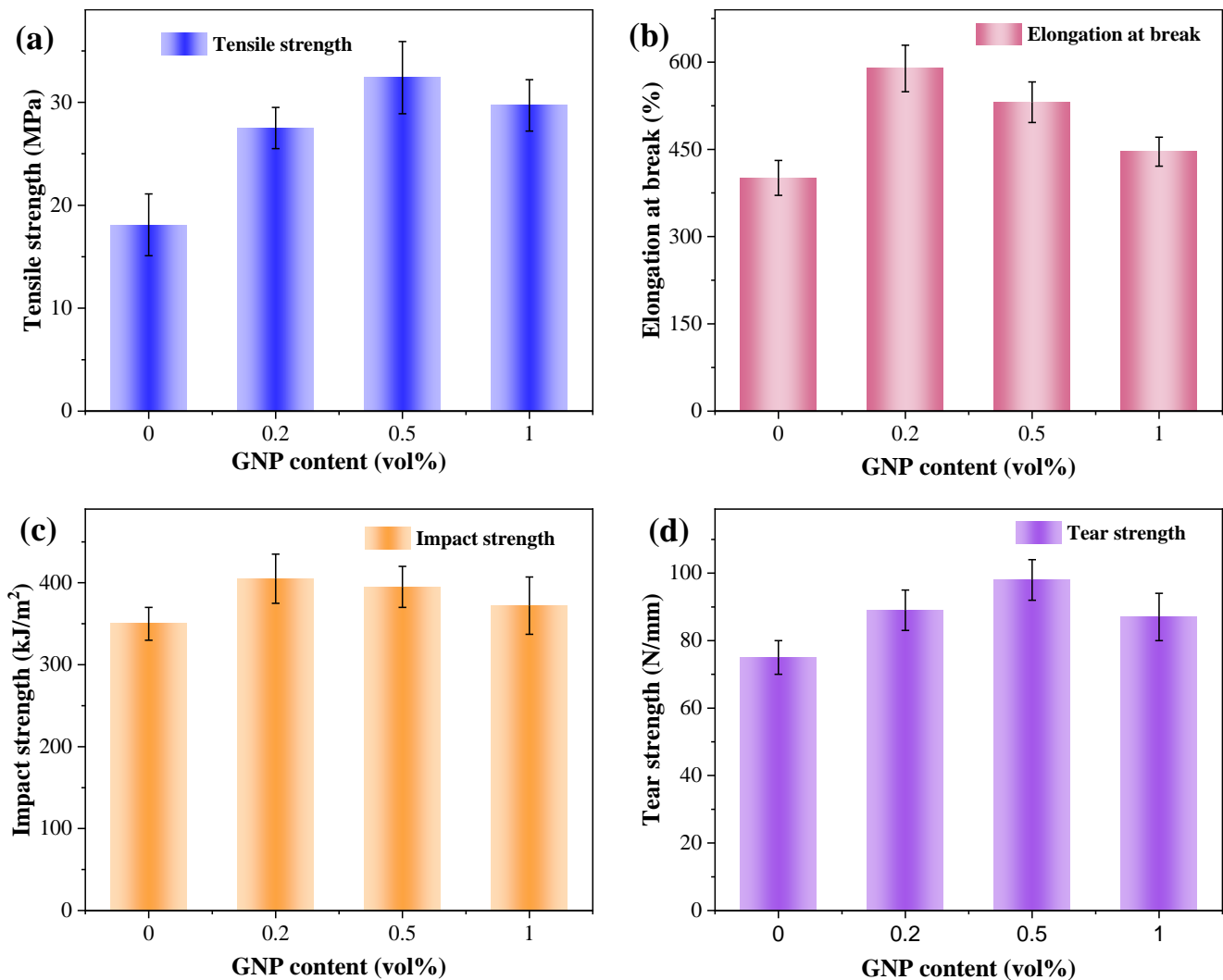

Figure 4. *(a) Tensile strength, (b) Elongation, (c) Impact strength and (d) Tear strength of PUA and its nanocomposite.*

As shown in Figure 4(d), the tear strength of the PUA/GNPs composite with 0.5 vol% GNPs content is approximately 98 N/mm, compared to 75 N/mm for the control sample. However, slight decreases in tear strength are observed when GNP loadings exceed 0.5 vol%. This indicates that incorporating a small amount of GNPs significantly enhances the tear strength of polyurea, reaching its peak at a 0.5 vol% GNP content. Further increases in GNP content beyond 0.5 vol% lead to changes in stress-induced crystallization (SIC). The crosslinking density of the PUA/GNPs composite rises with the addition of more GNPs. Higher crosslinking densities, exceeding 0.5 vol% GNPs, limit the extensibility of the network chains. The lateral confinement and adsorption effect of GNPs, coupled with increased crosslinking density, reduce the chain mobility of polyurea. This restriction hampers crystal lateral growth, resulting in decreased tensile strength when GNP content is above 0.5 vol%.

Through mechanical properties analysis, it was found that the sample with a filler content of 0.5 vol% obtained the best tensile strength and tear strength, better impact strength and elongation. Overall, the introduction of GNPs into polyurea significantly enhances its mechanical properties, with the optimal improvements observed at specific filler fractions. These enhancements are primarily due to the improved interfacial interactions and stress transfer capabilities provided by the GNPs, although excessive filler content can lead to aggregation and a subsequent decrease in performance.

3.4. Electrical Properties

Figure 5 illustrates the electrical conductivity of PUA/GNPs nanocomposites. Introducing 1.0 vol% GNPs significantly boosts the electrical conductivity from approximately 10^{-14} to around 10^{-10} S/cm. By fitting the experimental data into the power law equation, a percolation threshold of 1.15 vol% is identified, indicating the formation of a continuous conductive network for electron transfer. This low percolation threshold suggests two key points: (i) the GNPs are thin enough to mitigate the negative effects of poor through-plane conductivity, and (ii) the GNPs are likely to disperse uniformly within the matrix, making effective physical contact with each other.

For PUA/GNPs nanocomposites at 2.0 vol%, which is just beyond the percolation threshold, most GNPs are expected to connect intimately through tunneling or overlapping. This connection enhances both the sensitivity and stability of the composite. The nanocomposite film at this concentration exhibits a significant improvement in electrical conductivity ($\sim 10^{-7}$ S/cm). These improvements indicate the effectiveness of GNPs in enhancing both the mechanical and electrical properties of the polyurea matrix at higher filler loadings.
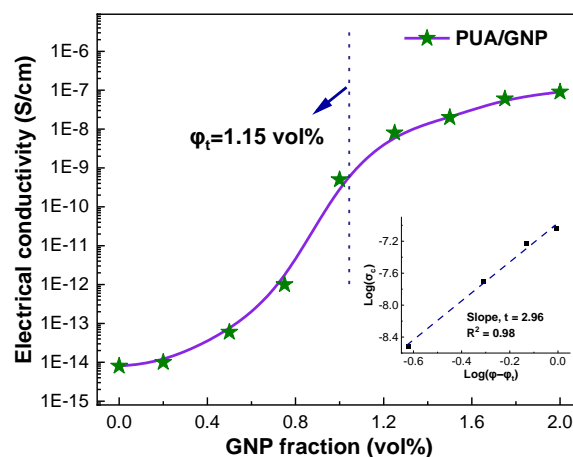

Figure 5. The electrical properties of PUA and its nanocomposites.

4. Conclusions

In this study, we explored the use of graphene platelets (GNPs) as fillers to enhance the electrical and mechanical properties of polyurea composites. The GNPs were obtained by ultrasonically exfoliating natural graphite and were then incorporated into the polyurea matrix through a straightforward mixing method. Utilizing IPDI and D2000, the graphene platelets were effectively integrated into the polyurea matrix, leading to a strong interface that significantly improved the mechanical properties of the composites. The results showed that the tensile strength, impact strength, and tear strength of the PUA/GNPs composites were enhanced by approximately 79%, 15.7%, and 30.6%, respectively. Additionally, the study examined the effect of graphene content on the electrical conductivity of the polyurea composites. Adjusting the graphene content resulted in a marked increase in electrical conductivity, highlighting the potential of these composites for applications that require conductivity. These findings add to the current understanding of PUA/GNPs nanocomposites and offer valuable insights into their synthesis and property improvements. The developed PUA/GNPs composites show significant promise for various industrial applications, including impact-resistant materials, protective coatings, and electronic devices, thereby contributing to advancements in the field of multifunctional materials.

Abbreviations

GNPs	Graphene Platelets
PUA	Polyurea
GO	Graphene Oxides
IPDI	Isophorone Diisocyanate

Acknowledgments

Author Contributions

Qiaoling Huang: Data curation, Writing – original draft

Shuang Guo: Funding acquisition, Methodology, Software, Supervision

Shuangshan Li: Data curation, Investigation, Supervision

Ziqi Gao: Data curation, Formal Analysis, Methodology

Shuning Ma: Conceptualization, Investigation

Sensen Han: Conceptualization, Project administration, Writing – review & editing

Conflicts of Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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