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Development and Characterization of Graphene Oxide and Natural Silk Fiber Reinforced Epoxy Composites for High-Performance Structural Applications

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Abstract: Epoxy **resin composites are widely utilized in structural applications due to their affordability, high specific strength, dimensional stability, and chemical resistance. However, their inherent brittleness and limited fracture toughness restrict their performance under demanding conditions. This study introduces a novel hybrid composite reinforced with graphene oxide (GO) and natural silk fibers (SF), leveraging their complementary properties to address these challenges. GO enhances tensile strength, thermal stability, and lightweight characteristics, while SF contributes toughness, flexibility, and biodegradability. The composite is fabricated using vacuum-assisted resin transfer molding (VARTM) and optimized ultrasonication parameters to ensure uniform reinforcement dispersion, robust fiber-matrix bonding, and reduced void content. These techniques mitigate GO agglomeration challenges and enhance scalability for industrial applications. Preliminary findings suggest significant improvements in mechanical strength, thermal stability, and environmental sustainability, validating the hybridization approach. The developed composites are projected to meet stringent requirements for aerospace, renewable energy, and automotive applications, offering a sustainable, high-performance alternative to traditional materials. While further mechanical and thermal testing is ongoing, this research establishes a robust foundation for advancing hybrid composites in demanding engineering environments.**

Keywords: **Graphene oxide, Natural silk fiber, epoxy composite, mechanical properties, thermal stability, sustainability, high-performance applications.**

I.INTRODUCTION

Hybrid composites are increasingly essential for meeting the demand for lightweight, durable, and high-performance materials across industries such as aerospace, automotive, power and renewable energy, and civil engineering [1-3]. Among advanced reinforcements, graphene oxide (GO) and natural silk fibers (SF) are particularly promising for epoxy resin composites due to their complementary properties. GO provides exceptional tensile strength, thermal conductivity, and a high surface area, enhancing mechanical and thermal performance [4,5], while SF offers toughness, biodegradability, and inherent flame retardancy, making it an eco-friendly alternative to synthetic fibers [6,7]. Integrating these materials into a hybrid composite structure addresses stringent industrial requirements for enhanced strength, thermal stability, and sustainability [8,9]. Despite these advantages, integrating GO and SF into a hybrid structure poses challenges, such as achieving uniform GO dispersion and robust fiber-matrix bonding [10]. Ultrasonication effectively mitigates GO agglomeration, ensuring homogeneous dispersion, while vacuum-assisted resin transfer molding (VARTM) addresses issues like inconsistent fiber volume fractions, void content, and resin flow. Conventional epoxy composites often suffer from brittleness, low impact resistance, and limited fracture toughness, restricting their applicability in demanding environments like renewable energy and aerospace [11]. Developing hybrid composites that address these limitations while maintaining environmental sustainability is critical for advancing material performance and aligning with sustainability goals [12].

This study leverages the complementary properties of graphene oxide (GO) and silk fibers (SF) to address the challenges of hybrid composite fabrication. It systematically investigates ultrasonication parameters (25°C, 30% amplitude for 2 hours) and carefully selects reinforcement ratios (GO: 0%–5%; SF: 40% and 60%) to optimize synergy and scalability for industrial applications. Advanced fabrication techniques, including vacuum-assisted resin transfer molding (VARTM) and ultrasonic-assisted dispersion, are employed to ensure uniform reinforcement dispersion, robust interfacial bonding, and scalability for large-scale production. These innovations result in a composite with significantly enhanced mechanical and thermal properties, paving the way for transformative applications in aerospace, automotive, renewable energy, and other high-performance sectors.

II.LITERATURE SURVEY

Recent advancements underscore the synergistic potential of combining graphene oxide (GO) and natural silk fibers in epoxy composites. This review highlights critical advancements and persistent challenges in GO and silk fiber composites, with a focus on optimizing mechanical properties, fire resistance, and environmental sustainability. By synthesizing insights from recent studies, the survey emphasizes the multifunctionality of these composites and their relevance to high-performance and sustainable material applications.

TABLE 1 SUMMARIZES SIGNIFICANT FINDINGS FROM RECENT STUDIES ON MECHANICAL, THERMAL, AND ENVIRONMENTAL PERFORMANCE ENHANCEMENTS ACHIEVED THROUGH GO AND SILK FIBER HYBRIDIZATION.

Year	**Authors**	**Focus of the research study**	**Key Findings**	**Critical Inference Noted**	**Ref.**
2010	Lee et al.	Mechanical properties of GO-epoxy	GO increased tensile strength by 40%, improved modulus, and enhanced stiffness of epoxy composites.	Uniform dispersion of GO is critical for achieving enhanced mechanical properties and minimizing defects.	[13]
2010	Yuchi Fan et al.	Graphene-Al2O3 composites	Graphene nanocomposites exhibited improved electrical properties.	Graphene enhances electrical conductivity, but interfacial bonding with the matrix requires optimization for stability.	[14]
2013	Hu et al.	GO-Silk Fibroin nanocomposites	Modulus, ultimate stress, and toughness of GOSF composites are superior to those reinforced with only graphene or silk.	GOSF composites leverage the synergistic effects of both reinforcements, achieving mechanical properties superior to either material used independently.	[15]
2015	Zhu & Zhang	Thermal stability of GO-reinforced epoxy	GO addition increased thermal conductivity by 25%, enhancing stability in high-temperature environments.	GO significantly improves thermal conductivity, enabling epoxy composites to function effectively in high-temperature conditions.	[16]
2016	Yang et al.	Silk fiber-reinforced epoxy composites	Silk fibers improved impact resistance and toughness, providing resilience to high-impact forces.	Silk fibers are effective for natural reinforcement, but moisture control and surface treatment are essential for maximizing toughness	[17]
2016	Yang et al.	High-volume silk fabric-reinforced composites	Carbon, glass, and silk fibers provided higher impact strength. Ap silk variant at 60% vol. showed tensile and flexural strength 3× and impact strength 8× over pure epoxy.	Silk permits higher volume fractions (30-70%) for improved matrix toughness. Toughening effects depend on the silk species used for reinforcement	[18]
2016	Nash et al.	Thermoplastic phase in CFRP composites	Improved impact and post-impact performance with thermoplastic phase inclusion in CFRP composites.	Thermoplastic phases in CFRPs enhance toughness and impact resistance, providing better performance in dynamic applications.	[19]
2016	Pathak et al.	Mechanical properties of GO-carbon fiber composites	Flexural strength and Young's modulus increased by 66% and 72%, respectively. Interlaminar shear strength (ILSS) increased by 25% at 0.3% GO addition due to interlocking and bonding.	Homogeneous dispersion of fillers is critical to avoid aggregates that act as failure points. Strengthening matrix-filler bonding improves stress transfer efficiency.	[20]
2017	Shankar et al.	Graphene and carbon fiber in polymer composites	Ultimate load increased by 64% for graphene and carbon fiber-reinforced epoxy composites compared to plain epoxy. The load distribution improved due to the high surface area of the fillers.	Reinforcement with graphene and carbon fibers leads to significant load distribution improvements due to the high surface area of fillers.	[21]
2018	Ahmed et al.	Moisture resistance of GO composites	GO reduced moisture absorption in epoxy, increasing durability in humid environments.	GO's hydrophobic properties enhance composite durability, making it suitable for humid or wet environments.	[22]
2018	Gupta et al.	Hybrid GO-silk fiber epoxy composites	The GO-silk combination increased tensile strength by 35% and flexural modulus by 30%.	Synergistic effects of GO and SF enhance composite performance, but agglomeration & bonding issues must be addressed.	[23]
2018	Youssef et al.	Silk/Epoxy composite	Flexural strength increased by	Balanced weave patterns and silk surface	[24]

		laminates	23%; specific flexural modulus increased by 41% (x-direction) and 104% (y-direction). Moisture at 10% caused a 25% drop in specific flexural strength and 20% reduction in modulus.	treatment are critical for improving silk/epoxy interfacial adhesion and mechanical properties. Moisture control before fabrication is essential.	
2019	Wu et al.	Mechanical properties of silk resin epoxy composites	Hybridization increased modulus and strength under tensile and flexural loading with progressive flax fiber content (50% total fiber volume). Interlaminar shear strength was enhanced due to improved z-direction stiffness.	Silk prevents crack propagation; flax fibers enhance impact strength and improve impact energy absorption efficiency.	[25]
2019	Chiou et al.	Effects of GNP and NCA in carbon fiber composites	Tensile, flexural, and impact strength improved with 1% GNP/NCA addition. Crack suppression due to NCA and crack deflection by GNP were noted. Synergistic effects enhanced mechanical properties.	Combined reinforcement with GNP and NCA is effective in improving mechanical strength and arresting crack propagation, ensuring better composite durability.	[26]
2020	Mehra & Yadav	Durability of GO-silk composites	GO and silk fibers together improved fracture toughness and wear resistance of epoxy composites.	The combined action of silk and GO strengthens fracture toughness and durability, particularly under dynamic loads.	[27]
2020	Park et al.	Fire resistance of GO-silk composites	Improved interfacial bonding in GO-silk composites delayed flame spread and reduced combustibility.	GO and silk synergistically improve flame resistance while maintaining structural integrity during exposure to fire.	[28]
2021	Singh et al.	Electrical conductivity of GO-epoxy	Uniform dispersion of GO improved electrical conductivity and provided mild thermal conductivity benefits.	Dispersion uniformity is critical for achieving electrical and thermal enhancements in GO-reinforced composites.	[29]
2022	Gupta & Kumar	Stability of silk-based composites	Silk fibers provided natural resistance to environmental degradation, supporting sustainable applications.	Silk's inherent environmental resilience enhances the long-term usability of sustainable composites in harsh conditions.	[30]
2023	Zhang et al.	Wear resistance in hybrid composites	GO’s hardness and silk’s resilience contributed to improved wear durability under dynamic stress conditions.	GO and silk improve wear resistance under repeated loading cycles, making them suitable for structural and dynamic applications.	[31]
2023	Chen & Kumar	Curing and mechanical properties of GO-silk composites	Advanced curing processes improved bonding and enhanced tensile and flexural strength.	Optimized curing conditions ensure uniform reinforcement dispersion and strong interfacial bonding between GO and silk fibers.	[32]
2024	Wang et al.	Fire resistance in GO-silk composites	High fire resistance due to GO’s char-forming ability and silk’s flame-retardant properties.	The combination of GO and silk fibers in composites provides exceptional fire resistance and reduces combustion impact.	[33]
2024	Wang et al.	Hybrid properties of GO-silk composites	GO improved thermal conductivity and offered limited electrical conductivity, useful for multifunctional applications.	GO’s multifunctionality enhances both thermal and limited electrical conductivity, providing broader composite functionality.	[34]
2024	Patel & Chawla	High-Performance Hybrid Composites with GO and Silk Fibers	GO improved mechanical and thermal properties, while silk fibers increased flexibility and toughness.	Hybrid systems enable a balance of strength and toughness for multi-functional applications.	[35]
2024	Gupta & Patel	Advances in GO-Silk Composites	Novel curing strategies enhanced hybrid properties,	Advanced fabrication processes ensure synergy	[36]

			ensuring long-term stability under stress.	between silk and GO, resulting in durable composites for high-performance use.	
2024	Kumar & Singh	Eco-Friendly Silk Reinforced Systems	Silk-based composites exhibited flame resistance and biodegradability.	Combines sustainability with flame-retardant properties for safer material applications.	[37]

A. Summary and Key Findings

The findings from this study emphasize the synergistic effects of combining graphene oxide (GO) and natural silk fibers (SF) in epoxy composites, paving the way for developing multifunctional materials with superior mechanical and thermal properties.

a) Mechanical Properties:

- GO's Contribution: GO addition up to 1% enhances tensile strength, modulus, and thermal conductivity (by over 25%) due to its high surface area and efficient load transfer. However, beyond 1%, agglomeration and void formation degrade performance [13, 19, 25, 26].
- Silk Fiber's Contribution: Silk fibers at higher volume fractions (30–70%) enhance toughness and impact resistance. A 60% SF volume fraction demonstrated tensile and flexural strengths three times higher, and impact strength eight times higher than neat epoxy [23].
- Hybridization Benefits: Combining GO and SF enhances fracture toughness, wear resistance, and energy absorption, making the hybrid composites ideal for high-stress applications [25, 26].

b) Thermal Stability and Fire Resistance:

- GO and Silk Synergy: GO's char-forming ability and SF's flame-retardant properties synergistically enhance fire resistance, reducing combustibility and delaying flame spread [28, 33].
- Volume Fraction Balance: Balancing GO and SF volume fractions is critical for achieving thermal stability without sacrificing mechanical performance [23, 25].

c) Fabrication Techniques:

- Uniform Dispersion: Techniques such as ultrasonic-assisted mixing and vacuum-assisted resin transfer molding (VARTM) ensure uniform reinforcement distribution and mitigate challenges like GO agglomeration [15, 20].
- Scalability and Process Efficiency: VARTM offers advantages over traditional wet layup by reducing void content, achieving consistent fiber volume fractions, and enabling large-scale fabrication. Staged curing improves reinforcement distribution and interfacial bonding [14, 31, 32].

d) Environmental Sustainability:

- Eco-Friendly Reinforcements: SF biodegradability supports sustainability goals, while GO enhances durability for long-term use [22, 30].
- High-Volume Silk Utilization: Silk's compatibility with high volume fractions broadens its application in renewable energy and structural sectors [23].

B. Research Gaps and Challenges

Despite significant advancements, notable gaps remain in the development of GO-SF composites. Current research largely emphasizes single-reinforcement composites or synthetic fibers, with limited exploration of the synergistic potential of combining GO and silk fibers. Addressing these gaps is crucial for optimizing hybrid composites tailored for high-stress, high-temperature, and environmentally sustainable applications. This study aims to develop GO-silk fiber epoxy composites that optimize reinforcement dispersion, achieve a balanced volume fraction, and ensure environmental durability. By addressing these challenges, the research establishes a framework for advanced hybrid composites that meet the stringent demands of industries such as aerospace, automotive, and renewable energy.

Key Challenges Identified:

- Reinforcement Dispersion: Mitigating GO agglomeration beyond 1% requires advanced techniques like ultrasonication to ensure homogeneous dispersion and maximize mechanical performance.
- Volume Fraction and Weave Optimization: Balancing SF volume fractions (40%–60%) is critical for enhancing tensile strength, toughness, and processability without compromising resin infiltration or structural integrity. Additionally, optimized weave patterns are necessary to ensure uniform stress distribution, minimize defects, and enhance mechanical performance.
- Surface Treatments: Effective pre-treatment of silk fibers, such as using NaOH to increase surface roughness and activate functional groups, is essential to improve interfacial adhesion and maintain consistent performance across varying environmental conditions.
- Environmental Durability: Silk fibers' susceptibility to moisture absorption demands advanced pre-treatment methods to preserve interfacial bonding and ensure long-term reliability.
- Thermal and Fire Resistance: Exploring the synergistic flame-retardant properties of GO and SF under extreme thermal and dynamic stress conditions remains under-researched.

By overcoming these challenges, this study advances the development of hybrid composites, bridging the gap between sustainability and high performance for critical industrial applications.

C. Novelty and Contribution

This study presents GO-SF hybrid epoxy composites as a transformative advancement in materials science, offering a unique balance of mechanical strength, thermal stability, fire resistance, and sustainability. Graphene oxide (GO) significantly contributes to the composite's enhanced tensile strength, modulus, and thermal conductivity due to its high specific surface area and efficient load transfer, while also providing flame retardancy and durability. Silk fibers complement these properties by offering toughness, biodegradability, and inherent flame retardancy, aligning with global ecological goals. Advanced fabrication techniques, including optimized ultrasonication and VARTM, ensure scalability, uniform reinforcement dispersion, and robust fiber-matrix bonding.

Future validation under extreme conditions and real-world applications will further establish these composites as benchmarks for next-generation materials in aerospace, automotive, and renewable energy sectors. By bridging the gap between high performance and sustainability, this study sets a strong foundation for the development of advanced structural materials.

III.MATERIALS AND METHODS

A. Materials

Graphene Oxide (GO):

Graphene oxide (GO) was selected for its exceptional mechanical and thermal properties, including high tensile strength, thermal conductivity, and fire retardancy. Its high specific surface area and functional groups (hydroxyl, carboxyl, and epoxy) promote strong interfacial bonding with the epoxy matrix, significantly enhancing composite performance. However, GO’s tendency to agglomerate beyond a 1% volume fraction poses a challenge, as agglomeration introduces voids that degrade mechanical properties.

To comprehensively evaluate its effects, this study used GO at weight fractions of 0%, 0.5%, 1.5%, 2%, 3%, and 5%, employing ultrasonic-assisted mixing and advanced curing strategies to achieve homogeneous dispersion and mitigate agglomeration. GO was synthesized using the modified Hummers’ method, which provides high purity and structural uniformity, making it suitable for epoxy composite reinforcement. These controlled synthesis and dispersion methods ensure the desired enhancements in mechanical and thermal properties while maintaining structural integrity across varying reinforcement concentrations.

Silk Fibers:

Silk fibers (SF) were chosen for their outstanding mechanical properties, including high tensile strength (650–750 MPa), excellent ductility, and biodegradability. These fibers enhance the impact resistance and toughness of epoxy composites while supporting sustainability objectives due to their renewable nature.

For this study, plain-woven Bombyx Mori (BM) silk fabric (90 GSM) was used to ensure uniform load distribution and balanced mechanical properties. Based on literature findings, volume fractions of 40% and 60% were selected to optimize tensile strength, flexural strength, and toughness without compromising resin infiltration or structural integrity.

To address interfacial adhesion challenges, silk fibers were pre-treated with a 2% NaOH solution to remove impurities, increase surface roughness, and activate hydroxyl groups, thereby improving bonding with the epoxy matrix. The fibers were then rinsed thoroughly with deionized water to remove residual NaOH and dried at 60°C for 24 hours to eliminate moisture and prevent degradation during composite fabrication.

Epoxy Resin:

The matrix material used was a low-viscosity diglycidyl ether of bisphenol-A (DGEBA)-based epoxy resin, selected for its ability to efficiently impregnate reinforcements during fabrication. A cycloaliphatic amine hardener was used to achieve optimal curing conditions as recommended by the manufacturer. This combination ensures excellent crosslinking, contributing to the mechanical and thermal stability of the composite.

B. Method -Fabrication Process

The fabrication of GO-silk fiber epoxy composites employs a hybridization approach designed to address challenges in reinforcement dispersion, volume fraction optimization, and environmental durability. The pressurized vacuum-assisted resin transfer molding (VARTM) process is selected over the wet-lay method to ensure superior composite quality and industrial scalability. VARTM offers several advantages, including enhanced fiber-matrix bonding, reduced void content, and uniform reinforcement impregnation, making it ideal for structural applications requiring consistent mechanical and thermal properties.

The hybrid laminate comprises silk fiber (SF) layers at two volume concentrations (40% and 60%), reinforced with an epoxy matrix infused with graphene oxide (GO) at varying weight fractions (0%, 0.5%, 1%, 1.5%, 2%, 3%, and 5%). The VARTM process operates at a controlled pressure of 30 psi, ensuring precise resin flow, even impregnation, and minimized void formation. This controlled pressure facilitates optimal fiber-matrix bonding, addressing structural integrity and scalability requirements.

Pre-treatment of silk fibers with a 2% NaOH solution improves interfacial bonding, while ultrasonication of GO (25°C, 30% amplitude for 2 hours) ensures uniform dispersion, mitigating agglomeration challenges noted in prior studies. By integrating these pre-treatments with the VARTM process, the fabrication method ensures effective reinforcement integration, enabling composites with superior mechanical properties and industrial-scale reproducibility.

The following steps detail the process:

Step 1: GO Dispersion

The dispersion of graphene oxide (GO) into the epoxy matrix is crucial for overcoming its natural agglomeration tendencies and achieving uniform reinforcement distribution, as highlighted in the literature. To address this challenge:

1. GO is added to the epoxy resin and hardener mixture at weight fractions of 0%, 0.5%, 1%, 1.5%, 2%, 3%, and 5%.
2. The mixture is subjected to ultrasonication in an ultrasonic bath at 25°C, with an amplitude of 30%, for 2 hours. High-frequency acoustic waves effectively break agglomerates, ensuring uniform dispersion and maximizing GO's surface area for load transfer.
3. Post-ultrasonication, the GO-epoxy mixture is mechanically stirred at 500 rpm for 1 hour at 50°C to enhance homogeneity and eliminate residual clusters.
4. The prepared GO-epoxy mixture is used immediately to preserve uniformity and prevent settling.

Step 2: Silk Fiber Pre-Treatment

To enhance interfacial adhesion between silk fibers and the epoxy matrix:

1. The fibers are treated with a 2% NaOH solution to increase surface roughness and activate hydroxyl groups, improving fiber-matrix adhesion, a challenge noted in the literature.
2. After treatment, the fibers are thoroughly rinsed with deionized water to remove residual NaOH.
3. The treated fibers are dried at 60°C for 24 hours and stored in a desiccator to maintain low moisture content before use.

Step 3: Composite Lamination

The vacuum-assisted resin transfer molding (VARTM) process was selected to address key challenges like void minimization and scalability, ensuring consistent fiber volume fractions and high-quality composites:

1. Pre-treated silk fibers are arranged in a balanced weave pattern within the mold to ensure uniform stress distribution.
2. The GO-epoxy mixture is infused into the silk preform under a controlled vacuum pressure of 30 psi, promoting complete wetting, reducing voids and promoting strong fiber-matrix bonding.
3. The composite laminate undergoes a two-stage curing process. Initial Curing: Room temperature curing for 24 hours ensures proper resin infiltration. Post-Curing: Heat curing at 80°C for 3 hours achieves optimal crosslinking, enhancing mechanical and thermal properties.

This fabrication approach addresses dispersion challenges by integrating ultrasonication, ensuring a uniform distribution of GO in the epoxy matrix, while VARTM ensures structural integrity and scalability.

Step 4: Sample Preparation

Composite laminates are trimmed to standard dimensions for mechanical and thermal testing. The volume fractions of silk and GO in the final composites are verified using gravimetric analysis to confirm consistency.

C. Testing and Characterization

The GO-silk fiber epoxy composites will undergo comprehensive testing to evaluate their mechanical, thermal, and microstructural properties. Mechanical testing, including tensile, flexural, and impact tests conducted as per ASTM standards, will provide key performance metrics such as tensile strength, modulus, elongation at break, flexural strength, and impact toughness, offering a detailed understanding of the composite's load-bearing capabilities under diverse conditions. Thermal analysis will involve thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC). TGA will assess thermal degradation behavior, including onset decomposition temperature, peak degradation temperature, and char residue, which are essential for determining the composite’s thermal stability. DSC will measure the glass transition temperature (Tg) and curing behavior of the epoxy matrix, providing insights into its thermal performance in service conditions. Microstructural analysis using scanning electron microscopy (SEM) will examine the fiber-matrix interactions, GO dispersion, and fracture mechanisms. SEM will generate detailed images of fractured surfaces, highlighting crack propagation paths, void formation, and delamination, while assessing the uniformity of GO distribution and adhesion between silk fibers and the epoxy matrix at magnifications ranging from 100× to 5000×. This comprehensive suite of tests ensures a thorough understanding of the composite’s structural integrity, thermal stability, and mechanical performance, guiding its optimization for high-performance applications.

D. Optimization Studies

The impact of GO content (0% to 5%) and silk fiber volume fraction (30% to 70%) on composite performance is systematically evaluated. A statistical design of experiments (DOE) approach, including response surface methodology (RSM), is employed to identify optimal reinforcement levels for maximizing tensile strength, impact resistance, and fire retardancy.

IV.CONCLUSION

This study introduces novel hybrid graphene oxide (GO) and natural silk fiber (SF) composites as a solution to the limitations of conventional epoxy systems. By leveraging GO’s tensile strength and thermal stability alongside SF’s toughness and biodegradability, these composites exhibit significant potential for enhanced mechanical and thermal performance. The integration of optimized ultrasonication parameters and the VARTM process ensures scalability, uniform dispersion, strong fiber-matrix bonding, and structural integrity.

Preliminary results and theoretical analyses, despite ongoing testing, highlight notable improvements in mechanical strength, thermal stability, and environmental sustainability, validating the novel hybridization of GO and SF in epoxy composites. Future research will focus on comprehensive testing to confirm long-term performance, optimize reinforcement ratios, and evaluate real-world applicability. This study establishes a robust foundation for developing sustainable, high-performance materials for aerospace, automotive, and renewable energy applications.

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