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Epoxy

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*Edited by Petrică Vizureanu
and Mădălina Simona Bălțatu*



Epoxy - Materials, Applications and Advanced Technologies

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Materials Science

Volume 16

Aims and Scope of the Series

Materials science has always occupied an extremely high position in the human development process. As we explore the oceans of stars, various industries have put forward more stringent requirements for the performance of materials, forcing us to pay more and more attention to the development of new materials. At the same time, the formation of a data-driven scientific paradigm is dramatically shortening the development cycle of new materials. The huge data generated by synergistically combining theories, high-throughput experiments, high-throughput computation, and artificial intelligence is greatly contributing to our ability to utilize materials science to solve real-world problems. The three topics of this book series - Metals and Nonmetals; Composite Materials; and Surface Science - will address important areas of advancement in materials science. There will be a range of interesting works published under these topics.

Meet the Series Editor



Prof. Chonghe Li received his Ph.D. from the Chinese Academy of Sciences in 1995. From 1995 to 2000, he worked as a researcher at the Shanghai Institute of Metallurgy, Chinese Academy of Sciences, where he also served as director of the research laboratory. In 2000, he was appointed Professor at the Institute of High-Performance Computing in Singapore, where he worked on computation and simulation in materials science until 2004. Since then, he has been a professor at the School of Materials Science and Engineering, Shanghai University, China, as well as the director of the Shanghai Specialty Casting Engineering and Technology Research Center. Prof. Li's research focuses on titanium alloys, titanium-aluminum single crystals, intermetallic compounds, theoretical calculations, alloy design, and special refractory materials. His broad scientific expertise is well recognized by the scientific community around the world. He is a member of the editorial board of the journal *Metals*. As an author, he has published more than 200 peer-reviewed papers, 2 books, and over 40 patents.

Meet the Volume Editors



Petrică Vizureanu obtained an MSc and Ph.D. in Heating Equipment at the “Gheorghe Asachi” Technical University, Iasi, Romania, in 1992 and 1999, respectively. Dr. Vizureanu is currently a full professor and scientific supervisor in Materials Engineering at the same university and the appointed director of the Department of Technologies and Equipment for Materials Processing. He is an editor and guest editor for many journals and publishing houses. His research focuses on expert systems for heating system programming, computer-assisted design for heating equipment, heating equipment for materials processing, heat transfer, biomaterials, and geopolymers. He has published more than 250 papers in international journals/conference proceedings and over 40 books.



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Preface

Epoxy resins are among the most important classes of thermosetting polymers, widely used across industries due to their outstanding mechanical strength, chemical resistance, excellent adhesion, and tunable properties. Over the decades, epoxy systems have evolved from traditional petroleum-based resins to highly engineered multifunctional materials that meet the demands of advanced technologies and sustainability-driven innovations. This book, *Epoxy – Materials, Applications and Advanced Technologies*, provides a comprehensive overview of recent progress in the development, modification, and application of epoxy-based materials — from classical uses to cutting-edge functional systems.

This book brings together six comprehensive chapters that explore the evolution, structure, and advanced applications of epoxy-based materials. Beginning with the historical development and prospects of epoxy systems, the volume traces the transition from conventional formulations to modern, high-performance, environmentally friendly alternatives. It highlights the industrial significance of epoxies as adhesives, coatings, and composite matrices, while also emphasizing the current shift toward sustainability through bio-derived polymers, such as epoxidized lignin. Further chapters discuss the design and preparation of advanced epoxy systems with low dielectric constants and high thermal conductivity, essential for electronic and thermal management applications. The role of epoxy nanodielectrics in high-voltage insulation systems is also analyzed, showing how nanoscale engineering enhances dielectric strength and long-term stability. Finally, the synergistic effects of flame-retardant additives and natural bamboo fiber reinforcement are presented, demonstrating innovative pathways toward multifunctional and eco-efficient epoxy composites.

Overall, this edited volume provides a unified overview of the state-of-the-art developments in epoxy science and technology. It aims to serve as a valuable reference for researchers, engineers, and graduate students in materials science, polymer chemistry, and related engineering fields, offering insights into both established applications and future directions of epoxy materials in sustainable, high-performance systems.

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Evolution and Prospects of Epoxy Materials: From Traditional to Advanced

Petrică Vizureanu, Andrei Pruteanu, Radu Claudiu Fierascu, Madalina Simona Baltatu, Paweł Pietrusiewicz, Andrei Victor Sandu and Marcin Nabiałek

Abstract

Epoxy resins are widely used in industry due to their outstanding mechanical, thermal, and chemical properties. Advances in epoxy-based technologies have enabled the development of nanocomposites, self-healing systems, and bio-based formulations, creating opportunities for high-performance and sustainable applications. This review discusses the structure and key properties of epoxy resins, with emphasis on recent innovations. The addition of nanomaterials such as graphene and carbon nanotubes enhances strength, conductivity, and thermal stability. At the same time, self-healing strategies improve durability, reduce maintenance, and extend service life. The shift toward bio-based epoxies obtained from renewable sources is essential to lowering environmental impact, and current research is directed at achieving performance comparable to conventional solutions. In industry, advanced epoxies are critical in aerospace, automotive, electronics, and construction. Their use in composites for aircraft, electric vehicles, and compact electronics demonstrates both versatility and potential in modern technologies. Recent progress in processing, including 3D printing and optimized curing, contributes to higher efficiency and customization. In addition, artificial intelligence and computational tools are increasingly applied to design formulations adapted to specific industrial demands. In conclusion, epoxy materials continue to evolve through innovation and sustainability. Future efforts aim to optimize performance, reduce costs, and integrate eco-friendly epoxies into major industrial sectors, ensuring ongoing progress in advanced materials.

Keywords: epoxy materials, epoxy's utility, properties of epoxy materials, advantages and disadvantages, classification of epoxy materials

1. Introduction

Epoxy materials have significantly transformed a wide range of industries due to their outstanding properties, such as mechanical strength, chemical resistance,

and general uses. Initially developed to function as coatings and adhesives, these polymers have evolved enough to enable their usage in a wide range of industries, including construction, automotive, aerospace, and electronics. This chapter explores the origins, key properties, and future potential of epoxy materials, discussing their evolution and development from traditional applications to innovative uses [1, 2].

Originally developed in the mid-twentieth century, epoxy materials debuted as strong and versatile materials, but have since undergone remarkable advancements. The advances in chemical and processing techniques have led to the creation of improved materials, whose evolution is marked by the growing role in structural design, electronics, and manufacturing practices.

The main category to which these materials belong is Resins, which include thermosets and thermoplastics. Thermoplastic resin softens when heated and can be reshaped, while thermosets cannot be remolded. The most common being polyester, epoxy, and vinyl ester resins. Less commonly used resins in composites include phenolics, ester cyanates, silicones, polyurethanes, and polyamides.

The origin of epoxy resins dates back to the early twentieth century when Dr. Pierre Castan in Switzerland and Dr. Sylvan Greenlee in the United States independently developed early formulations. Initially, epoxy resins were recognized for their excellent adhesive properties and were quickly adopted in industrial applications such as protective coatings and structural bonding.

During the mid-twentieth century, epoxy materials underwent significant advancements with the introduction of amine-based curing agents and reinforced composite systems. These innovations expanded their applications to the automotive, aerospace, and electrical industries. With improvements in formulation chemistry, epoxy resins began demonstrating superior thermal and chemical stability, making them indispensable in structural applications [3].

Although significant progress has been made in developing bio-based epoxy resins with enhanced thermal and mechanical properties, large-scale production remains limited due to several challenges—such as the high cost of biomass-derived raw materials and the complexity of processing bio-based epoxy monomers. To overcome these limitations and enhance their competitiveness, bio-based epoxy resins must not only achieve high-performance standards but also be designed with specific functional properties in mind. These include flame retardancy, degradability, recyclability, and reprocessability, shape memory behavior, and antibacterial activity, among others.

This emerging class of multifunctional bio-based epoxy resins has attracted growing attention in recent years, leading to an increasing number of studies. While numerous reviews have explored the synthesis and high-performance characteristics of bio-based epoxy resins derived from biomass, most of them do not focus on the broader spectrum of functionalities. Some reviews address only specific features, such as flame retardancy, but a comprehensive overview of functional bio-based epoxy resins is still lacking.

This chapter aims to fill that gap by summarizing recent developments in the field of bio-based epoxy resins with a variety of functional properties derived from renewable resources. The final section discusses the current challenges and future opportunities in the design and application of functional bio-based epoxy resins.

2. Classification of epoxy materials

Epoxy materials can be categorized based on their chemical composition, curing mechanisms, and application fields [4].

The detailed classifications, along with examples and their respective applications, are presented in **Table 1**.

This classification framework helps in understanding the diverse nature of epoxy materials and their suitability for different engineering and industrial applications.

Epoxy materials can be categorized based on their chemical composition, curing mechanisms, and application fields.

Classification Type	Category	Description	Examples	Applications
Based on chemical composition	Glycidyl epoxies	Includes epoxies with glycidyl ether or glycidyl ester groups and is widely used in coatings, adhesives, and composites.	DGEBA (diglycidyl ether of bisphenol A), epoxy novolac, cycloaliphatic epoxy	Aerospace composites, industrial coatings, structural adhesives
	Non-glycidyl epoxies	Comprises aliphatic and aromatic epoxy resins, offering specialized properties for high-performance applications.	Aliphatic epoxy resins, aromatic epoxy resins	UV-resistant coatings, electronic encapsulations, and biomedical applications
Based on curing mechanism	Thermosetting epoxies	Requires curing agents (e.g., amines and anhydrides) to create a rigid, cross-linked network commonly used in structural applications.	Bisphenol A epoxy, anhydride-cured epoxy	Structural composites, high-temperature coatings, and insulation
	UV-curable epoxies	Cures upon exposure to ultraviolet light, making it ideal for coatings, adhesives, and rapid prototyping.	Acrylated epoxy resins	Optical coatings, 3D printing, and electronics packaging
	Self-healing epoxies	Engineered with microcapsules or reversible chemical bonds that enable autonomous repair of cracks and damage.	Microcapsule-based epoxy, diels-alder epoxy	Aerospace materials, automotive coatings, smart composites
Based on application fields	Structural epoxies	High-strength epoxies used in aerospace, automotive, and construction industries.	Carbon fiber epoxy, glass fiber reinforced epoxy	Aircraft structures, automotive body panels, load-bearing composites

Classification Type	Category	Description	Examples	Applications
	Coatings and adhesives	Provides corrosion resistance, durability, and superior adhesion for industrial and protective applications.	Epoxy powder coating, solvent-based epoxy coating	Protective coatings, marine applications, and industrial adhesives
	Biomedical Epoxies	Biocompatible formulations designed for use in medical devices, implants, and tissue engineering.	PEG-based Epoxy, Epoxy Hydrogels	Dental composites, prosthetic coatings, and drug delivery systems
	Electronic and electrical epoxies	Utilized in encapsulation, insulation, and printed circuit board manufacturing for enhanced electrical performance.	Epoxy potting compounds, thermally conductive epoxy	Semiconductor packaging, circuit board protection, and electrical insulation

Table 1.
Classification.

3. Fundamental properties of epoxy materials

Epoxy resins have an important place in a wide array of industrial applications, due to their excellent properties, such as high strength, thermal and chemical resistances (**Figure 1**) [5]. The **Table 2** provides a detailed overview of the key properties of epoxy materials and their significance.

4. Manufacturing and processing of epoxy materials

Epoxy resins are primarily formed from epoxide-containing oligomers and curing agents. The base monomer most commonly used is epichlorohydrin, often reacted with bisphenol A to produce diglycidyl ether of bisphenol A—the dominant commercial epoxy resin, accounting for over 70% of usage [6].

The processing techniques and synthesis of different epoxy resins are presented below (**Figure 2**).

Epoxy resins are synthesized through the reaction of epichlorohydrin with bisphenol A (BPA) or other polyols [7].

The main methods of synthesis of epoxy resins include several distinct types. Glycidyl epoxy resins are obtained by the glycidylation reaction, resulting in diglycidyl ether of bisphenol A (DGEBA), one of the most widely used epoxy resins. Cycloaliphatic epoxy resins, on the other hand, are synthesized without glycidyl ether groups, offering superior resistance to UV radiation and excellent electrical insulation properties. Another important category is represented by epoxy-novolacs, which are formed by the reaction of phenolic novolac resins with epichlorohydrin, resulting in products with high crosslink density and high chemical resistance.

The curing process of epoxy resins involves crosslinking the polymer network by adding hardening agents (hardeners). Amine curing involves the use of polyamines

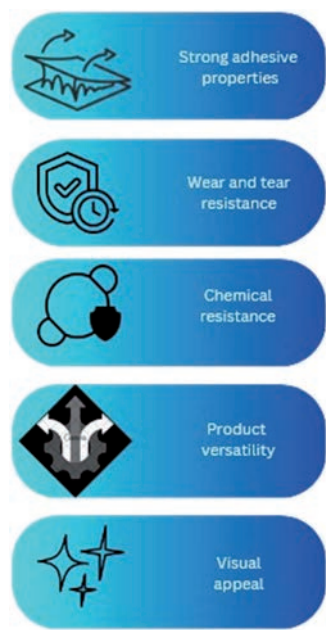


Figure 1.
The properties of epoxy materials.

Property	Description	Importance
High mechanical strength	Epoxies exhibit high tensile and flexural strength, making them suitable for load-bearing applications.	Essential for aerospace, automotive, and structural applications.
Chemical resistance	Resistant to acids, bases, and solvents, preventing material degradation.	Ideal for coatings, adhesives, and chemical processing industries.
Thermal stability	Can withstand temperatures up to 250°C, with advanced formulations exceeding 300°C.	Critical for high-temperature applications such as electronics and aerospace.
Adhesion and cohesion	Strong bonding ability to various substrates, including metals, plastics, and ceramics.	Used extensively in adhesives, composite materials, and protective coatings.
Low shrinkage	Minimal volume reduction during curing, ensuring dimensional stability.	Important for precision applications, including electronics and dental materials.
Dielectric properties	Excellent electrical insulation properties, with dielectric strengths exceeding 10 kV/mm.	Applied in electronics, encapsulation, and circuit board manufacturing.
Water and moisture resistance	Provides barrier properties against moisture ingress, preventing corrosion.	Suitable for marine coatings, construction, and protective encapsulation.
Tailorability	Properties can be modified using fillers, additives, and curing agents to meet specific needs.	Expands versatility in diverse industrial applications, including biomedical and sustainable technologies.
Fire resistance	Flame-retardant epoxy formulations with high limiting oxygen index (LOI) values.	Ensures safety in applications such as aircraft interiors and electrical insulation.

Table 2.
The properties of epoxy materials.

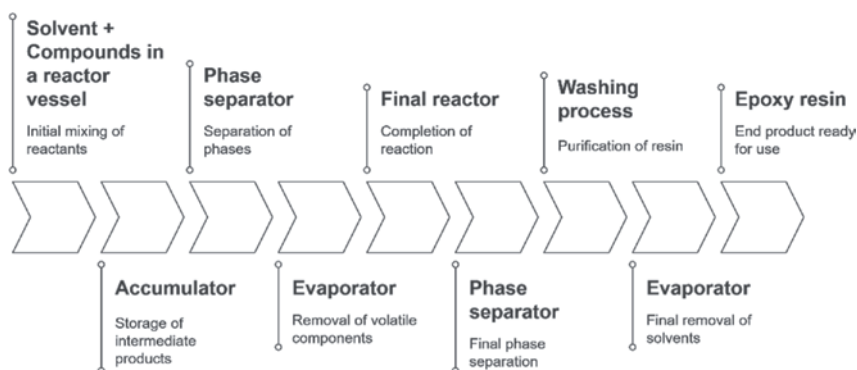


Figure 2.
Epoxy materials processing flow.

to obtain thermosetting polymers with a high degree of crosslinking and excellent mechanical properties. Anhydride curing is slower, but gives the resins increased thermal stability and advanced electrical insulation properties. Modern methods also include curing by UV radiation or other forms of radiation, which is frequently used in electronic and optical applications to quickly cure adhesives and coatings. Latent curing systems, on the other hand, use hardeners that become active only under specific temperature or humidity conditions, thus providing an extended working time before the polymerization reaction begins (**Figure 3**).

The choice of curing agent significantly influences the final properties of the epoxy material. Commonly used curing agents include aliphatic and aromatic amines, acid anhydrides, polyamides and imidazoles, each of which has a different effect on the behavior of the resin after curing. For example, aliphatic amines allow rapid curing at room temperature and provide good chemical resistance, while aromatic amines provide high thermal stability and are used in applications where resistance to high temperatures is required, such as in electronics or aerospace. Acid anhydrides are

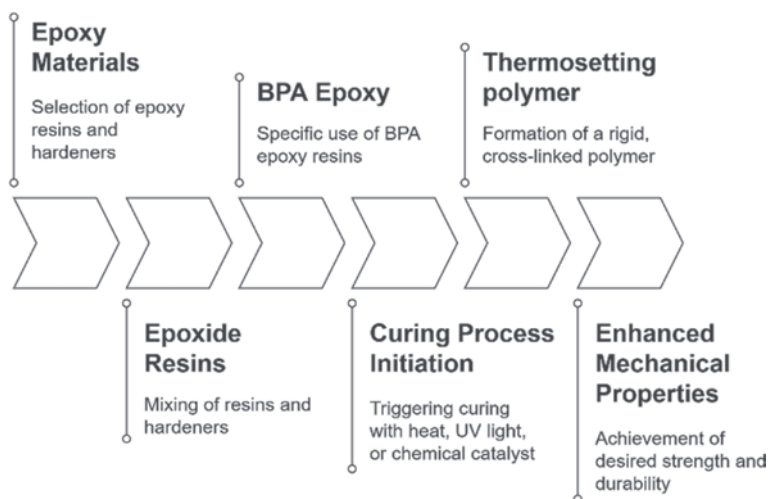


Figure 3.
Epoxy material composition and curing process.

preferred in electrical insulation due to their dielectric properties and good thermal stability. Polyamides provide flexibility and toughness, being suitable for adhesives or coatings subject to mechanical stress, and imidazoles are valued for their latent curing ability, which is useful in temperature-sensitive applications.

The type of hardener chosen thus influences the field of use of the epoxy resin, an aspect detailed in the next subchapter. Understanding the relationship between the base resin and the hardener is important for adapting the material to the specific requirements of the application.

The industrial-scale production of epoxy resins requires the application of strict quality control measures. Verification of the purity of raw materials, such as epichlorohydrin and bisphenol A, is essential to obtain consistent resin properties. Optimization of the curing process is achieved by controlling the temperature and monitoring the catalysts to ensure complete polymerization. Finally, the finished products undergo rigorous performance testing, which includes evaluation of tensile strength, impact resistance, and thermal stability, to meet industrial requirements.

The environmental impact and sustainability of epoxy materials are a major concern in the current context of sustainable development. Although epoxy resins are valued for their versatility, they present significant challenges in terms of recycling due to their thermosetting nature, which makes reprocessing after curing difficult.

Methods under investigation for recycling these materials include chemical depolymerization, which involves breaking down cured resins into reusable components, and mechanical recycling, in which epoxy waste is ground and reused as fillers for composite materials. Hybrid approaches, which combine chemical and mechanical methods to increase the efficiency of the recycling process, are also being investigated.

To reduce the environmental impact, researchers are developing epoxy resins based on renewable raw materials. One example is resins derived from vegetable oils, such as linseed, soybean or castor oil. Other options include lignin-based epoxies, which utilize biomass waste to produce partially bio-based resins, and furan-based epoxies, which use furan derivatives to improve biodegradability without compromising mechanical strength.

5. Applications of epoxy materials

Epoxy materials have a broad spectrum of applications across various industries due to their exceptional properties, such as high strength, chemical resistance, and electrical insulation [8, 9].

The applications of epoxy resins include potting and encapsulation, tooling, molding, adhesive, and bonding (**Figure 4**).

Epoxy resins are widely used for potting and encapsulation due to their strong resistance to alkalis, acids, and humidity. They also offer high temperature resistance, low shrinkage, excellent electrical insulation, and are environmentally safe. In electronics, the adhesive properties help with the encapsulation of modules, transformers, and motors [10].

Tooling has become increasingly popular in manufacturing because it offers advantages such as low cost, rapid production, and design flexibility. A significant advancement in this area is the development of hybrid molds, which integrate epoxy with metal fillers to enhance performance while also lowering costs and production time, although they do have some limitations. Epoxy molds are particularly favored

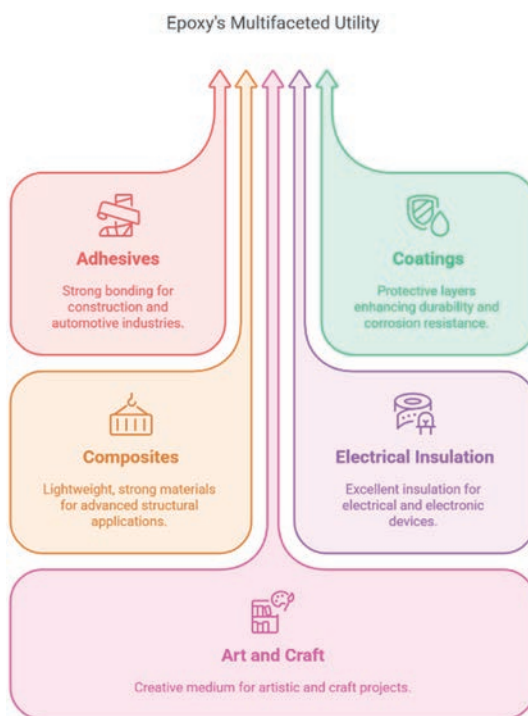


Figure 4.
The applications of epoxy materials.

for their dimensional accuracy and are often preferred over silicone or traditional tools when it comes to the quick manufacturing of low-volume batches [11].

Epoxy resins offer the highest adhesive strength among polymers, bonding a wide array of materials with low shrinkage. It is highly versatile in many applications. This property could be further improved by incorporating nanomaterials such as nano alumina, nano SiO₂, and graphene nanoplates [12].

Table 3 outlines the key applications of epoxy materials in different fields:

The versatility of epoxy materials allows for continuous innovation and expansion into new industries, making them indispensable in modern manufacturing and technology [13].

Epoxy materials offer several advantages that make them highly desirable for industrial, commercial, and scientific applications. Their exceptional properties contribute to their widespread use in various fields.

These advantages collectively make epoxy materials an important component in modern engineering and manufacturing.

Life cycle assessment and environmental regulations play a key role in guiding the sustainable production and use of epoxy materials. The current legislative framework focuses on reducing emissions of volatile organic compounds (VOCs) and promoting sustainability. In the European Union, the REACH regulation restricts the use of hazardous chemicals in the production process of epoxy resins, thereby ensuring the protection of human health and the environment. The international standard ISO 14040, dedicated to life cycle analysis (LCA), assesses the environmental impact of epoxy products from the extraction of raw materials to their final disposal. In

Industry	Application	Benefits
Aerospace	Structural adhesives, composites, coatings	High strength-to-weight ratio, durability, and thermal stability
Automotive	Structural parts, adhesives, coatings	Lightweight, impact resistance, and fuel efficiency enhancement
Construction	Flooring, adhesives, sealants	Chemical resistance, durability, and waterproofing
Electronics	Encapsulation, circuit boards, insulation	High dielectric strength, moisture resistance, and thermal stability
Biomedical	Dental composites, prosthetics, medical coatings	Biocompatibility, chemical resistance, and mechanical strength
Marine	Protective coatings, adhesives, composites	Corrosion resistance, water resistance, and high durability
Energy	Wind turbine blades, insulation in power systems	Structural integrity, electrical insulation, and resistance to environmental factors
Consumer Goods	Sports equipment, household adhesives, decorative coatings	Impact resistance, aesthetic finish, and long lifespan

Table 3.
Applications of epoxy resin materials.

addition, standards such as EPA Green Chemistry encourage the development of non-toxic epoxy formulations derived from renewable biological sources [14–16].

Despite their advantages, epoxy materials also have limitations that can affect performance in demanding applications. One of the major challenges is brittleness, as epoxies can be brittle and susceptible to cracking under impact conditions [17–19]. To improve toughness, various strategies are used, such as adding elastomeric modifiers (e.g., rubbers) to increase flexibility, manufacturing hybrid composites with carbon or glass fibers to increase shock resistance, and strengthening with nanoparticles (silica or graphene) to prevent crack propagation [12].

Long-term durability is another concern, especially in aggressive environments. Prolonged exposure to UV radiation can lead to photochemical degradation, manifested by yellowing and loss of mechanical properties, a problem that can be reduced by the use of UV stabilizers. In humid environments, hydrolytic degradation becomes relevant, as resins can absorb water, which leads to decreased adhesion—this phenomenon can be counteracted by the application of hydrophobic coatings [20–21]. Also, chemical attacks from contact with acids, solvents or bases can damage the epoxy structure, and this risk can be reduced by chemically resistant formulations [15].

6. Comparison with other polymer systems

Epoxy resins are often compared to polyurethane, polyester, and phenolic resins based on their properties (Table 4).

As shown Table 4, each resin type presents a unique balance of properties, and the choice between them depends largely on the specific performance requirements and application context [22, 23].

Property	Epoxy	Polyurethane	Polyester	Phenolic
Mechanical strength	High	Moderate	Moderate	High
Chemical resistance	Excellent	Moderate	Good	Excellent
Thermal stability	High	Moderate	Moderate	Very high
Flexibility	Low (brittle)	High	Moderate	Low
Curing speed	Moderate	Fast	Fast	Slow
Cost	Moderate to high	Low to moderate	Low	Moderate
Environmental impact	High (unless bio-based)	Moderate	High	Moderate

Table 4.
Advantages and disadvantages compared to other resins.

7. Conclusion

Epoxy materials have come a long way from their traditional applications to cutting-edge technological advancements. Their evolution has been driven by the need for stronger, more durable, and sustainable materials, leading to remarkable innovations in composite formulations and functional properties.

In recent years, research in the field of epoxy systems has focused not only on improving mechanical and thermal performance but also on addressing environmental concerns and expanding their functional versatility. The incorporation of nanomaterials such as carbon nanotubes, graphene, and silica nanoparticles has significantly enhanced the mechanical strength, electrical conductivity, and thermal stability of epoxy resins. These advanced formulations are now widely applied in aerospace components, electronic devices, and high-performance coatings.

At the same time, the development of bio-based epoxy resins from renewable raw materials like plant oils, lignin, and other biopolymers has opened new possibilities for reducing the environmental footprint of these materials without compromising their performance. Efforts in sustainability are further complemented by the emergence of self-healing epoxy systems, capable of autonomously repairing microcracks and extending service life, as well as smart epoxies with shape memory and stimuli-responsive behaviors, which show promise in biomedical and aerospace fields.

Innovative formulations have also improved the fire resistance and high-temperature performance of epoxy resins, making them suitable for critical applications such as aircraft interiors, electronic encapsulation, and protective coatings in harsh environments.

Looking ahead, the future of epoxy materials lies in enhancing their sustainability, functionality, and adaptability to meet the demands of emerging technologies. Key research directions include:

- Development of fully recyclable epoxies, aiming to overcome challenges related to end-of-life disposal.
- Integration with additive manufacturing and 3D printing enables the design of customized, complex structures with high performance.
- Enhanced conductive epoxies supporting the expansion of flexible electronics, sensors, and energy storage systems.

- Multifunctional epoxy systems, capable of combining mechanical, thermal, and electronic properties for next-generation smart materials.

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Applications of Epoxy Resin: Adhesives, Coatings, and Composites

Makinde-Isola Baraka Abiodun, Oladele Isiaka Oluwole, Akinwekomi Akeem Damilola, Bichang'a Denis Ondieki and Makinde Akindeji Ibrahim

Abstract

Epoxy resins are a class of thermoset polymeric materials used in various applications. They possess a range of good properties such as high toughness, rigidity, wear resistance, and durability. Besides, its strong adhesion ability and chemical resistance make them useful for several applications. Epoxies are utilized in the coatings and adhesive industries to protect and bond solutions to surfaces exposed to harsh environments. It provides corrosion resistance for metal structures and wear resistance for materials such as polymers, ceramics, and wood. They are also used in making lightweight and high-strength composite materials for use in the aerospace, automotive, and marine industries where they are used to manufacture parts such as the wings of an aircraft, fuselage, car body panels, boat hulls, and cargo tanks. Therefore, epoxy is a vital component of contemporary industrial and construction applications.

Keywords: epoxy resin, polymer, adhesion, protective coating, composites

1. Introduction

One of the most versatile thermosetting polymers is epoxy resin. Thermosetting polymers are usually made up of monomers and oligomers typically combined with a liquid hardener, such as another resin or catalyst. When the resin and hardener react together, a crosslinked structure is created by joining the monomers and oligomers into lengthy polymer chains [1]. The crosslinked structure resists sliding and rotation when a load is applied leading to a higher stiffness, strength, and hardness than thermoplastics. Epoxy resin possesses two or more epoxy groups referred to as epoxide or oxirane, in the molecule. It is widely used in several industries due to the properties they possess both during processing and after curing. It is capable of showing low viscosity during processing and requires a little amount of pressure to form products. They can also combine with several types of hardeners at low temperatures and elevated temperatures. After curing, they exhibit low cure shrinkage and residual stresses and gives a good chemical and heat resistance [2–4].

The properties of epoxy resin can range from hard and strong to tough and resilient. Depending on their constituents and their areas of applications, they can be modified to reach glass transition temperatures between 60°C and 250°C by using various prepolymerers. Epoxy resins exist in two groups, the aromatic epoxy saturated ring and the nonaromatic saturated ring epoxy. The aromatic epoxy saturated ring comes from the monomer diglycidylether of bisphenol A (DGEBA) and is widely used for outdoor application due to its resilience to UV radiation while 3,4-epoxycyclohexyl-3,4-epoxycyclohexanecarboxylate is the source for the nonaromatic epoxy [1, 5]. Diglycidylether of bisphenol A, or DGEBA, is the most widely used epoxy resin, making up more than 75% of all epoxy usage [6, 7]. **Figure 1** depicts DGEBA's chemical structure. Other common epoxy resin is novolac epoxy resin, bisphenol F epoxy resin, aliphatic epoxy resin, glycidylamine epoxy resin, and bio-based epoxy resin [8]. Some of characteristics of these epoxy resins and their applications are shown in **Table 1**.

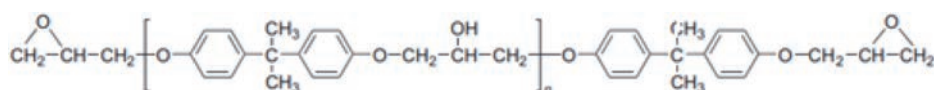


Figure 1.
Structure of diglycidylether of bisphenol A (DGEBA) [7].

Epoxy resin type	Chemical Structure	Properties	Applications
Bisphenol A diglycidyl ether		They are highly reactive and exhibit excellent adhesion, mechanical and thermal strength	Construction, structural adhesives, transportation, and furniture production
Novolac epoxy resin		They have high glass transition temperature (T _g) and high resistance to corrosion and thermal degradation	Electrical and electronic applications, high-temperature coating systems, electronic circuit boards, and enclosures
Bisphenol F epoxy resin		They show good physical properties and a good solution and melt viscosities	Coatings, electronics, and composite materials
Aliphatic epoxy resin		They have low viscosity and electrical loss properties. They also show less yellowing	Flooring systems, adhesives with UV resistance, and elastomers
Glycidylamine epoxy resin		They show good toughness, mechanical strength, and a high thermal stability	Aerospace composites, electronic packaging, and advanced adhesives

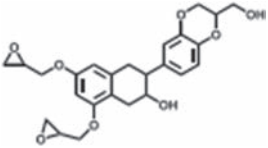
Epoxy resin type	Chemical Structure	Properties	Applications
Bio-based epoxy resin		They are tough and biocompatible. They also show low-temperature curing	Biodegradable adhesives, biomedical materials and equipments, and surface coatings and paints

Table 1.

Comparison of epoxy resin type, their properties, and applications [8–12].

Epoxy resins find applications in numerous industries as shown in **Figure 2**, for different use. They can be used as structural adhesives, surface coatings, engineering composites, and electrical insulation [1]. The percentage of their applications in these fields is shown in **Figure 3**. They are being highly used in electrical appliances, automobile parts [13], marine industries, aerospace industries, biomedical industries [14], jewelry, leisure, sports, and esthetics. Epoxy resin has been used to replace several components due to advancement in materials technology. They are being used

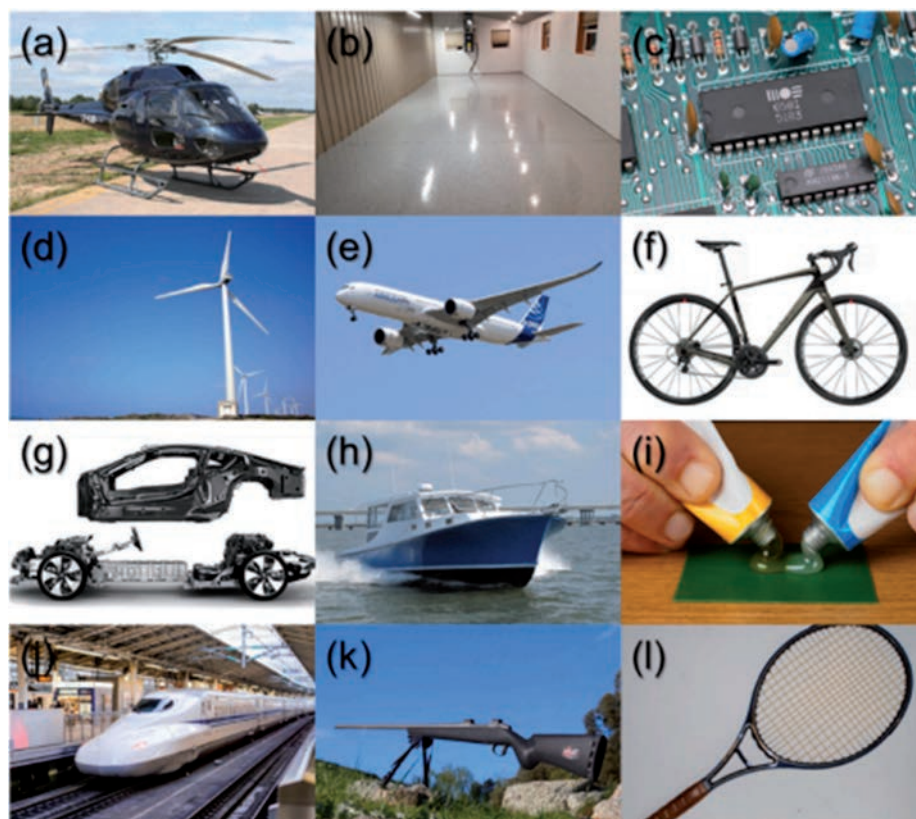


Figure 2.

Applications of epoxy resin (a) Helicopters; (b) floor finish; (c) electronic industry and applications; (d) wind turbines; (e) aircraft structure; (f) bicycle parts; (g) automotive structure; (h) ships and marine vehicles; (i) epoxy adhesives; (j) rail and railroad repair; (k) firearm coating; (l) sports, i.e., tennis racket [17].

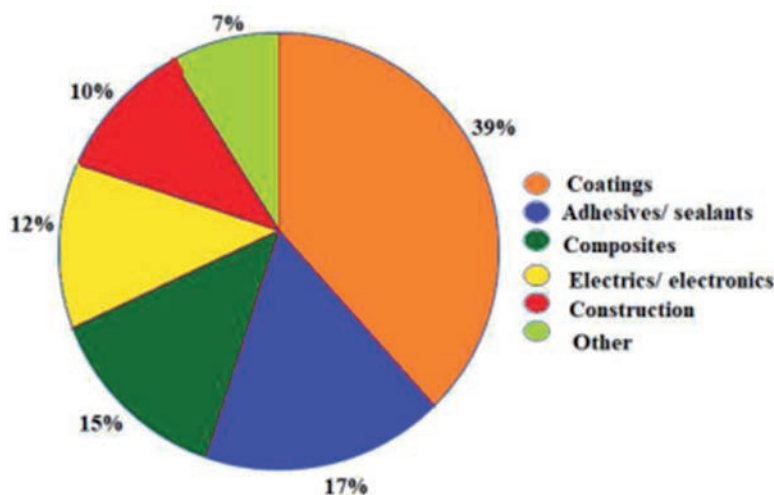


Figure 3.
Percentage of applications of epoxy resins in various fields [8].

to replace parts made from woods, metals, and other materials to save production costs, increase efficiency, and reduce weight in various industries particularly the automobile and aerospace industries [15]. They have also been combined with both natural and synthetic fibers to make parts such as car bumpers, dashboards, under-floor protection, fuselage, wind blades, boats, and electronic components [15, 16]. The need of epoxy resin is continuously growing everyday which this has drastically soared their global market [8, 9].

Due to rapid advancements, continuous development of technology, and need for sustainable materials in different industries, the use of epoxy resins is constantly evolving [15], and new types of epoxy resin with improved properties are being developed. Epoxy resins are being incorporated with nanofillers and bio-based epoxy resins from natural sources are being developed to enhance environmental sustainability [17]. Also, they are being used in emerging applications like flexible electronics, self-healing concrete, and biomedical devices. Therefore, an updated review on epoxy resin applications is necessary to provide an overview of the latest advancements, address the challenges and opportunities in different industries, and guide future research and development efforts.

2. Epoxy resin in adhesives

An adhesive is a high-strength glue that bonds different component materials together. They can be used on a wide range of substrates including for bonding metals to metals, metals to composites, metals to ceramics, metals to polymers, and even composites to composites [18]. They can also be used for bonding parts made from wood and cement and come in different forms such as in film form or paste form. **Figure 4** shows some of their applications. Epoxy adhesives also known as epoxy glue are one of the leading adhesive solutions and key use of epoxy resins in sectors including the aerospace, automotive, construction, manufacturing, biomedical, and electronics sectors [4]. Epoxy adhesives are used because of the unique properties

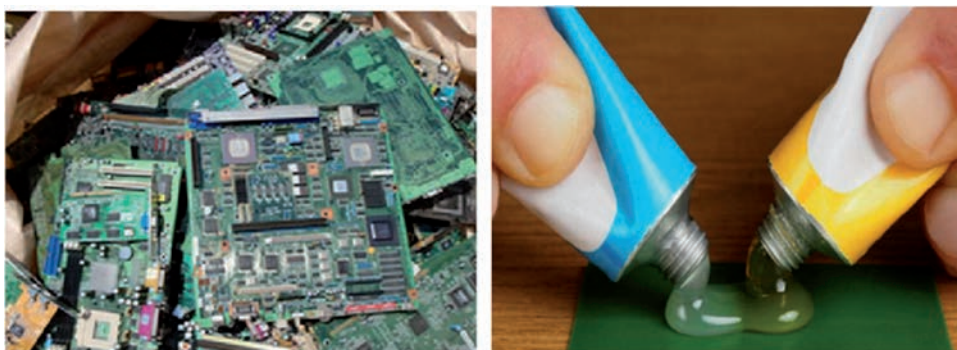


Figure 4.
Pictures of some applications of epoxy resin adhesive [23].

which they possess such as good bonding performance; low-temperature curing; waterproof, heat and chemical resistance; low shrinkage on cure, absence of volatile solvents, low creep, product durability; and improved engineering efficiency [4, 7]. They also account for almost 40% of market value, making them the greatest single end use. Epoxy resin adhesives have the ability to adapt to many material surfaces, including metals, wood, concrete, glass, ceramics, and many plastics compared to traditional joining methods, and this is due to the presence of polar hydroxyl and ether groups in the resin [19]. Also, different curing agents used in epoxy resins such as amines, aromatic amines, modified amines, polyamides, anhydrides, or acids offer different properties, resulting in their diverse and versatile properties. Their high resistance to environmental factors such as water, chemical exposure, pressure, and elevated temperature makes them suitable for replacing mechanical bonding methods such as screwing, drilling, and chiseling [20]. Epoxy adhesives are used for metal bonding, bonding of skin and core in sandwich composites, concrete bonding and wood adhesive, and a variety of other applications as stated in **Table 2**.

The use of adhesives in joining different parts together offers numerous advantages including the elimination or reduction of the need for mechanical fasteners which reduces cost; reduction of occurrence of fatigue cracking compared to metal connections as drilled holes for fasteners are potential sites for the initiation of fatigue cracks and provision of a lighter bond which reduces the overall weight of the part and enables the design of smooth external surfaces [34].

Epoxy for adhesives applications comes majorly in two forms; epoxy film adhesives and epoxy paste adhesives. Film adhesives provide a high strength, durability, and resistance to environmental factors to a bonded joint and has found applications mainly in aircraft structures such as wing structures, fuselage panels, honeycomb core components, and lightning strike protection [35, 36]. The film is applied by placing it in between the components to be bonded and then heated and pressurized to form a rigid bond. The epoxy paste adhesives come as a one-part system or a two-part system. The one-part systems are prepared and supplied by mixing all formulated components in advance including epoxy resin and curing agent. They are spread on the components and then cured by heat or pressure while the two-part system consists of a resin and hardener which are mixed together first before been applied on a component [37, 38]. **Table 3** shows some of the characteristics of film epoxy adhesive and paste epoxy adhesive.

Epoxy type	Application	Function	References
Novolac epoxy	Bonding of concrete to steel and masonry in buildings, electrical component encapsulation, printed circuit board encapsulation	It gives high thermal, fire, and chemical resistance to the component and also enhances flame retardancy	[21–23]
DGEBA (Diglycidylether of Bisphenol A)	Bonding wings and fuselage of airplane	It gives strong adhesion, corrosion resistance, and thermal stability	[24]
DGEBA-based two-part epoxy with fillers	Chassis and body panels of automobiles	Increases crash resistance and distributes loads efficiently across joints	[25, 26]
Silica filled epoxy	Electronic components packaging	Serves as substrate materials for electronic packaging	[23]
Rubber toughened epoxy	Repair and maintenance of aircraft parts	It enhances fracture toughness, fatigue performance, and thermal stability leading to enhanced structural integrity various conditions	[17]
Silver-filled epoxy	Semiconductor die attachment	It provides electrical and thermal conductivity, strong mechanical bonding, thermal stability, and resistance to environmental factors	[27, 28]
Cycloaliphatic epoxy resin	Potting, encapsulating, and bonding electronic components	It offers high electrical insulation and thermal stability	[29–31]
Modified epoxy (epoxy-urethane)	Windshield bonding and crash-resistant zones	High peel strength, thermal and impact resistance	[32, 33]

Table 2.
Applications of epoxy adhesives.

Film epoxy adhesives	Paste epoxy adhesives
Film epoxy adhesives come in a preformed shape like solid sheets or rolls	Paste epoxy adhesives come in a paste form and are usually viscous and spreadable
They are suitable for bonding components with a flat and large surface areas	They are suitable for bonding irregular, complex or small surface areas and for filling gaps
It does not require mixing or metering, and application is clean and precise	The one-part system does not require mixing while the two-part system requires mixing of the resin and hardener
They are usually cured with heat or pressure	They can be cured at room temperature or with heat
The bonding gives a high strength and consistent performance	The bonding provides a high impact and chemical resistance
They are mostly used in aerospace structures such as honeycomb and sandwich panels	They are mostly used in the automotive, construction, electronic repairs and in general bonding
They possess high thermal stability and highly suitable for elevated-temperature applications	They have a high bond strength and mostly used for room temperature applications
They generally exhibit a long shelf life especially when stored properly	The one-part system has a long shelf life while the two-part system has a shorter shelf life once mixed

Table 3.
Characteristics of film and paste epoxy adhesives [19, 39].

3. Epoxy resin in coatings

Coatings or paints are very common and can be seen almost everywhere such as in cars, on wall buildings and even on household materials. A coating is a thin film of liquid mixtures that are applied on the surface of materials for different functions. They can be applied for decoration purposes, for protection of the surface of a material from corrosion and external environments, and to impart specific properties on a material [40, 41]. Examples of specialized applications of surface coatings include anti-fouling paints for ship hulls and coatings for food and beverage containers to protect the contents in them as shown in **Figure 5**.

Epoxy resin is highly utilized in used industrial coating and has applications in different industries including pipeline protection in the oil and gas industries, warehouse floor sealing, coating of automobile parts and marine equipment, and coating of pipe fitting [43]. They are majorly used to protect material surfaces and to avoid corrosion and decay of metals and other materials.

Epoxy resin coatings provide a great degree of flexibility to a variety of substrates, and this makes them more affordable than other alternative solutions to protection of surfaces [44, 45]. They possess excellent mechanical properties such as resistance to abrasion and corrosive species, toughness, durability, and endurance, and this makes them an excellent choice of surface treatment for different parts in harsh environments. They are used as the protection layer in automobiles, to protect oil and gas pipelines or wastewater pipelines against corrosion, to improve the performance of metals used in factories, concrete flooring, and other areas with heavy footage. They are also used to improve the corrosion resistance of painted materials by incorporating them as binders in paints [1]. They have found applications in several industries including electrical, naval, locomotive, and industrial production application.

Epoxide resin and polyamine hardener are incorporated together to induce an electrochemical reaction which forms epoxy coatings. When the two materials are mixed together, curing occurs after some hours, and the wet epoxy layer is converted

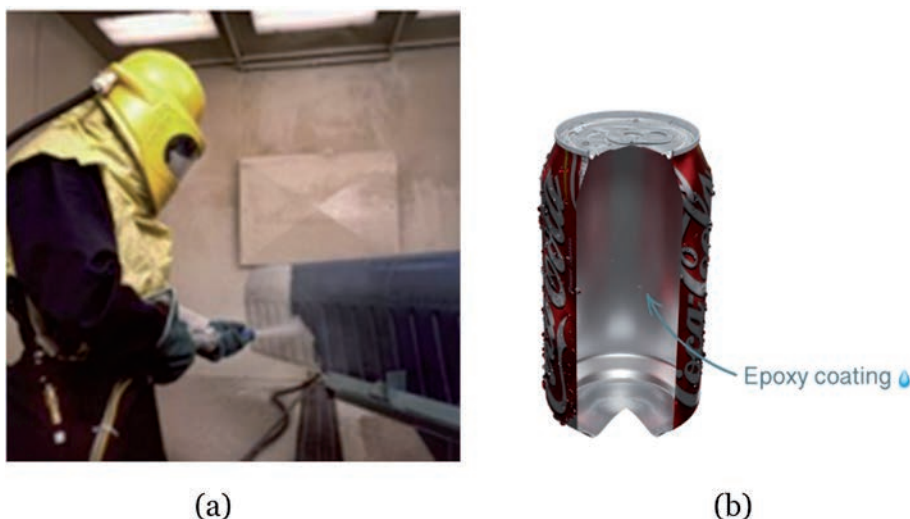


Figure 5.
Liquid epoxy coating on (a) steel pipes [42] (b) food cans.

to a tough and durable solid after which they can be used for different application. They can be incorporated with other resins such as phenolics, alkyds, and amino-resins, they can also be esterified by heating with resin acids. Although some solvent-based methods were once employed, they have mostly been phased out for safety and health reasons. Example of use as surface coatings is the two-part epoxy system highly used in marine industry for prevention of corrosion on ship and boats parts made from steel. For fluidized bed coating of metal parts to prevent corrosion, epoxy coating systems are also offered in powder form, complete with an appropriate hardening system. Electrostatic spraying is an additional method of processing them. Preheating metal parts to 150°C is a standard practice in these applications, and this is done to promote adhesion and start the coating system's curing process. After this, reheating of the coating is done to completely cure the resin. Powder coatings are highly helpful for the application of thick coating layers to items with irregular and complex shapes since they are electrically and chemically resistant. Because epoxy is reactive, the coatings made from them come out tough and adhere firmly to the substrate compared to thermoplastic powders. Intumescent coatings used in steel constructions' fire protection are also frequently made from epoxy resins. In this case, heat flux causes the resin phase to expand 10–20 times, gradually transforming it into a carbonaceous char with reduced thermal conductivity that protects the metallic material underneath [7].

3.1 Epoxy corrosion resistance coating

Epoxy resin has gained widespread application in anti-corrosion materials due to its exceptional mechanical qualities, which include corrosion protection, resistance to chemicals, and high adherence to metal surfaces. Epoxy resin containing two or more epoxy groups is commonly used for this application, and also due to the presence of several functional groups in them, they can be tailored to meet different applications. By undergoing condensation events with hydroxyl and isocyanate groups and ring-opening reactions with amino and carboxyl groups, the epoxy and hydroxyl groups can facilitate modification [46].

Several modified epoxy resins have been developed by to enhance corrosion resistance. Organosilicon resins have been added to epoxy to improve corrosion resistance of some materials by reducing the surface energy and forming hydrophobic layers; adding polyaniline to epoxy coatings increases its conductivity and enhance corrosion resistance by producing a passivating effect on the substrate [47–49].

3.2 Road and bridge coatings

Worldwide, the use of polymers in construction materials is growing daily, and epoxy resin is crucial to the creation of effective building materials [50]. Concrete and masonry mortar based on epoxy resin in particular have a lot of promise since they offer intriguing features, qualities, and uses [51]. Epoxy resin-based materials have proven to be able to meet many of the requirements of both present and future building projects while also overcoming many of the limits of traditional Portland cement-based materials. The surfaces of roads and bridges have been repaired and protected with epoxy resin coatings. Many polymers and traditional Portland cement have a longer setting time and weaker adhesion, and thus, they cannot be used as binding materials when restoring older buildings. Conversely, epoxy has a high level of adhesion, and by altering the kind and amount of hardener in the mixture, the setting

time of mortar and concrete made with epoxy resin may be customized [50]. They are used for the treatment of spalling which is a problem normally encountered on roads and bridges. Spalling occurs due to the accumulation and combination of thaw-freeze cycles, road gritting, and spillage by vehicles. Epoxy resin with amine hardeners at room temperatures is commonly utilized for this treatment. The resin and hardener formulations form a film when coated on a structure, and it prevents spalling on bridge surfaces and road structures. It also slows down the growth and propagation of cracks when used for sealing cracks on these structures. It bonds the damaged parts into an integral part and prevents the penetration of water into the structure. Epoxy resin can also be used for several purposes in construction such as road marking, fuel spill prevention, lightweight surfacing for bridge decks, waterproof layer between concrete and toppings, repairing slippery road surfaces, and repairing roads made from concrete [52].

The properties of cured epoxy resin depend on the type of epoxy resin, the curing agent used, and the quantity of curing agent used. Because the qualities of cured epoxy resins rely on the kind of epoxy resin, curing agent, and curing technique; if epoxy resin, hardener type, and hardener concentrations are not chosen appropriately for use in a composition, the material may not perform its intended function [53]. It is therefore important for people involved in construction works to study polymeric materials technologies so as to be able to utilize the versatility that polymeric materials provide for the development of the polymer-modified concrete technology.

4. Epoxy composites

The exceptional properties of epoxy resin such as chemical stability, high strength, and thermal resistance have spurred their use in the development of composite materials. Epoxy composites have become indispensable in various high-tech industries including the aerospace, marine, automotive, construction, electronics, sports, and even in biomedical application [54]. Different forms of reinforcement such as fibers (natural and synthetic), fillers, and additives are usually incorporated into epoxy resin to make epoxy composites. The addition of these reinforcements helps to achieve the desired property of the composite such as improved mechanical properties (like tensile strength and fracture strength), electrical conductivity, thermal stability, biocompatibility, environmental resistance, and environmental sustainability [55].

The ability of epoxy resin to be incorporated with various types of fibers and fillers compared to other thermosetting polymers have enabled the achievement of these specific properties. Additionally, they mix well with aramid, glass, carbon, and natural fibers, allowing for the optimization of features including stiffness, delamination resistance, and interlayer strength [55]. Fatigue resistance and other mechanical attributes of epoxy composites are greatly enhanced by the addition of nanoparticles like silicon dioxide (SiO_2) or titanium dioxide (TiO_2). Additionally, the incorporation of graphene into epoxy resin matrices results in a significant improvement in electrical conductivity and thermal shock resistance, creating new opportunities in sophisticated technologies such as electronics [54, 56, 57]. In short-fiber and hybrid graphene composites, the combination of fibrous and particle fillers has a synergistic effect that improves resistance to delamination and cracking. One type of hybrid reinforcement that increases toughness and ensures better stress transfer within the material is polyaryletherketone interlayers. These advancements enable the creation of composites with remarkable properties for challenging conditions [54, 58].

Due to various environmental benefits and increased environmental awareness, environmentally friendly products are the focus of industrial companies, and sustainable development is becoming more and more popular worldwide. Researchers keep exploring the production and development of renewable composite materials that are fully or partially bio-based, known as biocomposites. Prior to this awareness, synthetic fibers and fillers have been used to produce epoxy composites; however, certain limitations like high energy demand, high cost, adverse environmental impact, and non-biodegradability have spurred researchers to look for an alternative such as natural fibers and fillers [59]. Natural fibers are used in the making of several eco-friendly materials and offer numerous advantages such as reduction of pollution, conversion of waste to valuable materials, reduction in the depletion of petroleum reserves, and reduced materials and energy usage [60]. Natural fiber-based epoxy composites or epoxy biocomposites consist of natural fibers or filler in the epoxy resin matrix and are less expensive and more environmental friendly than synthetic fiber epoxy composites and have found numerous applications in automobiles, aircraft, constructions, electronics, and other industries. Some of their applications in this field is shown in **Table 4**. Epoxy resin has also been reinforced with natural fibers like sisal, coir, bagasse, pineapple, agave, kenaf, jute, egg shell powder, snail shell powder, chitosan, HAp to obtain different properties such as high stiffness and strength, reduced weight, biocompatibility, and most especially, environmental degradability. The mechanical properties of epoxy resin reinforced with some of these fillers and

Industry	Applications	Function
Marine	Boat hulls and decks, buoys and underwater equipment	They provide resistance to water, saltwater, and corrosion, thereby ensuring long-term durability in harsh marine environments
Sporting goods	Bicycle frames, golf clubs, tennis rackets, and skateboards	They provide an aspect ratio by reducing the weight of the components while enhancing their performance
Aerospace	Wings, fuselage panels, engine parts, honeycomb structures, and interior structures	a. They improve fuel efficiency and performance by reducing the weight of the components. b. They also provide excellent mechanical properties and durability. c. They provide resistance to heat and flame
Automotive	Automobile body panels, underfloor protection	Reduced weight leads to improvement in fuel efficiency while maintaining strength and durability
Electronics	Printed circuit boards, lighting poles, and power line insulators	d. They serve as materials for encapsulation and potting of sensitive components. e. They also offer excellent electrical insulation properties
Biomedical	Implants, prosthetics, and imaging (MRI)	They provide biocompatibility and durability, thereby ensuring safe and reliable performance
Structural	Beams, panels, and insulated boards	They provide high strength and durability
Chemical	Subterranean storage tanks, composite containers for substances, ducts, and stacks	f. They offer resistance to chemicals and corrosion g. They provide thermal stability, strong adhesion, and structural integrity

Table 4.
Applications of epoxy resin in composite materials [60–62].

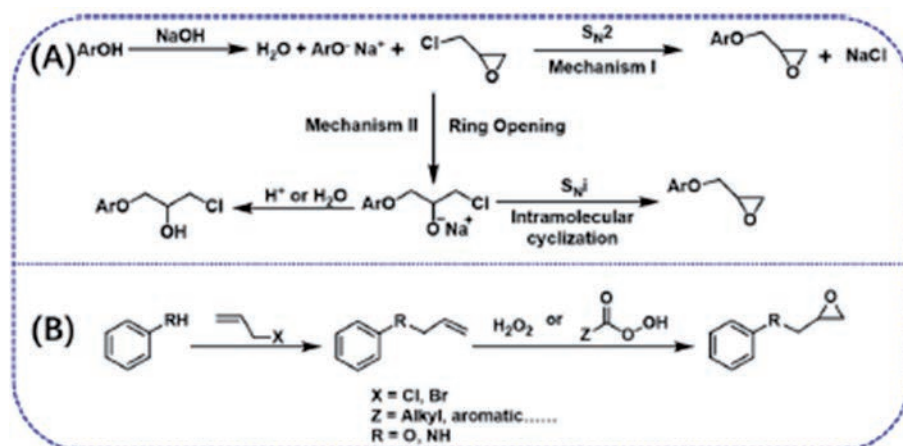
fibers is presented in **Table 5**. They have been used for the production of parts such as multipurpose panels, roofings, storage tanks, beams, and pedestrian overpasses.

Among the novel materials utilized in composites are nanomaterials, also known as nanocomposites, which stand out from traditional composite materials because of their superior mechanical qualities. They are used in different forms such as polymer-clay nanocomposites and carbon nanotubes to improve the properties of materials used in high-performance applications like aerospace, biomedical industries, and automobile [67]. Carbon-based epoxy composites which are one of the promising materials used in advanced technologies have shown unique chemical and physical properties with the incorporation of nanofillers into them. Likewise, nanoclay-reinforced polymer-based composites have attracted a lot of interest from the academic and business communities because even a small amount of nanoclay shows good optical transparency and significantly improves the mechanical performance of the neat polymers [68, 69].

Epoxy resin development primarily relies on non-renewable petroleum resources, typically epoxy monomers of the DGEBA type. Most of the basic ingredients used to make these thermoset resins are hazardous to human health. These epoxy resins are made with compounds that have the potential to harm human health and the environment, such as causing cancer and disrupting hormones. The creation of epoxy resins generated from biological sources is therefore gaining traction as a more sustainable and environmentally friendly substitute. Compounds of biological origins include lignin, rosin, vanillin, and eugenol, and vegetable oils (e.g., soybean, linseed, and castor oil), rosin, and sugars/carbohydrates like sorbitol and isosorbide offer a good starting point for the creation of environmentally friendly epoxy resins. Phenolic rings found in these compounds are perfect for producing high-performance polymers. Using biomass as a raw source, bio-based epoxy resins have been the subject of extensive research in recent decades. To increase epoxy resins' uses and competitiveness, bio-based epoxy resins with functional attributes such as recyclability, flame retardancy, degradability, and antibacterial properties have been developed [70–72]. Applications for these resins are numerous and include 3D printing, composites, adhesives, and coatings. With the growing demand from consumers for sustainable products, the development of bio-based epoxy resins is expected to continue, driven by their balance of performance, cost-efficiency, and environmental benefits.

Epoxy resin type	Bio-Filler/ Reinforcement	Tensile strength (MPa)	Flexural strength (MPa)	Modulus (GPa)	Impact resistance (kJ/ m ²)
Epoxy (DGEBA)	Flax fiber	75–100	120–140	5.5–6.0	35–40
Epoxy (DGEBA)	Jute fiber	12.46	39.08	—	2.63
Epoxy (DGEBA)	Hemp	75.14	126.07		
Epoxy (DGEBA)	Coir	13.05	35.04	—	175
Epoxy	Sisal	132.73	288.6		
Epoxy (DGEBF)	hemp	60–85	100–130	4.5–6.5	30–38
Bio-based Epoxy (e.g., epoxidized linseed oil)	Flax fiber	45–65	75–95	3.0–4.2	22–28

Table 5.
Epoxy resins vs. mechanical performance in bio-based composites [63–66].

**Figure 6.**

Bio-epoxy resin synthesis using phenolic chemicals by (A) glycidylation reaction and (B) double-bond oxidation [70].

Bio-based epoxy composites can be synthesized through various methods, such as glycidylation reactions using plant phenols and epichlorohydrin, double-bond oxidation with oxidative reagents like hydrogen peroxide, or the introduction of acetal structures using aldehydes and polyols as shown in **Figure 6**. These processes leverage renewable resources like bio-oil, vanillin, and glycerol to create epoxy resins with unique properties. Functionalization techniques can further enhance these resins with flame retardancy, recyclability, or shape memory capabilities. While the addition of dynamic bonds or the formation of reversible dynamic covalent bonds (like ester bonds) during the curing process creates recyclable bio-based epoxy resin or shape memory epoxy resin, the addition of elements like phosphorus or structures that produce high char yield at elevated temperatures, such as rigid structures or heterocyclic structures in the intermediate, is used to create flame retardancy or recyclability properties [70, 73–75].

5. Other applications

Epoxy resin being a highly versatile polymeric material has also found applications in electronics and insulating materials. Their capacity to function dependably in harsh environments, such as high temperatures, high humidity, and UV radiation exposure, is the main factor behind their widespread use. They also have good mechanical qualities, resistance to chemicals and water, and exceptional electrical and dielectric insulating qualities [76–79]. Epoxy resin can be used for sealing electronics devices made from metals and other plastics. They also serve as coatings for protecting materials from impact, shock, vibrations, moisture, and chemicals [80]. Epoxy resins have been employed in a range of electronic components including switch gear, motors, and generators [81] as shown in **Table 6**.

Epoxy resins are also widely used in biomedical applications for different functions. Their properties such as biocompatibility, mechanical strength, flexibility, lightweight, and resistance to harsh environments have spurred their usage. In human clinical settings, collagen-based epoxy composites have been utilized as aortic heart valves, vascular grafts, and wound dressings; there is a lot of

Materials used	Electronic component	Uses
Epoxy resin + filler (silica, fiberglass) + additives (flame retardants, coupling agents, and pigments)	Semiconductor	Protecting integrated circuit components from environmental factors like humidity, temperature fluctuations, radiation, moisture, and physical harm
epoxy–fiberglass composite	electronics and microelectronics, radio	Sealant for chip and printed circuit board assembly
Epoxy (BPA/BPF) + flexible modifiers (polyurethane hybrids) + alumina/silica fillers	Transformers, PCBs, and sensors	Seals components from environmental damage such as humidity, chemicals, and vibrations
Silver-filled epoxy (epoxy + Ag flakes) or thermally conductive fillers (AlN, BN)	Heat sinks and capacitors	Bonds components while providing thermal/electrical conductivity or insulation.
Epoxy novolac resin + phenolic hardeners + silica/alumina fillers	Microchips, diodes, and transistors	High-temperature resistance, protects against mechanical and chemical damage.
Epoxy resin + boron nitride (BN) or aluminum oxide (Al_2O_3) fillers	CPUs, power modules	Dissipates heat while maintaining electrical insulation
Cycloaliphatic epoxy + UV stabilizers + low-stress hardeners	LED encapsulation and displays	Maximizes light transmission while protecting against yellowing and UV damage
Epoxy (BPA or cycloaliphatic) + silica nanoparticles + toughening agents (rubber/CTBN)	Flip-chips, BGAs	Reinforces solder joints, prevents cracking under thermal cycling

Table 6.
Uses of epoxy resin polymer in electronics [79, 82–84].

promise for shape memory polymer foams in biological applications like embolic sponges. Because of their exceptional chemical inertness, low electrical and high thermal conductivities, wide optical transparency, and other special qualities, nanodiamond-epoxy derivatives have found extensive use in biological systems (**Figure 7**) [23, 85].



Figure 7.
Applications of epoxy resin in biomedical engineering (a) Prosthetic limbs [86, 87] (b) 3D printed maxillofacial.

6. Future prospects

Epoxy composites have the potential for further development in terms of their properties and applications, which could significantly impact their use in a wide range of industries. The prospects for future development are based on improving mechanical properties, standardizing for extreme conditions, and developing sustainable materials with little or no adverse effect on the environment. The epoxy resin market is projected to grow from \$12.64 billion in 2024 to around \$13.76 billion in 2025, with a compound annual growth rate (CAGR) of 8.9%. By 2029, the market is anticipated to reach \$19.93 billion, driven by advancements in electronics, compatibility with biological materials, circular economy initiatives, and the proliferation of electric vehicles [88]. The growing use of bio-based formulations, which are more environmentally friendly and sustainable, is one of the major developments. Also, it is anticipated that the development of smart epoxy resins with sensors for real-time monitoring, rapid curing methods for quicker production, and improved adhesion qualities would increase the range of uses for these resins. Because epoxy resins are lightweight, strong, and long-lasting, the market is also benefiting from the growing demand in the electronics, automotive, and aerospace industries. Because of growing industrial demand, environmental initiatives, and technological advancements, the epoxy resin market is anticipated to expand rapidly.

7. Conclusion

Epoxy resin demand has grown significantly due to technological advancement in various fields and is predicted to continue doing so in the years to come. Epoxy resins have found applications in various industries ranging from automotive and aerospace to constructions, electronics, and biomedical engineering due to their unique combination of high mechanical strength, good chemical resistance, and high thermal stability. There have also been numerous advancements in the production of bio-based and functional epoxy resin to enhance sustainability and due to environmental concerns. The spur in the development of more sustainable and high-performance epoxy resins will definitely drive their future applications making them an indispensable material in the global market.

Conflict of interest

The authors declare no conflict of interest.

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Lignins and Epoxidized Lignin-Based Biopolymers

Andreea Creteanu and Madalina Vieriu

Abstract

Lignins are some of the most common natural polymers on Earth. They are located in vascular plant cell walls that are formed by randomly crosslinked phenolic molecules ensuring that plants are mechanically protected. Those biopolymers also possess antioxidant and antibacterial effects, aiding plants fight against biological and chemical attacks. The industrial and medical vastly fields benefit from the use of lignins and lignin-based biopolymers to produce antibacterial agents, antioxidant additives, UV protection agents, hydrogel-forming molecules, nanoparticles, and reinforcing agents or fillers in composite materials. By altering the lignin structure through epoxidation, lignins can be customized for particular uses and provide greater sustainability than conventional petrochemical-based resins. Green and sustainable methods of producing polymers have brought lignins and their derivatives at the top of research themes list because of their positive effects on environmental impacts of greenhouse gas emissions. The mechanical, thermal, and chemical characteristics of lignin-based epoxy resins are used in flame retardants with a high capacity for charring, in adhesives with superior mechanical and thermal qualities, and in coatings with UV-blocking and anticorrosion qualities. Epoxidized lignin-based biopolymers also show great potential in medical fields.

Keywords: lignin, epoxidation, natural polymer, formulations, medical field

1. Introduction

Lignin, a common byproduct of the paper and pulp industry, serves as a renewable raw resource. It is a biomass-based phenolic copolymer, serving as a cost-effective and appealing source of aromatic chemicals. Lignin and cellulose are the most prevalent natural polymers on Earth. The primary physiological role of lignin is providing plant cell walls stiffness and durability. Lignin enhances mechanical strength to the degree that towering trees, exceeding 100 meters in height, can maintain their upright posture [1–3].

The growing apprehension over environmental challenges, including fossil fuel depletion and climate change, has positioned wood and wood-based materials in the spotlight because of their positive impacts on lowering carbon footprints and greenhouse gas emissions. The potential for wood to be utilized in innovative and challenging construction projects mostly hinges on adhesive technology. The critical concern must encompass the transition to eco-friendly adhesives, the reduction of carbon

dioxide emissions, and the adoption of entirely sustainable solutions. The environmental sustainability of engineered wood products, recognized as the sole renewable and sustainable construction material, is influenced by the adhesives employed, which are often synthesized from oil-derived raw materials such as petroleum and natural gas [4, 5].

Certain compounds, like cellulose and sweet liquor from wood and its byproducts, are combined in order to produce lignin. The demand for lignin will continue to grow due to factors like rising construction costs and the growing demand for electronics and cars [6].

Because of their established pulp and paper industries, which produce significant amounts of lignin as a byproduct, Europe and North America are the top markets for lignin-based dispersants. Due to its high availability in those areas, a great deal of research and development has been done to identify alternative uses, which is anticipated to boost market expansion in the near future [7].

According to research, the size of the worldwide lignin market is expected to grow from almost 1.3 billion US dollars in 2024 to 2 billion US dollars in a decade [7].

2. Lignin

The term “lignin” originates from the Latin word “lignum,” which signifies wood. Lignin constitutes around 30% by weight of wood, making it the second most prevalent component after cellulose. In contrast to cellulose, which is a linear polymer

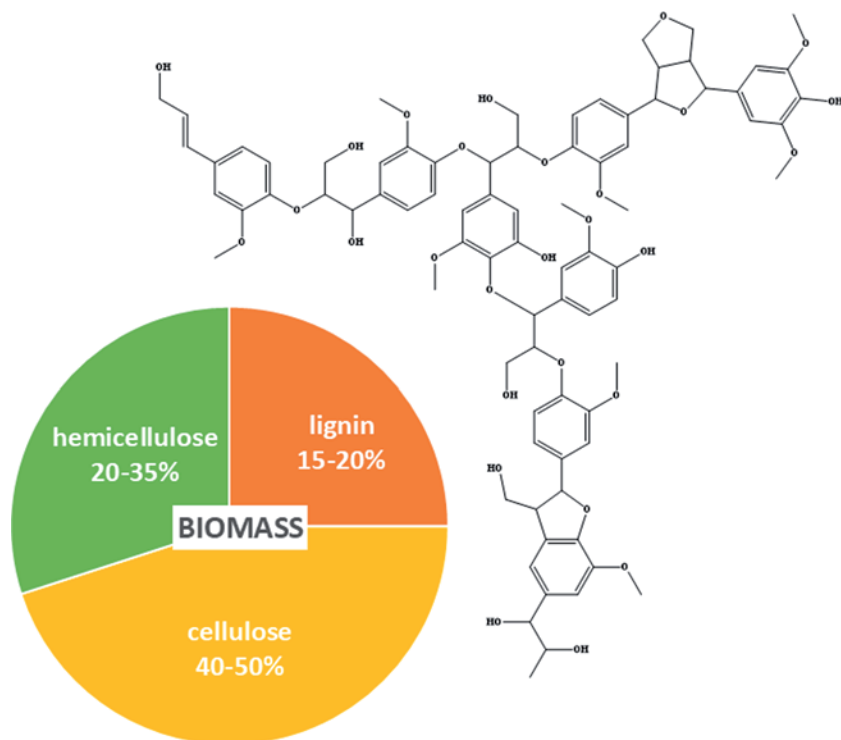


Figure 1.
Structure of lignin and the proportion of the major components of biomass.

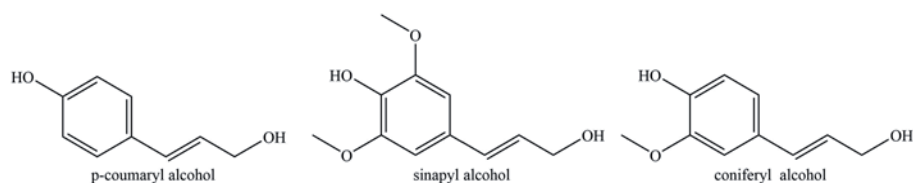


Figure 2.
Primary lignin precursors.

with partially crystalline structure, lignin possesses a non-crystalline structure, and it is a non-carbohydrate polymer rather than a polysaccharide. Because of its qualities, lignin is a substance that shows promise for a number of industrial uses. **Figure 1.** provides a simplified depiction of lignin's intricate chemical structure [1].

A method called delignification is used for extracting lignin from biomass. Lignin is mostly aromatic and almost insoluble in common organic solvents, such as pentane, benzene, toluene, and naphthalene. Lignin can be considered a mixture of several compounds that possess analogous chemical structures. Compounds with identical molecular weight but distinct molecular structures are referred to as isomeric compounds. Compounds with varying molecular weights but identical structures are referred to as homologs. Lignin is not classified as isomeric compounds nor as homologs [8, 9].

Most plants conduct a conversion process called lignification, which uses carbohydrate intermediates to synthesize the aromatic polymer lignin from carbon dioxide. The synthesis of lignin precursors, such as trans-p-coumaryl alcohol, trans-p-sinapyl alcohol, and trans-p-coniferyl alcohol, is the first step in the two-stage metabolic mechanism of lignification. Their chemical structures are presented in **Figure 2.** The second step includes the conversion of those precursors into lignin [10].

Because it is a polymer of such aromatic precursors, lignin processing can be used to provide a variety of aromatic compounds such as p-hydroxybenzoic acid vanillin, vanillyl alcohol, resveratrol, gallic acid, xiamenmycin, and gatrodin [11–13].

3. Lignin-based epoxy resins

Environmental legislation has focused on a green design methodology and the production of bio-based adhesives from raw materials to foster a more sustainable and conscientious society. Consequently, developing an eco-friendly adhesive from renewable materials is pivotal in addressing the current sustainability requirements of the wood industry. In structural applications, it is essential for the adhesive joint to possess strength equal to or exceeding that of wood. An efficient adhesive for wood necessitates an array of characteristics such as steady bond performance, good water resistance, suitable viscosity, and mass uniformity. Traditional wood adhesives are formulated using phenol formaldehyde, urea formaldehyde, melamine formaldehyde, and polymeric diphenylmethane diisocyanate resin. Epoxy resins are thermosetting polymers with a variety of characteristics related to their structure and uses, such as advanced composites, coatings, and adhesives in constructions. Epoxy resins serve as efficient chemical cross-linkers, demonstrating superior attributes such as robust bonding capability and corrosives and heat resistance. The predominant form of epoxy resin is diglycidyl ether bisphenol A due to its high reactivity with several curing agents, including amine hardeners, isocyanates, and acid anhydrides, all of

which are often detrimental to the environment. Substituting those curing chemicals (hardeners) with more environmentally friendly alternatives presents currently a significant technological challenge for numerous researchers [4, 7, 14].

It is essential to identify bio/raw materials suitable for epoxy resin synthesis without significant modification or functionalization. Research and industry fields have shown a great deal of interest in bio-based thermoset polymers derived from natural resources such as sugars, polysaccharides, vegetable fats, lignins, lipids, proteins, and other monomers. In addition to promoting the development of effective and environmentally friendly technologies and consumer goods, the conversion of waste biomass and industrial byproducts, such as tannins and lignins, into specialty value-added chemicals and materials unwittingly draws attention to the issue associated with agricultural and forest waste. The primary focus is on natural phenolic compounds, tannins, and lignins, which are suggested as viable alternatives to commercial or manufactured epoxy resins and curing agents [4, 15].

Lignin-based epoxy resins can be obtained through several methods, primarily involving the chemical modification of lignin to introduce epoxy groups, followed by curing with a hardener. Those methods include direct epoxidation of lignin, epoxidation after lignin modification (e.g., methylation), and blending lignin with petroleum-based epoxy resins.

Here is a more detailed look at the methods [16]:

1. Direct Epoxidation of Lignin:

That involves reacting lignin with an epoxidizing agent, like epichlorohydrin, to introduce epoxy groups into the lignin structure. That method can be applied to both unmodified and modified lignins.

2. Epoxidation After Lignin Modification

- **Methylation:** Lignin can be modified with formaldehyde or glyoxal to introduce methylol groups, which can then be epoxidized.
- **Phenolation:** Lignin can be reacted with phenol to create phenolated lignin, which is then epoxidized.
- **Other Modifications:** Other chemical modifications, such as demethylation, can also be used to enhance lignin's reactivity for epoxidation.

3. Blending with Petroleum-Based Epoxy Resins:

To make lignin-epoxy composites, lignin can be mixed with commercially available epoxy resins, such as diglycidyl ether of bisphenol A. The addition of lignin in such formulations is used frequently to further improve the epoxy resin's toughness or thermal stability [4, 17].

4. Other Methods:

- **Depolymerization:** Lignin can be partially depolymerized to create smaller molecules with more reactive sites for epoxidation.

- **Organosolv Lignin:** Lignin extracted using organic solvents (organosolv lignin) can be more easily epoxidized due to its lower polydispersity and higher purity.
- **Enzymatic Hydrolysis Lignin:** That type of lignin, obtained from enzymatic hydrolysis residues, has shown potential for epoxy applications due to its chemical reactivity.

Key Considerations:

- **Lignin Source:**

The type of lignin (e.g., kraft lignin, organosolv lignin, and enzymatic hydrolysis lignin) can influence the properties of the resulting epoxy resin [18].

- **Epoxidation Conditions:**

The reaction conditions (e.g., temperature, catalyst, and solvent) during epoxidation are essential to the reaction's effectiveness and selectivity.

- **Curing Agents:**

The choice of curing agent (hardener) is also important for achieving desired properties in the final epoxy composite.

In summary, the creation of lignin-based epoxy resins involves a variety of chemical and physical modifications to lignin, enabling its incorporation into epoxy formulations for various applications [14, 16, 19].

4. Epoxy modification of lignin

There are several methods for the epoxidation of acetone soluble fraction of kraft lignin (AKL). Effectively introducing epoxy groups into the lignin structure is achieved through lignin epoxidation through the reaction with epichlorohydrin. The best synthesis conditions were found to be 8 hours at 55°C with a 6.3 NaOH/L molar ratio that provided a 99% yield and a high epoxy content (approximately 8). The AKL reaction with epichlorohydrin was used to epoxidize lignin at a molar ratio of 1:1.5 (wt%) lignin to NaOH. Dimethylformamide (DMF) was employed as an additional solvent because all of the lignin samples had good solubility in it. 0.8 g of tetrabutylammonium bromide were added as catalyst to the lignin solution in DMF containing 80 g of epichlorohydrin after 8 g AKL sample had been dissolved in 40 g of DMF and stirred for 10 minutes at room temperature. At 60°C, the reaction mixture was refluxed and stirred for 3 hours. To the reaction mixture that had cooled, 50 mL of a 40 weight percent NaOH solution containing 1.2 weight percent tetrabutylammonium bromide was then added dropwise. When 1000 mL deionized water was added to the reaction mixture to precipitate epoxidized lignin, the reaction proceeded for 8 hours at room temperature. After vacuum filtering out the surplus solvent, the epoxidized lignin was extracted and repeatedly rinsed with deionized water to get rid of the salt that had been produced and the unreacted epichlorohydrin. The epoxidized lignin was then dried for 48 hours at 40°C and 76 kPa in vacuum [4].

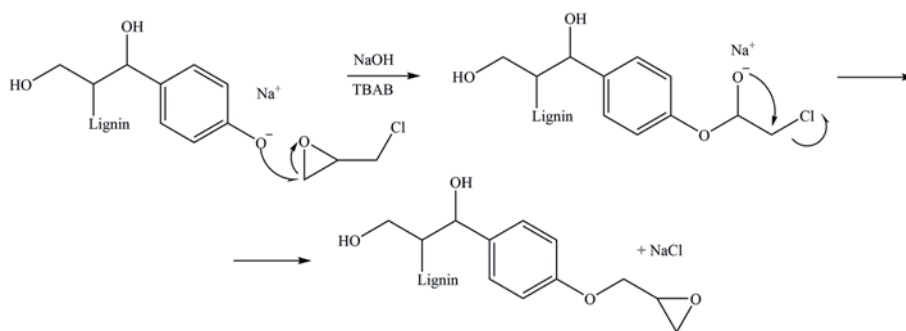


Figure 3.
Lignin epoxidation reaction.

In compliance with ASTM D 1652, the epoxy equivalent weight of the epoxy lignin was determined. In a 150 mL flask, 0.4 g of the substance was dissolved in 10 mL of methylene chloride. Next, a few drops of crystal violet indicator were added to the flask along with 10 mL of tetraethyl ammonium bromide 0.25 g/mL solution in glacial acetic acid. Using a standardized 0.1 M perchloric acid, the solution was titrated until the endpoint showed a color shift from vivid blue to green [4, 17, 20].

Lignin was effectively epoxidized under the specified reaction conditions, resulting in the formation of a black powder-epoxy lignin. The lignin epoxidation reaction shown in **Figure 3** typically requires 1–8 hours, and it is generally conducted in organic solvents due to the limited solubility of epichlorohydrin in water. The inclusion of water may stabilize the highly polar intermediate product in the lignin epoxidation reaction, increasing the reaction rate [4].

Since the reaction is highly exothermic, it is negatively impacted by the elevated temperature. Unmodified lignin exhibits poor solubility in water at pH levels below 10, whereas the epoxidized lignin produced demonstrates water solubility at pH levels exceeding 8. Increased water solubility and decreased intermolecular hydrogen bonding are the outcomes of the reduction of phenolic hydroxyl groups and the addition of the epoxy group. It was determined that the epoxy lignin's equivalent weight was 4.8 mmol per gram of resin, which is less than but equivalent to the commercial diglycidyl ether bisphenol epoxy content of 5.3 mmol/g. Consequently, that synthetic epoxy lignin may serve as an appropriate substitute for epoxy resins [4].

5. The importance of lignin in pharmaceutical industry

Lignin is garnering heightened interest in the medical field owing to its biocompatibility, biodegradability, antioxidant characteristics, and potential as a drug delivery system. It is being investigated for its potential usefulness in cancer treatment, wound healing, and as a prebiotic [21, 22].

There are numerous pharmacological applications of lignin:

1. Drug Delivery:

Lignin has been evaluated as carrier for active pharmaceutical ingredients. Lignin, especially in nanoparticle form, can encapsulate and transport pharmaceuticals,

particularly hydrophobic compounds, thereby rendering them stability and enabling a controlled release.

Lignin can be utilized as an excipient in tablets to augment the drug release rate and enhance the bioavailability of specific drugs, such as aspirin.

Hydrogels with adjustable qualities for use in biomedical devices can be developed with lignin. Functional hydrophilic groups like hydroxyls, carbonyls, and methoxyls are abundant in it and allow for easy chemical modification.

Targeted Drug Delivery: The chemical structure of lignin, characterized by its aromatic and aliphatic hydroxyl groups, facilitates modification with ligands or other chemical entities, so permitting the targeted delivery of drugs to specific tissues or cells [3, 21, 22].

2. Additional Biomedical Applications:

Lignin possesses antioxidant capabilities that may safeguard against oxidative stress and associated disorders, while its antibacterial and antifungal properties can be utilized in wound healing and several other applications. It has been demonstrated that lignin and its derivatives are active against both gram-positive and gram-negative bacterial strains. The disruption of membrane potential, interaction with membrane proteins, inhibition of ATP synthesis, induction of cell death akin to apoptosis, activation of antimicrobial peptides, synergistic effects with other antimicrobials, and modification of microbial metabolic pathways are some of the many mechanisms that determine antimicrobial activity. Because of all of that, lignin may be used as a natural antibacterial or preservative in food, medical, and pharmaceutical products [3, 22].

Depending on its source, extraction technique, and level of modification, lignin's antibacterial activity can change [21].

Combining lignin with conventional antibiotics can increase their effectiveness. Antibiotic resistance can be prevented, and the necessary dosages of antibiotics can be decreased because to the synergistic effects [3].

By chemically altering lignin through procedures such as oxidation, its antibacterial qualities can be improved. More effective and targeted antibacterial activity is demonstrated by modified lignin [3].

Lignin-based materials are applicable in wound dressings and other uses to enhance tissue regeneration and provide protection against infection. As an antibacterial ingredient in coatings, films, and other medical applications, lignin can be more effectively delivered and interact with microbial cells when it is incorporated into nanoparticles [22].

Lignin's biocompatibility renders it a good candidate for applications in bone tissue engineering.

Lignin functions as a prebiotic, fostering the proliferation of advantageous intestinal bacteria, hence enhancing digestive health. That characteristic could be used in functional meals and dietary supplements and is crucial for preserving a healthy digestive tract [23].

It has been discovered that some lignin derivatives have cytotoxic effects on cancer cells, preventing them from proliferating and growing [24, 25].

Some research indicates that by regulating the activity of inflammatory mediators, lignin and its derivatives can lessen inflammation. Strong antioxidant properties are attributed to lignin's phenolic structure, which allows it to scavenge free radicals. That activity can be used to generate medications and health supplements and is helpful in lowering oxidative stress in biological systems [3, 23, 24].

3. Green Chemistry and Sustainability—Renewable Resource:

Lignin is a renewable resource obtained from biomass, rendering it an eco-friendly substitute for certain synthetic compounds utilized in pharmaceuticals.

Lignin-based compounds possess biodegradability, hence diminishing the environmental impact of pharmaceutical production and disposal.

Lignin can be transformed into precursors for the synthesis of active medicinal compounds, hence promoting a more sustainable pharmaceutical sector [3].

Since it efficiently protects against oxidative stress brought on by environmental factors, such as pollution and UV rays, lignin's antioxidant properties make it a crucial component of skincare products. A useful ingredient in anti-aging creams, serums, and sunscreens is lignin. Furthermore, lignin's thickening and emulsifying properties enhance the texture and longevity of cosmetics [24, 26].

6. Environmental applications

Lignin is a useful tool in bioremediation efforts because it may be used by micro-organisms that break down environmental contaminants like industrial chemicals and pesticides.

By strengthening soil structure, boosting nutrient availability, and offering defense against infections, lignin and its derivatives can promote plant growth. More sustainable farming methods may result from that [4, 27].

Lignin has been used as a binder for wood and organic municipal solid waste, improving the thermal and physical properties of the briquette. Typically, biomass, such as agricultural waste or coal, is used to make briquettes. and a binder is needed when a substance cannot produce a strong densified form. By encouraging inter-particulate bonding, it facilitates the formation of the densified form [4, 14].

Renewable basic materials such as bioplastics can be made from lignin. Increased thermal stability, firmness, and UV resistance are characteristics of lignin-based composites that are critical for electronics, packaging materials, and automotive components [3].

Because it may be structurally altered and combined with various polymers to improve strength, thermal stability, and biodegradability, lignin is a useful component for composites. Those lignin-based polymers, which provide sustainable substitutes for oil-derived plastics, are being utilized more and more in construction materials, automobile parts, packaging, and other applications [3, 26, 28–30].

Carbon fibers synthesized from lignin are being researched as a sustainable and affordable substitute for those made from polyacrylonitrile for the automobile, sporting goods, and aerospace industries.

Because of their high conductivity and surface area, lignin-based carbon fibers can also be utilized in energy storage applications, such as in battery and supercapacitor electrodes [31].

Lignin is a sustainable substitute for oil-based adhesives in wood products. Strong bonding qualities are offered by lignin-based adhesives, which are also eco-friendly and emit less formaldehyde [32].

By acting as a binder, lignin can increase the durability and lessen the environmental effect of building materials like concrete and asphalt.

Lignin-derived hydrogels are used for water purification and agriculture [33].

Chemical modification of lignin can yield bio-based solvents, including substitutes for dimethyl sulfoxide. Those solvents provide a more environmentally friendly

option to conventional solvents and are used in a variety of industrial processes, such as chemical synthesis.

Lignin-based products can be employed to soil improvement. By boosting the soil's organic matter content, lignin serves to improve soil stability, minimize erosion, and encourage healthy plant development while lowering the need for artificial fertilizers.

Lignin is helpful in water treatment and environmental remediation because of its capacity to absorb organic contaminants and heavy metals. Heavy metals in the soil can bind with lignin, which lowers their toxicity and bioavailability. Through that chelation process, contaminated soils can be cleaned up and made safer for use in agriculture [27, 32–34].

By encouraging the creation of soil aggregates, lignin can improve the structure of soil. By increasing soil aeration and porosity, those aggregates lessen compaction and erosion. Because of its ability to hold water, lignin helps to improve soil water retention. It can retain vital elements including calcium, magnesium, potassium, and ammonium because of its high cation exchange capacity [35, 36].

By stabilizing the soil's surface, lignin lessens the effects of water and wind erosion.

Lignin supports a diverse and active microbial community in the soil, critical to the structure, nutrient cycling, and general health of the soil. Lignin has the ability to affect how quickly other organic components in the soil break down. Lignin can control the breakdown of plant leftovers by interacting with enzymes made by soil microbes, which over time helps to ensure a balanced release of nutrients.

Mulches based on lignin are used in agriculture to control soil temperature, inhibit weeds, and hold onto soil moisture. Compared to traditional plastic mulches, biodegradable lignin-based mulches are more beneficial since as they break down, they are adding organic matter to the soil, increasing its fertility over time. Although the biodegradability of lignin is a big plus, it might be difficult to make sure the mulch does not break down too quickly and lasts the entire growing season. By lowering evaporation, lignin mulches also aid in the retention of soil moisture [35, 37].

Because synthetic chemicals are avoided in organic farming systems, lignin-based mulches are particularly advantageous. Lignin-based mulches have use outside of agriculture, such as soil improvement, weed control, and esthetics in landscaping. They are a desirable alternative for sustainable landscaping techniques because of their organic look and advantages for the environment. Over time, lignin's slow breakdown increases the amount of organic matter in the soil, enhancing its quality for subsequent plants [38].

Vanillin produced from the pods of the vanilla orchid represents roughly 5% of the world's vanilla. It is the only phenolic compound that can be mass-produced from biomass. Thus, 95% of vanillin is produced artificially, with 15% of the synthetic vanillin coming from lignin. It can be synthesized through numerous methods using ferulic acid and Kraft lignin [13, 39, 40].

7. Conclusions

Lignin is frequently seen as a waste of the paper and pulp industries. Its transformation into profitable goods like minerals, chemicals, and biofuels, however, is consistent with the circular economy's tenets of resource maximization and waste minimization. Industries may lessen their environmental effect and support more sustainable production methods by turning lignin into useful products. Its use in the

production of biofuels, biochemicals, and bio-materials encourages sustainability and helps to lower carbon footprints.

Additional research is required to comprehensively examine lignin's potential in medicines, enhance its extraction and modification techniques, and resolve existing restrictions.

The continuous study of lignin valorization is essential to the advancement of circular economy principles, which turn waste resources into useful products.

Conflict of interest

The authors declare no conflict of interest.

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Preparation and Application of Advanced Low-Dielectric and High-Thermal-Conductivity Epoxy Resins

Zheng Liu

Abstract

Epoxy resins exhibit excellent insulation and mechanical properties, chemical resistance, and ease of processing, which make them widely applicable in printed circuit boards, potting sealants, and high-voltage power distribution networks. However, conventional epoxy resins (e.g., the bisphenol A-type epoxy resin) possess a relatively high dielectric constant ($\epsilon \approx 3.4\text{--}3.6$) and low intrinsic thermal conductivity ($\lambda \approx 0.20\text{ W/(m}\cdot\text{K)}$), leading to significant electromagnetic signal loss and inefficient heat dissipation. As an emerging branch of functional epoxy resins, the preparation of low-dielectric and high-thermal-conductivity epoxy resins has garnered widespread attention, and no systematic summary of this domain is available. Additionally, the testing methods for the dielectric and thermal-conductivity properties of resins have not been standardized, and the performance data obtained by different testing methods are poorly comparable. Thus, this chapter systematically examines the key factors influencing the dielectric and thermal-conductivity properties of advanced low-dielectric-constant and high-thermal-conductivity epoxy resins, as well as the associated testing methods. It then delves into the preparation techniques and recent academic advancements in this field. Finally, it explores the future development trends and application prospects of these materials. It is anticipated that the systematic summary of this chapter will assist researchers in this domain to acquaint themselves with common approaches and frontier research both domestically and internationally, and thereby support them in conducting innovative endeavors.

Keywords: functional epoxy resins, low dielectric constant, high thermal conductivity, mechanism, preparation, application

1. Introduction

Epoxy resin (EP), renowned for its exceptional electrical insulation, mechanical strength, chemical resistance, and ease of processing, is widely utilized in industries,

such as electronics, electrical engineering, and electronic packaging. It holds approximately 70% of the thermosetting resin market [1]. Epoxy resin serves as a critical insulating material in applications ranging from small electronic components to large-scale generators and motors, and from high-voltage power distribution and transmission systems [2].

However, with the development of electrical equipment towards higher voltage and higher capacity, as well as the rapid advancement of electronic information technology, especially the high frequency, digitalization, high power, high density and high integration of electronic components, the internal heat generation of epoxy resin has significantly increased. This accelerates the aging process, affects quality stability, and poses serious threats to the stability and reliability of equipment, components, and high-power systems. Therefore, it is urgent to develop a new generation of low-dielectric and high-thermal-conductivity epoxy resins (**Figure 1**) [3].

Presently, researchers both domestically and internationally employ two primary strategies to reduce the ϵ value of epoxy resins: physical blending and chemical structural modification [4]. Physical blending involves incorporating inorganic nanofillers, such as hollow microcapsules, hollow glass microspheres, and porous ceramic particles, into the epoxy resin to lower its ϵ value. However, this method often introduces new interfaces within the resin, and at higher filler concentrations, agglomeration can occur, which is detrimental to achieving a reduced ϵ value. Chemical structural modification, on the other hand, involves synthesizing and introducing compounds with specific functional groups to alter the epoxy resin's properties [5]. This approach typically includes integrating hollow structures like polyhedral oligomeric silsesquioxane (POSS) or adamantane, which decrease the density of polar molecules in the cured epoxy network. Additionally, low-polarity molecules or groups, such as carbon-silicon (C-Si), oxygen-silicon (O-Si), and carbon-fluorine (C-F), are introduced to reduce the molecular polarizability of the cured epoxy network [6]. Despite these

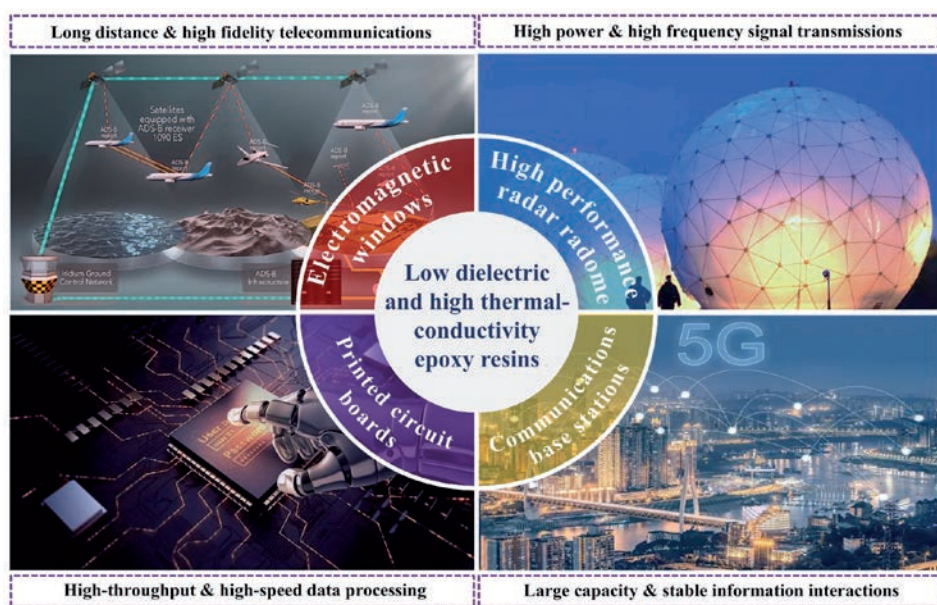


Figure 1.
Schematic diagram of the application for low-dielectric and high-thermal-conductivity epoxy resins.

benefits, incorporating hollow structures into the cross-linked network can hinder the close packing of molecular chains during curing, thereby impeding efficient phonon transmission and negatively impacting thermal-conductivity enhancement. In summary, while both methods aim to reduce the ϵ value, they each present unique challenges that need to be addressed for optimal performance.

To enhance the thermal conductivity of epoxy resins, two primary strategies are commonly employed. The first approach involves incorporating high-thermal-conductivity fillers into the epoxy resin matrix to boost its thermal performance. The second method focuses on increasing the microstructural order within the cured epoxy network, thereby reducing phonon scattering and improving intrinsic thermal conductivity [7]. However, adding excessive amounts of thermal conductivity fillers can introduce interfacial thermal barriers within the composite, leading to increased phonon scattering and hindering further improvements in the thermal-conductivity (λ) value. Additionally, most common thermal-conductivity fillers contain a significant number of free electrons, resulting in higher dielectric constants (ϵ) compared to the epoxy resin matrix. The introduction of new interfaces due to filler incorporation also creates strong interfacial polarization effects, making it challenging to simultaneously enhance both dielectric and thermal conductivity properties. Research has shown that the thermal conductivity of polymer composites is more influenced by the resin's λ value than by the fillers themselves. In contrast, designing and synthesizing intrinsically liquid crystal epoxy resins involves introducing liquid crystal units into epoxy monomers or curing agents. This results in locally ordered, crystal-like structures within the cured network, enhancing the molecular order and promoting closer packing of molecular chains [8]. Consequently, this approach can effectively reduce dipole polarization under an external electric field and minimize phonon scattering, potentially achieving simultaneous improvements in both dielectric and thermal-conductivity properties of the cured epoxy resin products [9]. However, the focuses of the above-mentioned researches are different, and there has been no systematic summary of this field. Besides, due to the fact that the testing methods for the dielectric and thermal conductivity properties of resins have not been standardized, the performance data obtained by different testing methods are not comparable.

Based on this, this chapter offers the first comprehensive overview of the key factors and testing methods related to the dielectric and thermal-conductivity properties of advanced low-dielectric and thermally conductive epoxy resins. Then, the preparation and recent research work in low-dielectric and high-thermal-conductive epoxy resins are summarized. Finally, we explore the future development trends and potential applications of these materials.

2. Intrinsic mechanism and test method of low-dielectric and high-thermal-conductivity epoxy resins

This section primarily delves into the microscopic action mechanisms of dielectric and thermal-conductivity properties for low-dielectric and high-thermal-conductivity epoxy resins, furnishing a theoretical foundation for subsequent preparation approaches. Furthermore, this section elaborately presents the testing methods and their parameters, with the intent of aiding subsequent researchers in selecting appropriate testing methods and standards in accordance with sample characteristics and testing conditions.

2.1 Influencing factors of dielectric properties for low-dielectric and high-thermal-conductivity epoxy resins

The dielectric characteristics of epoxy resins, represented by their dielectric constant (ϵ) and dielectric loss ($\tan\delta$), primarily stem from the polarization capabilities of their cured network structure when subjected to an electric field. The polarization capacity of epoxy resin molecules is typically quantified by molecular polarizability (α). On one hand, the ϵ of epoxy resins signifies the extent of their ability to store electrical charge, serving as a macroscopic parameter that describes the polarization behavior of the epoxy resin's molecular structure. Conversely, α is a microscopic parameter that reveals the polarization features at the molecular level. The Clausius-Mossotti equation establishes the connection between the macroscopic dielectric properties of cured epoxy resins and their microscopic molecular polarization abilities, as illustrated in Eq. (1) [10]:

$$\frac{\epsilon - 1}{\epsilon + 2} \times \frac{M}{\rho} = \frac{N_A}{3\epsilon_0} \times \alpha \quad (1)$$

Among, α , M , and ρ , respectively, represent the molecular polarizability, relative molecular mass, and density of the epoxy resins. N_A is Avogadro's constant, and ϵ_0 is the vacuum capacitance, which is 8.85×10^{12} F/m.

It is evident that the ϵ value of the cured epoxy resin is directly related to its α . Consequently, an efficient approach to decrease the ϵ value of the epoxy resin is to reduce the α of its molecular structure. When subjected to an external electric field, the charge distribution within molecules or specific groups in the cured epoxy resin network alters accordingly. This phenomenon is known as polarization. This includes various types of polarization, such as electronic, atomic, orientational, or interfacial polarization.

Electronic polarization (**Figure 2a**) [11] describes the movement of valence electrons relative to the atomic nuclei within the curing network of epoxy resin. Typically, compared to the strong electric field generated by the atomic nuclei, the external electric field is considerably weaker. As a result, the displacement of valence electrons is relatively constrained due to the robust binding force exerted by the atomic nuclei. Atomic polarization (**Figure 2b**) [12] describes the relative shift of the

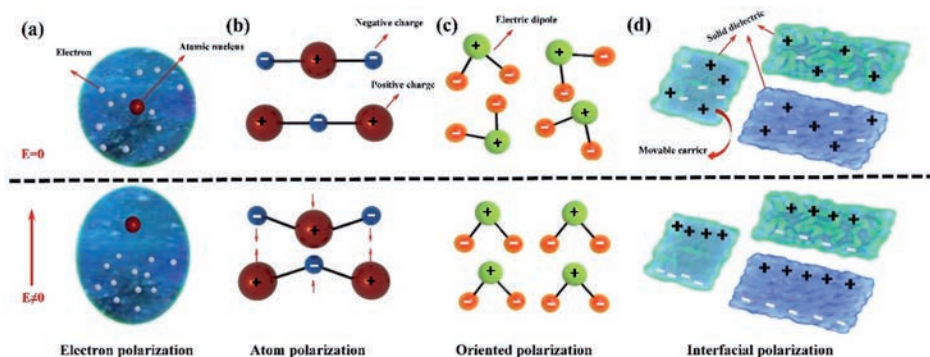


Figure 2. Dielectric polarization diagram: electron polarization (a), atomic polarization (b), orientation polarization (c), and interface polarization (d).

atomic nucleus caused by an external electric field. When subjected to the same external electric field, the movement of the atomic nucleus is significantly less pronounced compared to that of the electron. These two forms of polarization are collectively known as induced polarization, and their polarizability remains constant, regardless of temperature changes.

Orientation polarization (**Figure 2c**) [13] within the epoxy resin curing network pertains to the alignment of dipoles (comprising both the permanent dipoles resulting from polar groups within the molecular structure and the induced dipoles formed when the centers of positive and negative charges of non-polar groups shift under an external electric field) along the direction of the electric field. Commonly, a considerable number of oxygen-containing functional groups exist within the cured epoxy resin network (such as hydroxyl groups generated after the ring-opening reaction of epoxy groups) or polar groups introduced by amine or anhydride curing agents. The permanent dipoles therein will undergo orientation under the effect of an external electric field, thereby generating a macroscopic dipole moment. Furthermore, the molecular structure of traditional epoxy resin curing networks exhibits relatively low symmetry. Under the influence of an external electric field, the centers of positive and negative charges do not coincide, generating induced dipoles, which further augment the orientation polarization effect of the epoxy resin curing network. The extent of orientation polarization in epoxy resin primarily hinges on the number of permanent dipoles (the quantity of polar groups in the molecular structure), the number of induced dipoles (the symmetry of the molecular structure), and the intermolecular interaction forces (the resistance to the directional migration of dipoles). Generally speaking, the lower the number of polar groups in the cured epoxy resin network and the better the symmetry of the molecular structure, the fewer the number of induced dipoles and permanent dipoles generated within the network under the influence of an external electric field, which is more conducive to attenuating the orientation polarization effect. Simultaneously, during the process of dipole deflection and orientation under the influence of an external electric field, it is necessary to overcome molecular inertia and rotational resistance. Hence, the stronger the intermolecular interaction forces, the more unfavorable it is for dipole deflection, which is also beneficial for weakening the orientation polarization effect.

Interface polarization (**Figure 2d**) [14] arises when elements at the interface exhibit varying polarities and conductive properties. When an electric field is applied, this can lead to the accumulation of charges at the boundary between the two materials, resulting in polarization. Such polarization commonly takes place at the interfaces between dielectrics that have differing dielectric constants or conductivities. Additionally, homogeneous polymer dielectrics can experience interface polarization because of internal impurities, defects, or the presence of both crystalline and amorphous regions within the material. Therefore, when adding functional fillers to prepare low-dielectric and high-thermal-conductivity epoxy resins, the increase in interfacial polarization is usually detrimental to the corresponding properties.

The molecular polarizability is usually the sum of the above several polarization forms. Under the action of an external electric field, the lower the degree of polarization of an electric dielectric material, the lower its ϵ value will be. Therefore, from the perspective of polarization, the polarization rate of the epoxy resin molecules can be reduced by designing the molecular structure or adding low-dielectric fillers, and finally a type of epoxy resin with lower ϵ and $\tan\delta$ values can be obtained.

2.2 Influencing factors of thermal conductivity property for low-dielectric and high-thermal-conductivity epoxy resins

Thermal conductivity arises from the interactions and energy transfer between microscopic particles within a material. Epoxy resins, being saturated systems devoid of free electrons, exhibit restricted molecular motion, with phonons serving as the primary carriers of thermal energy [15]. As an amorphous polymer, conventional epoxy resin lacks a well-ordered structure, leading to a thermal conduction mechanism that depends on the random arrangement of molecules or atoms. These entities undergo thermal vibrations around their equilibrium positions and sequentially transfer energy to neighboring units. Due to this disordered and gradual energy propagation, the overall thermal conductivity of epoxy resins is relatively low. By adding functional fillers or chemically modifying the matrix structure of epoxy resin, the microscopic action mechanisms of these two approaches are not completely identical.

2.2.1 Thermal conductivity mechanisms of low-dielectric and high-thermal-conductivity epoxy resins by blending modification

The thermal conduction pathway theory in the presence of fillers is currently the widely accepted explanation for the enhancement of λ in filled epoxy resin composites. When the filler content is low, the fillers are encapsulated by the matrix and cannot form an interconnected conductive network, resulting in a dispersed structure akin to an archipelago [16]. In this case, the matrix dominates the system and exhibits high thermal resistance, leading to poor heat transfer through the matrix, and thus the thermal conductivity of the epoxy resin does not improve significantly. As the amount of thermal conductive fillers gradually increases, the fillers begin to contact each other, forming effective thermal conduction pathways. Heat flows more efficiently along these lower-resistance filler channels, thereby enhancing the thermal conductivity of the epoxy resin. When the filler content reaches a critical threshold, the fillers interconnect to form a continuous thermal conduction network, which dramatically improves the λ of the epoxy resin.

2.2.2 Thermal conductivity mechanisms of the intrinsic low-dielectric and high-thermal-conductivity epoxy resins

When designing and preparing low-dielectric high-thermal-conductivity epoxy resins, it is usually achieved by introducing or creating micro-regularly arranged and oriented structures within the cross-linked network of the epoxy resin to form local crystalline or quasi-crystalline structures, thereby increasing the amorphous or crystalline regions of the epoxy resin (**Figure 3**) [17]. This is because the thermal conduction mechanism of the crystalline part mainly relies on the thermal vibrations in the regular lattice, allowing thermal energy to diffuse smoothly in the polymer, resulting in less energy loss and high-thermal-conductivity efficiency, thereby significantly enhancing the inherent thermal conductivity of the epoxy resin.

However, due to the structural complexity of low-dielectric and high-thermal-conductivity epoxy resins, their thermal conduction mechanism is also intricate. In the intrinsic thermally conductive liquid crystal epoxy resin structure, both crystalline-like regions and amorphous regions coexist. These regions do not form complete crystalline domains but instead consist of numerous mesomorphic regions. Within these mesomorphic regions, liquid crystal units are highly ordered, exhibiting

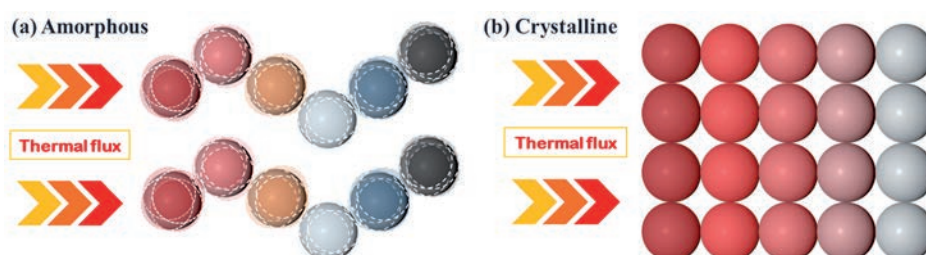


Figure 3.
Thermally conductive mechanism of common epoxy resin (a) and mesomorphic region of low-dielectric and high-thermal-conductivity epoxy resins (b).

local anisotropy. The regular lattice arrangement in mesomorphic regions facilitates heat flow along the orientation direction, resulting in efficient heat transfer. Although the areas between mesomorphic regions remain amorphous, they are interconnected through strong chemical bonds. The disordered arrangement of mesomorphic regions and varying orientation directions lead to macroscopic isotropy in the overall liquid crystal epoxy resin. The presence of mesomorphic regions and the strong covalent bonding between them effectively reduce phonon scattering within the material, enhancing phonon propagation efficiency and significantly improving the intrinsic thermal conductivity of the liquid crystal epoxy resin [18].

2.3 Testing method of low-dielectric and high-thermal-conductivity epoxy resins

2.3.1 Testing method of dielectric properties for low-dielectric and high-thermal-conductivity epoxy resins

The common testing methods for dielectric properties of low-dielectric and high-thermal-conductivity epoxy resins include the resonator cavity method and transmission line method.

2.3.1.1 Resonator cavity method

The resonator cavity method (**Figure 4a**) [19] calculates the values of ϵ and $\tan\delta$ of the sample according to the variations in resonant frequency and quality factor in the resonant cavity (capacitor) when the sample is placed or not. The specific calculation formulas are shown in Eqs. (2) and (3).

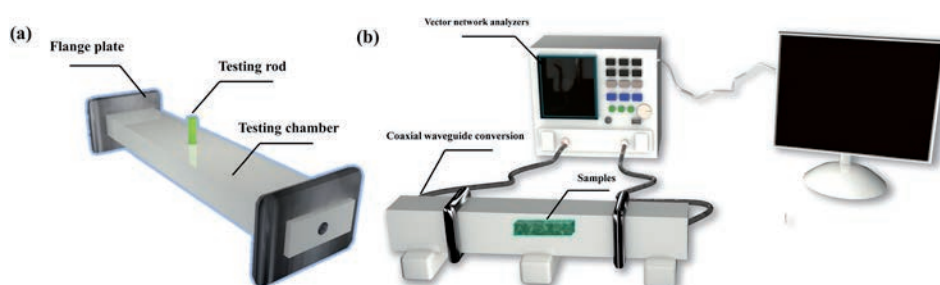


Figure 4.
Schematic diagram of testing principle for resonator cavity method (a) and transmission line method (b).

$$\varepsilon = \varepsilon' = 1 + \frac{1}{2} \times \frac{V}{\Delta V} \times \frac{f_0 - f}{f} \quad (2)$$

$$\tan \delta = \frac{Vf}{4\Delta Vf + 2(f_0 - f)V} \times \left(\frac{1}{Q} - \frac{1}{Q_0} \right) \quad (3)$$

Where, f_0 and f denote the resonant frequencies of the resonant cavity when the sample is not placed and when the sample is placed, respectively; Q_0 and Q stand for the quality factors of the resonant cavity when the sample is not placed and when the sample is placed, respectively; V and ΔV represent the volume of the empty cavity and the volume of the placed sample, respectively.

2.3.1.2 Transmission line method

Transmission line method (**Figure 4b**) [20] primarily measures the scattering parameters (S_{mn}) at both ends of the test sample, thereby obtaining the reflection coefficient (Γ_c) and transmission coefficient (T_d) of the test sample. Subsequently, in accordance with the Nicolson-Ross-Weir (NRW) transmission/reflection algorithm, the real part (ε'), the imaginary part (ε'') of the complex permittivity, and the $\tan \delta$ value of material under test are inversely solved.

Employing the transmission line method and the vector network analyzer, the scattering parameters S_{11} and S_{21} of two ports can be directly measured. Through the measurement of the reflected and transmitted signals, the Γ_c and T_d of polymer-based transparent wave composite materials can be acquired. The specific calculation formulas are shown in Eqs. (4) and (5) [21].

$$S_{11} = S_{22} = \frac{\Gamma_c (1 - T_d^2)}{1 - \Gamma_c T_d^2} \quad (4)$$

$$S_{12} = S_{21} = \frac{T_d (1 - T_d^2)}{1 - \Gamma_c T_d^2} \quad (5)$$

Test methods for microwave dielectric properties		Frequency	Applicable scope
Resonator cavity method	Perturbation method	2 Hz ~ 18 GHz	$\varepsilon = 2 \sim 10$, $\tan \delta = 1 \times 10^{-4} \sim 5 \times 10^{-3}$
	Open cavity method	2 Hz ~ 30 GHz	$\varepsilon = 2 \sim 10$, $\tan \delta = 1 \times 10^{-4} \sim 5 \times 10^{-3}$
	Shielded cavity method	1 Hz ~ 30 GHz	$\varepsilon = 2 \sim 10$, $\tan \delta = 1 \times 10^{-4} \sim 5 \times 10^{-3}$
Transmission line method	Coaxial cable	0.2 ~ 110 GHz	Applicable to high-loss materials, with stringent requirements for sample shape and surface condition.
	Waveguide tube		
	Transmission line		

Table 1.
Two kinds of microwave dielectric performance test methods.

Consequently, based on the measured parameters of S_{11} and S_{12} , Γ_c and T_d are obtained by solving according to Eqs. (4) and (5), and then the dielectric performance parameters of the material under test are derived based on the NRW algorithm.

The merit of the transmission line method lies in its ability to conduct wideband testing (**Table 1**). Nevertheless, as the reflected waves of electromagnetic waves on the front and back surfaces of the sample under test generate certain superimposition, the dielectric performance parameters derived near these points are unreliable. Furthermore, this method has rather high demands on the surface size and morphology of the sample. It is necessary to ensure that the sample is in complete and tight contact with the waveguide wall to prevent electromagnetic wave leakage and minimize measurement errors.

Hence, in the dielectric property testing process of low-dielectric and thermally conductive epoxy resins, an appropriate testing standard needs to be selected in accordance with the frequency range of the transmitted electromagnetic waves. Commonly, for frequencies lower than 2 GHz, the resonator cavity method is adopted for testing; whereas for microwave frequency bands at 2 GHz and above, the transmission line method is typically utilized [22].

2.3.2 Testing method of thermal conductivity property for low-dielectric and high-thermal-conductivity epoxy resins

The measurement approaches for the λ of low-dielectric and high-thermal-conductivity epoxy resins are mainly classified as steady-state measurement methods and non-steady-state measurement methods. The classical steady-state heat flow method utilizes the steady-state heat transfer process to measure the λ value under equilibrium conditions, namely, the heat transfer rate is equivalent to the heat dissipation rate.

2.3.2.1 Steady-state measurement method

The classical steady-state approaches mainly comprise the Guarded Hot Plate Method and the Guarded Heat Flow Meter Method [23].

Guarded Hot Plate Method (Figure 5a): The Guarded Hot Plate Method in compliance with the international standard ISO 8302 serves as the reference technique for the measurement of the λ value, since it does not necessitate calibration with samples of known λ values. Two identical samples can be symmetrically positioned on the main heating plate, or a single sample can be placed on one side of the main heating plate. The main heating plate induces a stable temperature gradient in the samples, while a heater or cooler is located on one side of the sample to regulate its temperature. Measurements can be conducted in different gas environments or vacuum conditions. The temperature gradient of the sample offers the driving force for heat transfer.

Guarded Heat Flow Meter Method (Figure 5b): The Guarded Heat Flow Meter Method is a quasi-steady-state modality and a variation of the Guarded Hot Plate Technique. During the testing procedure, the sample is subjected to a compressive load between two polished metal surfaces. The upper surface functions as a heat source maintaining the test temperature, while the lower surface constitutes a calibrated heat flow transducer. The temperature of the sample is ascertained by temperature sensors on both sides of the metal surfaces of the specimen. λ is computed based on the calibration factor, specimen thickness, and specimen temperature.

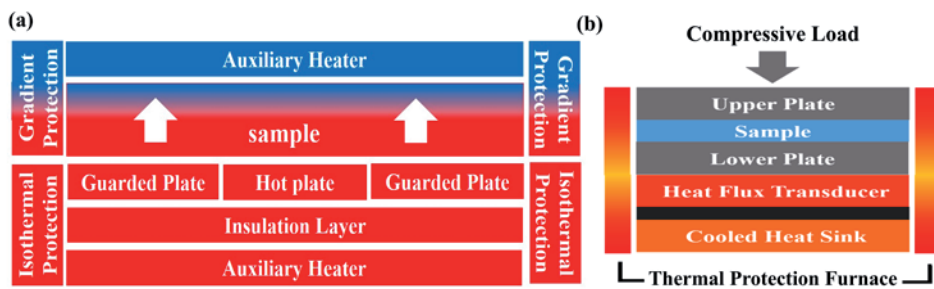


Figure 5. Schematic diagram of testing principle for guarded hot plate method (a) and guarded heat flow meter method (b).

The principle of the steady-state measurement method is distinct and it can directly acquire the λ value. It is applicable to a broad range of temperature measurements. Nevertheless, the steady-state measurement method is frequently time-consuming and demands stringent measurement conditions, such as the adiabatic condition of the measurement system, temperature control during the measurement process, and the requisite parameters, along with the shape and size of the sample.

2.3.2.2 Non-steady-state measurement method

Non-steady-state measurement method has emerged in the recent decades and mainly encompasses the laser method, the transient plane source technique, and the temperature wave analysis method. These methods possess the merits of short measurement time, high accuracy, and low environmental requirements [24].

Laser method (Figure 6a): It is based on measuring the temperature variation on the rear side of a thin disk-shaped sample (this temperature variation is induced by short energy pulses on the front surface). The front surface of the sample is pulsed-heated, and the heat flow conducts one-dimensionally (1D) to the rear side. The temperature rise curve on the rear side is detected by an infrared detector or a contact temperature sensor. A high-speed recorder collects the data representing the temperature variation. The λ is calculated based on the shape of the temperature-time curve (thermal map) and the thickness of the sample.

Hot disk method (Figure 6b): A thin resistance element, configured in a double helix pattern, functions as both the heat source and temperature sensor. This element is positioned between two planar samples of identical material. A constant heating power is applied to the element, causing its resistance to vary over time, which results in changes in the voltage across the element. The temporal variation in resistance can be monitored by measuring the voltage change. The temperature response curve is established based on the relationship between the resistance change and temperature, allowing for the determination of the physical model of the heat transfer process. This method is capable of determining the anisotropic values of λ .

Temperature Wave Analysis Method (Figure 6c): The temperature wave analysis method is a means for measuring the λ in the thickness direction of thin plate specimens. Its principle lies in detecting the phase shift of the temperature wave as it propagates along the thickness direction. This approach utilizes resistive elements to generate and sense the temperature wave, typically covering the opposite sides of the specimen, with one functioning as a heater and the other as a sensor. By measuring

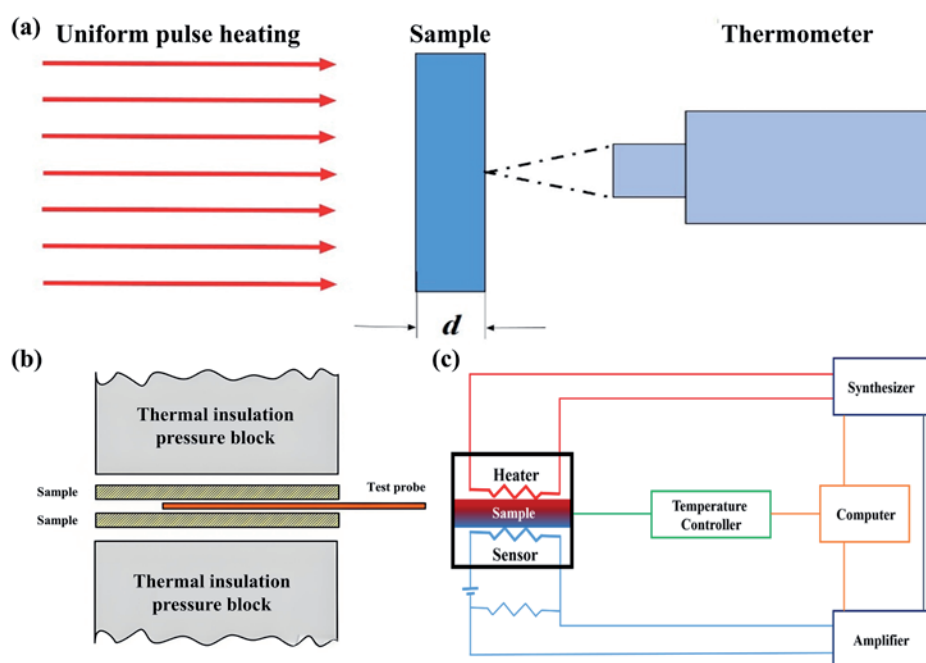


Figure 6.
 Schematic diagram of testing principle for laser flash method (a), hot disk method (b), and temperature wave analysis method (c).

the phase shift of the temperature wave within the specimen and combining it with the angular frequency of the temperature wave and the thickness of the specimen, the λ can be computed.

Currently, the performance testing approaches for low-dielectric-constant and high-thermal-conductivity epoxy resins have not been unified, and the data from different testing methods cannot be directly compared. Hence, in the future, more meticulous research work can still be carried out regarding the unified testing methods for the thermal conductivity of epoxy resins [25].

3. Preparation of low-dielectric and high-thermal-conductivity epoxy resins by blending modification

Physical filling modification implies the addition of ceramic fillers with low-dielectric constant and high-thermal conductivity into traditional epoxy resins for the fabrication of low-dielectric and high-thermal-conductivity epoxy resin composites. This approach is currently the most prevalent preparation method in engineering applications. Commonly used fillers that possess both thermal conductivity and dielectric properties mainly encompass alumina (Al_2O_3), silica (SiO_2), silicon carbide (SiC), boron nitride (BN), etc. Based on the variance of filler types added, they can be categorized into three types: single fillers, compound fillers, and heterogeneous fillers. However, since the dielectric constants of the above-mentioned fillers are all higher than that of the epoxy resin matrix, the dielectric constant of the epoxy resin composites prepared usually increases with the increase of the filler dosage.

3.1 Modification by the single filler

Currently, researchers both domestically and internationally have significantly improved the λ of epoxy resins by introducing a single filler. Ordinarily, the higher the thermal conductivity of filler per se, the better the thermal conductivity performance of the final epoxy resin composite under the same additive dosage [26, 27].

Huang et al. [28] prepared SiC nanowires (SiCnws)/epoxy resin via the physical filling method. When the volume fraction of SiCnws is 3.91%, the thermal conductivity of epoxy resin is 0.43 W/m·K, approximately twice that of pure epoxy resin. Wu et al. [29] employed 4,4'-bis(4-hydroxybenzylidene) diphenyl ether (SBPDAE) as the matrix, cationic initiator (1-butyl-3-methylimidazolium (BMIM)) as the curing agent, and spherical boron nitride (s-BN) as the filler to fabricate s-BN/SBPDAE composites through the blending-casting method. When the volume fraction of s-BN reaches 60%, the thermal conductivity of the composite material amounts to 9.36 W/(m·K), which is approximately 46.8 times higher than that of the pure epoxy resin (0.20 W/(m·K)). Meanwhile, the ε of the above epoxy resins are all within the range of 4–6. It can be discerned from the aforementioned studies that the incorporation of high-thermal-conductivity fillers can establish thermal conduction path, which is conducive to the formation of an efficient thermal conduction network within the resin and conspicuously boosts its thermal conductivity coefficient. Nevertheless, since the dielectric constants of high-thermal-conductivity fillers are commonly higher than that of the pure resin, it leads to an increase in its dielectric constant.

3.2 Modification by the compound filler

However, the enhancement effect of filling and modifying epoxy resin composites with a single thermal conductive filler on the λ is extremely limited, and it is perpetually challenging to break away from the reliance on high dosages and the associated issues such as excessive cost. Studies have demonstrated that incorporating a combination of multiple thermal conductive fillers into the epoxy resin enables the synergistic construction of a more efficient thermal conduction network by integrating their respective advantages, thereby significantly elevating the thermal conductivity. Its performance is conspicuously superior to that of composites modified with a single thermal conductive filler.

Wu et al. [30] employed bisphenol A epoxy resin as the matrix and high-thermal-conductivity aluminum nitride (AlN) and zinc oxide whiskers (ZnOw) as the thermal conductive fillers. The research demonstrated that the epoxy resin thermal conductive composites filled with the AlN/ZnOw hybrid fillers exhibited superior thermal conductivity and mechanical properties compared to those filled with single AlN or ZnOw under the same dosage conditions. Mai et al. [31] mixed two fillers, namely aluminum oxide (Al_2O_3) and boron nitride (BN), and added them to epoxy resin to fabricate BN/ Al_2O_3 /EP composites. The experimental results revealed that the λ of the BN/ Al_2O_3 /EP composites was higher than that of the resins only containing Al_2O_3 . When the volume fractions of Al_2O_3 and BN were 68.82 and 5.3%, respectively, the thermal conductivity of the BN/ Al_2O_3 /EP composites reached 4.44 W/(m·K). By contrast, the compound fillers with different particle sizes can form a denser thermal conduction network (akin to the “sand-stone structure”), which is conducive

to elevating the thermal conductivity of the material at the same filler dosage. Nevertheless, this approach still gives rise to the issue that the dielectric constant ascends with the increase of the filler dosage.

3.3 Modification by the heterogeneous filler

Studies have demonstrated that the heterostructured thermally conductive fillers constructed based on the optimized design of microstructures can give full play to the individual merits of single thermal conductive fillers, realizing a synergistic improvement effect. Through the utilization of their distinctive structural and morphological characteristics, these fillers are more prone to form efficient thermal conduction paths within polymer matrices, and simultaneously effectively prevent the increase in interfacial thermal resistance and the agglomeration phenomenon of the thermal conductive fillers themselves.

Zou et al. [32] fabricated $\text{Al}_2\text{O}_3@\text{BNNS}$ heterostructured fillers ($\text{Al}_2\text{O}_3/\text{BNNS} = 1/7$, wt/wt) by coating boron nitride nanosheet (BNNS) onto the surface of Al_2O_3 , and subsequently blended and compounded to prepare $\text{Al}_2\text{O}_3@\text{BNNS}/\text{epoxy}$ resin. When the volume fraction of $\text{Al}_2\text{O}_3@\text{BNNS}$ is 65%, the λ of the resin attains $2.43 \text{ W}/(\text{m}\cdot\text{K})$, which is conspicuously superior to those of the single $\text{Al}_2\text{O}_3/\text{epoxy}$ resin ($\lambda = 1.39 \text{ W}/(\text{m}\cdot\text{K})$) and the simply blended $\text{Al}_2\text{O}_3\&\text{BNNS}/\text{epoxy}$ resin (1/7, wt/wt, $\lambda = 1.94 \text{ W}/(\text{m}\cdot\text{K})$) with the same dosage (65 vol%), and is significantly higher than that of pure epoxy resin ($\lambda = 0.21 \text{ W}/(\text{m}\cdot\text{K})$).

Vu et al. [33] prepared heterostructured thermally conductive fillers with diverse ratios by employing oxidized carbon nanotubes (oCNTs) and silane coupling agent modified boron nitride (f-BN), encompassing $\text{oCNTs}@f\text{-BN}_{10}$ ($\text{oCNTs}/f\text{-BN}_{10} = 1/10$, wt/wt), $\text{oCNTs}@f\text{-BN}_{20}$ ($\text{oCNTs}/f\text{-BN}_{10} = 1/20$, wt/wt), and $\text{oCNTs}@f\text{-BN}_{40}$ ($\text{oCNTs}/f\text{-BN}_{10} = 1/40$, wt/wt), and further conducted blending and compounding to fabricate $\text{oCNTs}@f\text{-BN}/\text{epoxy}$ resin thermally conductive composites. The experimental outcomes reveal that when the mass fraction of $\text{oCNTs}@f\text{-BN}_{10}$ is 20%, the thermal conductivity of the composite reaches $1.26 \text{ W}/(\text{m}\cdot\text{K})$, approximately six times that of pure epoxy resin ($\lambda = 0.21 \text{ W}/(\text{m}\cdot\text{K})$), and is superior to those of $\text{oCNTs}@f\text{-BN}_{20}/\text{epoxy}$ resin ($\lambda = 0.89 \text{ W}/(\text{m}\cdot\text{K})$), single $f\text{-BN}_{10}/\text{epoxy}$ resin ($\lambda = 0.40 \text{ W}/(\text{m}\cdot\text{K})$), and $\text{oCNTs}@f\text{-BN}_{40}/\text{epoxy}$ resin ($\lambda = 0.67 \text{ W}/(\text{m}\cdot\text{K})$) with the same filler dosage. The aforementioned studies simplify the material preparation process and augment the filler loading through the fabrication of heterogeneous structure fillers.

Among the aforementioned three approaches, the introduction of an excessive amount of thermal conductive fillers will result in the formation of interfacial thermal barriers within the composite material, augmenting phonon scattering and thereby being detrimental to the further enhancement of its λ . Concurrently, due to the abundance of free electrons within common thermal conductive fillers, their ϵ typically exceeds that of the epoxy resin matrix. The introduction of new interfaces after filling will trigger a pronounced interfacial polarization effect, making it arduous to achieve the coordinated improvement of the dielectric and thermal conductive properties of epoxy resin. Furthermore, existing studies indicate that the thermal λ is far more reliant on the thermal conductivity of the matrix than on the thermal conductive fillers per se. Hence, relevant research institutions both domestically and internationally have initiated studies on the design and synthesis of intrinsically low-dielectric and high-thermal-conductivity epoxy resins.

4. Synthesis of intrinsic low-dielectric and high-thermal-conductivity epoxy resins

The fabrication of intrinsic low-dielectric and high thermally conductive epoxy resin is primarily based on the following two research design concepts or approaches.

4.1 Synthesis of liquid crystal epoxy monomers

Commencing from the molecular structure of liquid crystal epoxy monomers, through the selection of the type of liquid crystal group, determination of their positions, optimization of the rigid-flexible structure coordination of liquid crystal epoxy molecules, and introduction of groups with strong intermolecular forces, liquid crystal epoxy monomers are synthesized, and subsequently, intrinsically high thermally conductive liquid crystal epoxy resins are fabricated. This constitutes the current key research direction.

In 2003, Takezawa et al. [34] made the first report on the research of intrinsic thermally conductive liquid crystal epoxy resins. They employed diphenyl benzoate as the mesogenic moiety and aromatic diamine as the curing agent to synthesize three epoxy monomers with varying lengths of flexible segments on the main chain, and systematically investigated the impact of the length of the flexible segments on the thermal conductivity. The experimental outcomes revealed that when the number of carbon atoms in the flexible carbon chain on the main chain was 8, 6, and 4, respectively, the λ of the liquid crystal epoxy resins were 0.85, 0.89, and 0.96 W/(m·K), respectively, approximately five times that of traditional epoxy resins. Meanwhile, the ordered structure of liquid crystals helps to reduce the dipole polarization, thereby lowering the dielectric constant of the liquid crystals.

Furthermore, as the length of the flexible segments shortened, the corresponding thermal conductivity increased. This was because overly long flexible carbon chains were disadvantageous for the regular arrangement of the three-dimensional (3D) epoxy resin network, thereby influencing its λ and ϵ . Consequently, the rational design of the combination of rigid groups and flexible segments is the key to fabricating intrinsically high thermally conductive liquid crystal epoxy resins.

Tokushige et al. [35] synthesized two types of aromatic ester-type liquid crystal epoxy monomers and fabricated liquid crystal epoxy resins through cationic photopolymerization. The two cured resins presented smectic and near-crystalline liquid crystal textures, respectively, with corresponding λ values of 0.31 and 0.43 W/(m·K), respectively. Giang et al. [36] designed and synthesized several methyleneamine-type liquid crystal epoxy monomers and examined the influence of the main chain structure on the properties of epoxy resins. The results demonstrated that the ordered trend of mesogenic units directly affected the thermal conductivity of the material; the higher the structural orderliness, the higher the corresponding λ , with the maximum reaching 0.45 W/(m·K). Meanwhile, the two types of resins also have relatively low ϵ .

In our previous research [37], the novel fluorine-containing liquid crystal epoxy compounds (TFSAEy, **Figure 7**) with fluorinated groups, biphenyl units, and flexible alkyl chains are first synthesized. The obtained ϵ and $\tan\delta$ values of TFSAE at 1 MHz are 2.54 and 0.025, respectively, significantly lower than those of conventional epoxy resins (E-51, 3.52 and 0.038). Additionally, the λ of TFSAEy is 0.36 W/(m·K), 71.4% higher than that of E-51 (0.21 W/(m·K)).

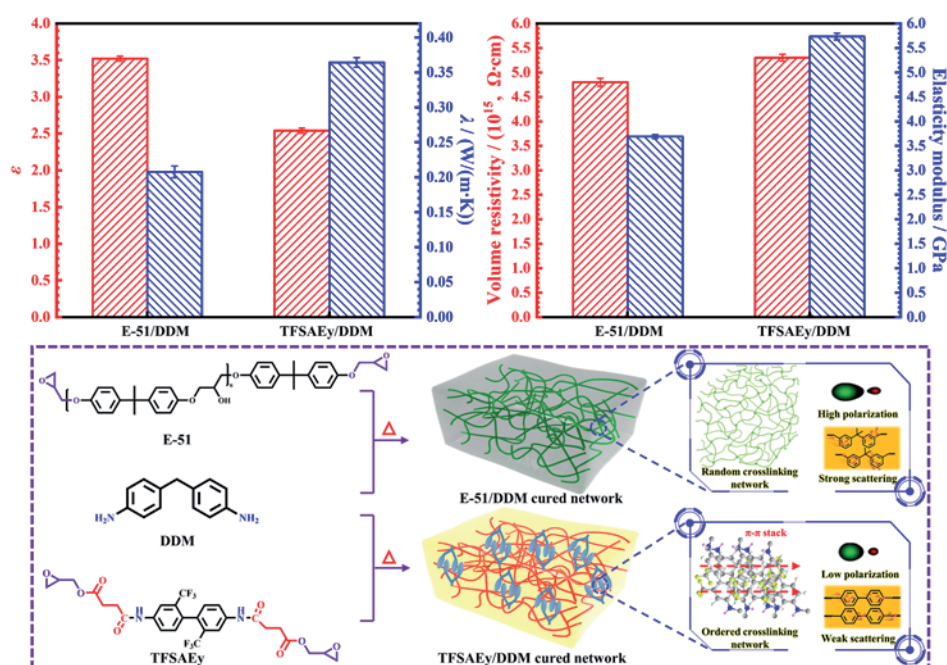


Figure 7. Dielectric and thermal performance and the microscopic molecular curing network structure of TFSAEy resins [37].

Apart from the main chain structure, the side chain structure of epoxy monomers also exerts a considerable influence on the thermal conductivity of epoxy resins. Guo et al. [38] synthesized liquid crystal epoxy monomers (LCEn) featuring side chain substituents and placed emphasis on exploring the impact of side chain length on epoxy resins. The main chain of the liquid crystal epoxy monomers was an aromatic ester mesogenic unit, and the carbon chain lengths (n) of the side chains were 2, 6, and 10, respectively. The λ of the cured liquid crystal epoxy resins were 0.25, 0.29, and 0.26 W/(m·K), respectively.

Additionally, in the molecular structure design of liquid crystal epoxy monomers, the incorporation of strong intermolecular force groups is also encompassed. This is due to the fact that molecular orientation can enhance phonon vibration for effective heat transfer. Hence, through the introduction of certain groups with strong intermolecular forces, liquid crystal epoxy monomers can achieve the ordered arrangement and orientation of mesogenic units while forming a three-dimensional cross-linked network, thereby enhancing the intrinsic thermal conductivity of epoxy resins. Jeong et al. [39] fabricated a cycloaliphatic epoxy compound LCE-CB06 with cyanobiphenyl mesogenic end groups. After curing and cross-linking with diamine, the liquid crystal epoxy resin still retained the oriented liquid crystal region. They ascribed this phenomenon to the partial charge of the cyanobiphenyl mesogenic end groups, which gives rise to dipole-dipole and π - π interactions between adjacent mesogenic units. These interactions serve as weak cross-links, improving the orientation of the liquid crystal (**Figure 8**). Precisely because of this anisotropic molecular orientation in the microstructure, the intrinsic λ of the fabricated liquid crystal epoxy resin can reach 0.46 W/(m·K).

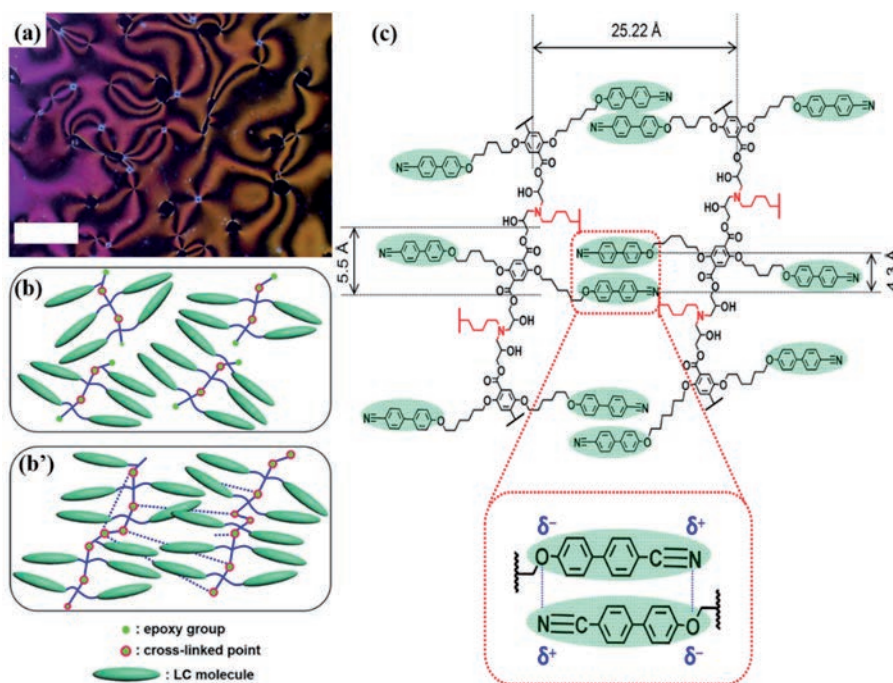


Figure 8.

Polarization optical microscope (POM) photographs of LCE-CBo6 taken at 106°C (a, scale bars indicate 50 μm), schematics describing molecular structures of the mixture after pre-cure (b) and after post-cure (b') [39].

4.2 Optimization of curing agents and regulation of curing networks for epoxy resins

Proceeding from the structure and functional design of curing agents or cross-linking agents, intrinsic thermally conductive liquid crystal epoxy resins can be prepared by introducing liquid crystal groups into the small molecules of curing agents or cross-linking agents; or by promoting the ordered arrangement of epoxy monomers via the interaction forces between curing agents or cross-linking agents, thereby enhancing the thermal conductivity and dielectric performance.

Kim et al. [40] devised and fabricated three types of liquid crystal epoxy monomers based on methyleneimine mesogenic units. The outcomes demonstrated that the thermal conductivity of liquid crystal epoxy resins was intimately associated with the framework structure of the liquid crystal epoxy. The higher the orderliness of the framework structure, the greater the λ and the lower ϵ value of the liquid crystal epoxy resin. Fukushima et al. [41] utilized a liquid crystal epoxy resin as the matrix (the mesogenic units formed a nanoscale high-order structure, and the controllable nanostructured polymer could restrain phonon scattering, enabling the intrinsic λ to reach five times that of ordinary epoxy resins).

Moreover, the higher thermal conductivity and lower dielectric constant were achieved by combining nanofillers with liquid crystal epoxy resins and utilizing the synergistic effect between nanofillers and liquid crystal groups through the introduction and dispersion of nanofillers.

5. Special molding methods of low-dielectric and high-thermal-conductivity epoxy resins

Through the optimization and regulation of the molding mode and curing process, as well as the effect of external conditions (external force, external field, temperature) during the molding process, the molecular chain segments are fully stretched and arranged to form a certain crystalline structure, thereby enhancing the intrinsic thermal conductivity and dielectric performance of epoxy resins.

The action of external fields can influence the orientation and arrangement of molecular chains during the curing process. Laurence et al. [42] investigated the structure and orientation of epoxy resins obtained from the reaction of liquid crystal epoxy monomers and diamines under the influence of a magnetic field. It was found that within a very short reaction time, the orientation degree of molecular chains along the magnetic field axis was extremely high. Harada et al. [43] studied the effect of the magnetic field on the thermal conductivity of epoxy resins during the curing process. Using bis(4-amino-3-methylphenol) terephthalate diglycidyl ether (DGETAM) as the epoxy monomer and 4,4'-diaminodiphenyl ether (DDE) as the curing agent, curing was carried out under different magnetic field intensities. The results indicated that the λ of the curing system under non-magnetic conditions was relatively low, at 0.43 W/(m·K). However, the cured system under a 10 T magnetic field exhibited significant anisotropy in structure and performance, with λ values of 0.32 W/(m·K) perpendicular to the magnetic field and 0.89 W/(m·K) along the magnetic field. This was mainly attributed to the highly oriented and ordered network structure of the epoxy resin along the magnetic field direction, which significantly increased its λ .

Liu et al. [44] fabricated BNNS/epoxy resin composites with high thermal conductivity through an extrusion-thermal pressing molding method. When the BNNS content was 40 wt%, the thermal conductivity of the BNNS/epoxy composite reached 5.86 W/(m·K), which was significantly superior to those of pure epoxy resin curing products (0.21 W/(m·K)) and BNNS/epoxy resin prepared by simple blending molding with the same filler content (3.03 W/(m·K)), and it also possessed good thermal stability (glass transition temperature of 114°C) and mechanical properties (storage modulus of 4324 MPa). Zeng et al. [45] utilized tetramethylbiphenyl bisphenol diglycidyl ether (LCE-BHBTB) containing a biphenyl mesogenic unit as the epoxy monomer, 4,4'-diaminodiphenyl sulfone (DDS) as the curing agent, and triphenylphosphine (TPP) as the accelerator. They achieved a high orientation of the biphenyl liquid crystal units in LCE-BHBTB via electrospinning and then cured it within the liquid crystal temperature range to prepare LCE-BHBTB/DDS resin curing product films (**Figure 9**). The results demonstrated that the LCE-BHBTB/DDS resin curing product films had the lowest ϵ (2.5) and an ultra-high λ (0.8 W/(m·K)), both of which were lower than the 3.2 and 0.25 W/(m·K) of diglycidyl ether of bisphenol A (DGEBA)/DDS resin curing products, and its λ could be adjusted by the fiber diameter. This material holds broad application prospects in the fields of flexible electronics and smart wearable devices in the future. The aforementioned studies demonstrate that via special processing approaches, the order degree of the epoxy resin curing network can be elevated, and it is also beneficial for the improvement of the dielectric and thermal conductivity properties of the resin.

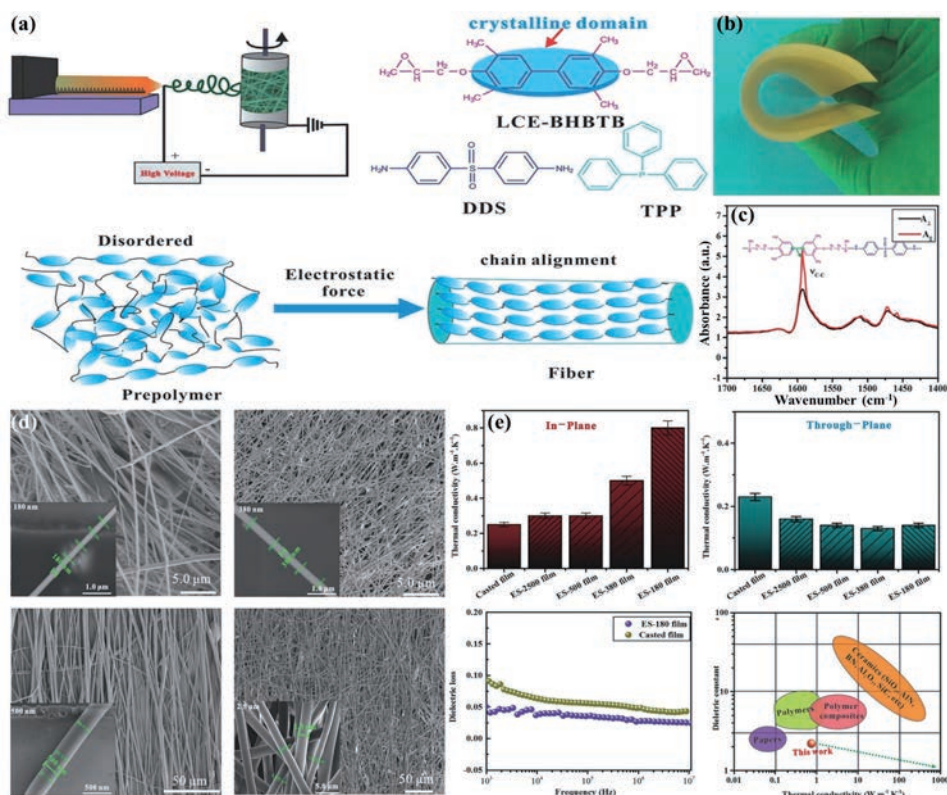


Figure 9.

Schematic diagram of preparation (a), digital photograph (b), and normalized Fourier transform infrared (FT-IR) spectroscopy (fiber diameter for 180 nm, c) of the LCE-BHBTB/DDS resin films, scanning electron microscopy (SEM) images of the LCE-BHBTB/DDS resin films with the different fiber diameters (d), and the testing results of λ , ϵ , and $\tan \delta$ (e) [45].

6. Application of low-dielectric and high-thermal-conductivity epoxy resins

Low-dielectric and high-thermal-conductivity epoxy resins, as high-performance resins, have manifested significant application values in domains, such as electronics, communications, automobiles, and aerospace [46].

Low-dielectric and high- thermal-conductivity epoxy resins are mainly used as packaging materials for electronic chips and as substrates for printed circuit boards (PCBs).

As a key component of integrated circuits, low-dielectric and high-thermal-conductivity epoxy resins play a crucial role in supporting the interconnection of electronic components and ensuring their good connection and data transmission. This requires PCBs to have low ϵ and $\tan \delta$ values to achieve high characteristic impedance and reduce interference and crosstalk in the signal transmission process. In the field of high-frequency communications, low-dielectric epoxy resins (such as those with fluorine-containing curing agent systems) achieve a dielectric constant < 3.0 and a loss factor < 0.005 by reducing dipole polarization [47].

Furthermore, with the advent of the 5G/6G communication era, the signal transmission speed on PCBs has significantly increased. Therefore, in addition to ensuring

their low-dielectric constant and loss, it is also required that they have good thermal conductivity to promptly disperse the heat generated by electronic components. Through optimizing the addition ratio of functional fillers or structural design of epoxy resin, the low-dielectric and high-thermal-conductivity epoxy resins have prominently enhanced the thermal conductivity (reaching up to 0.6 ~ 3.39 W/m·K), while concurrently reducing the dielectric loss (for example, the dielectric constant of biphenyl-type liquid crystal epoxy can be as low as 2.5). This effectively resolves the contradiction between chip heat dissipation and signal transmission stability, rendering them suitable for high-density integration scenarios, such as high-frequency integrated circuits and 5G communication equipment [48].

With the rapid development of high-throughput/high-frequency communication technologies, the future information electronics field will have even higher demands for low-dielectric and high-thermal-conductivity epoxy resins. For instance, low-dielectric and high-thermal-conductivity epoxy resins for the superconducting motor field need to simultaneously possess ultra-low dielectric loss, low-temperature resistance, and high insulation performance requirements. Therefore, the continuous attention of worldwide researchers in the epoxy resin field is still needed.

7. Conclusions

This chapter comprehensively reviews the microscopic mechanisms, performance testing methods, and application scenarios of low-dielectric-constant and high-thermal-conductivity epoxy resins, summarizing the latest research advancements in this field. Traditional epoxy resins generally exhibit low thermal conductivity and high dielectric constants, which limit their suitability for high-performance applications in the electronic information domain. By conducting an in-depth analysis of the key factors influencing the dielectric and thermal properties of epoxy resins, researchers can develop targeted strategies to design and synthesize novel epoxy resins with improved low dielectric constants and high thermal conductivities. This represents a critical direction for future advancements in epoxy resin technology.

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Conflict of interest

The authors declare no conflict of interest.

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Epoxy-Based Nanodielectrics for High Voltage Insulation Systems

Mihir Velani

Abstract

Over the years, polymeric insulators have emerged with excellent dielectric and other physical properties with the help of increased familiarity with chemistry and physics and improved manufacturing technology. The applications of epoxy nanodielectrics in high-voltage insulation and electronics have increased greatly in recent decades. In the energy industry, various polymer materials, including thermoplastics, thermosets, glass, and natural fibers, have been employed because of their superior performance and long lifespan in extreme environments. Epoxy resins containing appropriate nano and micro-organic particles have gained popularity for practical applications in various electrical power apparatuses and systems. By incorporating nanofiller into epoxy resin matrices, these materials exhibit significant improvements in dielectric strength, thermal conductivity, partial discharge resistance, and long-term reliability compared to conventional epoxy systems. This chapter presents an inclusive overview of epoxy nanodielectrics, addressing material design, nanofiller selection, interfacial engineering, and synthesis methods essential for achieving uniform dispersion and optimized-blended properties. Key electrical, thermal, and mechanical characteristics are discussed for high-voltage apparatuses such as transformer bushings, current and voltage transformers, switchgear, and cast resin transformers. Additionally, the chapter addresses challenges related to the synthesis processes and scalability of epoxy nanocomposites, along with future trends toward multifunctional and environmentally sustainable insulating materials. The applicable test standards and material characterization methods are highlighted to provide comprehensive understanding of performance evaluation of epoxy nanodielectrics in HV systems.

Keywords: dielectric spectroscopy, glass transition temperature, partial discharge resistance, polymer-filler interface, power apparatus applications, scaling-up challenges, surface functionalization

1. Introduction

Solid insulating materials are indispensable part of electrical power systems. For a long time, porcelain (ceramic) has been popular for its superior dielectric properties and robustness. As the power transmission voltages increase, the requirement of lightweight new insulations with multifunctional properties has led to significant growth in polymeric materials. Epoxy has emerged as an innovative product, offering

excellent dielectric, thermal, and mechanical properties due to its unique processability and compatibility with a range of inorganic micro- and nanofillers [1–3]. Over the past two decades, the addition of nanofillers into epoxy has transformed epoxy resins into advanced nanodielectrics that claim excellent dielectric strength, improved thermal conductivity, and high partial discharge (PD) resistance and weatherproof.

This chapter presents a typical review of epoxy-based nanodielectrics, probing key aspects including selection and synthesis of the material, interfacial engineering, conception of material properties, characterization methods, applications, and the recent developments in the stirring field.

2. Polymeric insulators: Background and evolution

2.1 Early insulating materials

Over more than four decades, porcelain insulators were dominating solid insulation systems in transmission lines and power apparatuses owing to its chemical inertness and withstand ability to perform in harsh environmental conditions. Conversely, their heavy weight, brittleness, and manufacturing complexity made them restricted to large and complex power equipment. Gradually, the industry moved to glass insulators offering transparency in fault detection and lower production erraticism. Yet, glass suffered from mechanical fragility and was not well-suited for high-voltage station equipment. In parallel, mica and cotton-based materials were adopted for capacitors and cables, often impregnated with oil to improve dielectric strength. These were effective in the short term but prone to moisture ingress and thermal degradation, leading to premature failure [4, 5].

By the mid-twentieth century, oil-impregnated paper (OIP) systems became standard for transformer bushings and cables. While OIP systems provided high dielectric strength, their susceptibility to moisture and the need for maintenance triggered off research into more robust alternatives.

2.2 Polymeric revolution

The statistics reveal a sharp rise in electricity demand, which particularly impacts the power system in terms of operating voltage levels, overall size, and system intricacy. While higher voltage levels have enhanced the robustness and cost-effectiveness of power networks, solid insulators continue to confront challenges related to weight, cost, size constraints, and the combined influence of electric fields and thermal stresses. Despite these limitations, solid insulators remain integral components of electrical equipment, providing both insulation and mechanical support. However, they are constantly subjected to over voltages as well as extreme environmental and pollution conditions, which can compromise their performance [6–9]. To address these challenges, it is crucial to devise next-generation insulators that offer enhanced electrical and thermal properties. Material innovation has evolved as existing technologies met barriers and material science progressed.

The progress of electrical insulation materials has always paralleled advances in electrical equipment. In the past, devices such as transformers, cables, and capacitors relied on mica, cotton threads or fabrics, and oil-impregnated paper for insulation. Over time, non-ceramic or polymeric insulators emerged, with the first such materials being used in 1963 [10]. Each phase of material innovation was compelled by



Figure 1.
Timeline of solid insulator development.

constraints in the existing technology, in tandem with advancements in materials science and manufacturing. The insulators were particularly introduced to overcome the limitations of ceramic types in specific environments. Due to ongoing research and development, it is now feasible to devise polymeric insulators for specific applications, delivering enhanced reliability and performance. The introduction of synthetic polymers represented a paradigm shift. Thermoplastics such as polyethylene and polyvinyl chloride (PVC) offered lightweight alternatives but were constrained by thermal stability. The breakthrough came with thermosetting polymers, particularly epoxy resins. Epoxies demonstrated excellent moldability, dimensional stability, and dielectric performance. Their cross-linked structure enabled them to withstand high electrical and thermal stress. Industrial adoption accelerated in the 1960s and 1970s, with epoxy systems replacing ceramics with medium- and high-voltage switchgear, instrument transformers, and cast-resin transformers [11, 12]. **Figure 1** depicts a timeline with approximate periods during which different solid insulators gained popularity in electrical power systems.

The ability to engineer epoxy formulations to specific requirements—by varying curing agents, modifiers, and fillers—offered unprecedented flexibility. This “polymeric revolution” not only condensed reliance on ceramics but also allowed compact equipment designs, reduced installation weight, and enhanced manufacturing scalability.

2.3 Nanotechnology revolution

Nanodielectrics revolutionized the power sector in the twenty-first century. Unlike traditional micro-fillers, nanofillers influence the material at the interfacial level, creating new pathways for charge trapping, dielectric relaxation, and thermal conduction [13]. Epoxy nanodielectrics demonstrated noticeable improvements in dielectric breakdown strength, partial discharge resistance, and thermal conductivity compared to conventional systems [14]. Carbon nanotubes, graphene, silica nanoparticles, and boron nitride nanosheets became subjects of extensive research. Today, epoxy nanodielectrics are being evaluated for a wide range of power equipment, from transformer bushings and gas-insulated switchgear to cast-resin transformers. Their multifunctionality, combining electrical and thermal enhancements, positions them as a base material for the next generation of high-voltage technology.

3. Fundamentals of epoxy nanodielectrics

3.1 Epoxy resin matrix

Thermosetting epoxy resins are formed by the reaction of epoxide groups with curing agents, for instance, anhydrides or amines. The resulting cross-linked network imparts mechanical rigidity, thermal resistance, and dielectric stability. Two common industrial grades are bisphenol-A diglycidyl ether (DGEBA) and bisphenol-F

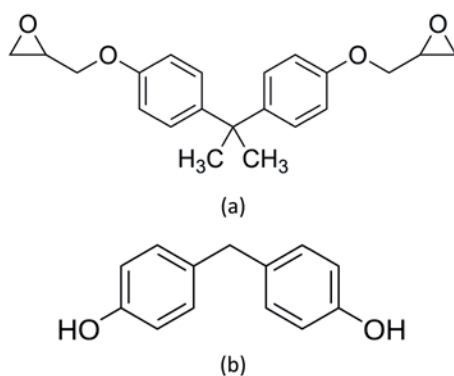


Figure 2.
Chemical structure of (a) bisphenol-A diglycidyl ether (DGEBA) (b) bisphenol-F.

epoxies [15]; the chemical structures are illustrated in **Figure 2**. The key properties of epoxy matrices include the following:

- High dielectric strength and high electrical resistivity
- Low loss factor
- High treeing-tracking resistance
- Excellent chemical resistance and moisture resistance
- Tunable mechanical and thermal properties through curing chemistry
- Strong adhesion to fibrous and metallic reinforcements.

3.2 Nanofillers and nanodielectrics

A class of hybrid organic-inorganic materials is known as nanodielectrics and also termed as nanocomposites. They contain a trace amount of inorganic nanofillers homogeneously dispersed in polymers with at least one dimension in the 1–100 nm range [2]. **Figure 3** provides a schematic representation; the polymer-based nanodielectrics are intended for illustrative purposes only. Nanofillers are introduced to overcome the limitations of neat epoxy resins. Due to their small size (1–100 nm) and high surface area, polymer-based nanocomposites form large and strong interfacial regions with the polymer matrix. As a result, they are increasingly used in electrical machines, solid-insulated switchgear, HV cables, dense electronic assemblies, energy

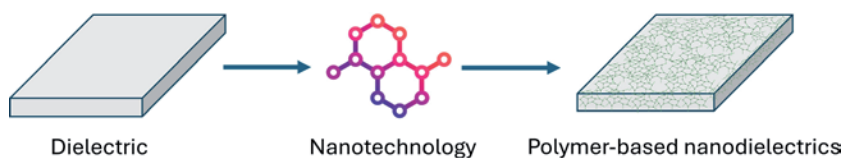


Figure 3.
Schematic representation of nanodielectric formation via nanoparticle dispersion.

Nanofiller type	Materials	Property improvement
Oxides	SiO ₂ , Al ₂ O ₃ , TiO ₂	Dielectric strength, PD (partial discharge) resistance
Nitrides	AlN (aluminum nitride), BN	High thermal conductivity
Carbon-based	CNTs, Graphene	Mechanical reinforcement, conductivity control
Clays	Montmorillonite	Barrier to moisture, mechanical strength

Table 1.
Common nanofillers used in epoxy insulation.

generation, or storage systems [3, 13, 16, 17]. These materials are becoming popular in smart applications for energy, healthcare, and photonic and organic electronic devices [18].

Epoxy nanodielectrics have grabbed attention for HV indoor and outdoor applications due to their affordable and structural versatility. This adaptability stems from the integration of particulate nano- and micro-scale fillers such as alumina, alumina (Al₂O₃), boron nitride (BN), magnesium oxide (MgO), nano clay, silica (SiO₂), titania (TiO₂), zinc oxide (ZnO), graphene [19], graphene dioxide [20], and many others. Moreover, carbon nanotubes (CNTs), graphene and nitride nanosheets, have become the subject of extensive research. Commonly used nanofillers in epoxy insulation are summarized in **Table 1**.

Integrating nanofillers into epoxy resin considerably improves key functional properties, including dielectric permittivity, dielectric breakdown strength, treeing resistance, tracking and erosion resistance, glass transition temperature (T_g), thermal conductivity, and elastic modulus [13]. Furthermore, there is still debate on the interfacial dynamics, which are affected by a variety of factors. Notwithstanding substantial progressions in the field, two persistent technological challenges continue to impede progress: (1) Ensuring homogeneous dispersion of fillers within the host matrix, and (2) Optimizing both the selection and surface functionalization of nanofillers to enhance compatibility and performance [21]. The uniform dispersion is dependent on the intrinsic properties of the base material, type, size, shape and properties of nanofillers, the dispersion method, and the used solvent. Both the synthesis process and the surface condition of the nano fillers give positive results. This makes it necessary to consider a range of factors to investigate the performance of the nanodielectrics. The materials are evaluated and characterized with the help of sophisticated instruments and methods.

4. Synthesis techniques

The synthesis process significantly affects the performance of epoxy nanodielectrics. Additionally, nanoparticles' size and shape, surface treatment and chemistry, dispersion in the epoxy matrix, and processing environment need precise control [22–24]. These ensure good interfacial regions between epoxy resin and nanoparticles.

4.1 Material selection

To begin, the synthesis process requires the selection of a base resin, solvent and curing agent, and nanofiller system.

- a. *Epoxy resins*: DGEBA is the most often utilized epoxy type due to its dielectric stability, good mechanical strength, and ease of processing. Cycloaliphatic epoxies are recommended when higher thermal resistance, chemical stability, toughness, and low viscosity are required [25]. In contrast, Novolac, is the precursor of Bakelite, which is a hard cross-linked thermosetting polymer [26].
- b. *Curing agents*: Amine, anhydride, and acid-based hardeners regulate crosslink density and thermal behavior. Optimized curing periods are important for effective mixing with nanofillers [27].
- c. *Nanofillers*: Common fillers include Al_2O_3 , BaTiO_2 (barium titanate), BeO (Beryllium Oxide), BN, Nano Clay, SiO_2 , TiO_2 , and ZnO [19, 20, 28]. Recently, carbon-based allotropes such as CNTs and graphene are progressively employed for multifunctional properties [29].

4.2 Surface functionalization and interfacial engineering

Direct incorporation of nanoparticles into epoxy often results in agglomeration due to high surface energy and poor compatibility with the polymer matrix. Therefore, a special treatment called surface functionalization is required for improved interaction between base material and nanofillers. Surface functionalization is the process of adding specific chemical molecules to a material's surface to change its properties, while interfacial engineering is a broader approach that optimizes the boundary between varied materials.

- a. *Sol-gel method*: In the sol-gel process for epoxy-nanodielectrics, inorganic nanofillers undergo hydrolysis and condensation, followed by surface modification for compatibility. These are uniformly dispersed into the epoxy matrix and cured, yielding composites with enhanced dielectric and thermal properties [30, 31].
- b. *Silane coupling agents*: Organosilanes are commonly applied to increase chemical bonding between inorganic fillers and the organic epoxy resin. Silane-treated ZnO nanoparticles, for example, demonstrate improved wettability and stable filler dispersion [32].
- c. *Polymer grafting*: In certain cases, nanoparticles are grafted with polymer chains to achieve enhanced miscibility and minimize phase separation [33].
- d. *Plasma and chemical treatments*: Surface activation through plasma oxidation or acid treatment is used for CNTs and graphene, introducing oxygen-containing groups that facilitate bonding with the epoxy matrix [34].

By stabilizing dispersion and controlling interfacial polarization, progressive interface design significantly improves dielectric and thermal properties.

4.3 Dispersion techniques

Achieving uniform dispersion of nanofillers within the base resin is the most crucial step in the synthesis of nanodielectrics. Several techniques are used, often in combination, to avoid agglomeration of nanoparticles [35].

- a. *Mechanical stirring*: This is the initial phase where nanoparticles are incorporated into the epoxy resin using high shear mixers. However, it sometimes results in poor uniformity with high filler concentrations.
- b. *Ball milling*: Ball milling offers a uniform dispersion of nanofillers for certain strong filler system, but it can lead to epoxy resin contamination if not accurately monitored.
- c. *Ultrasonication*: This method employs high-frequency ultrasonic waves to ensure uniform dispersion of nanofillers in epoxy resin. The ultrasonic probe sonication (usually at 20 kHz, 750 W) proves to be more effective than bath sonication (typically at 40 kHz, 180 W). It is important to control the sonication duration as prolonged sonication produces excessive heat that could degrade the resin's molecular structure.
- d. *Solution casting*: The epoxy is first stirred with a solvent (ethanol/methanol/acetone). Then, the nanoparticles are uniformly dispersed in the mixture of epoxy and solvent. This mixture is heated on a hot plate to evaporate the solvent. However, this process occasionally leaves behind the residues that may compromise the dielectric purity if not closely monitored.
- e. *In-situ polymerization*: It is a new method that involves dispersing nanoparticles in monomers prior to polymerization. This ensures enhanced interactions between the base matrix and fillers, resulting in better interfacial bonding [36].

4.4 Mixing and degassing

To avoid particle agglomeration, the epoxy and nanofillers must be homogeneously mixed with solvent and curing agents. Afterwards, vacuum degassing is typically performed to extract the trapped gases and air bubbles which could form voids and result in premature dielectric/thermal failure.

4.5 Curing process

The cross-linking density, mechanical integrity, and thermal stability of epoxy nanocomposites are determined by the curing process. Two major stages should be considered:

1. *Conventional thermal curing*: This process involves controlled heating cycles (e.g., room-temperature pre-curing followed by post-curing at 100–120°C) to allow for gradual cross-linking between epoxy resin and nanofillers and stress relaxation. Here, the curing temperature must be less than the glass transition temperature of the epoxy.
2. *Ultraviolet (UV) and electron-beam curing*: New approaches that support rapid polymerization and precise control are becoming more common. These strategies are especially preferred for thin-film coatings, but they have been remained less explored for bulk insulation applications [37].

It is essential to regulate curing contraction carefully, as improper treatment may result in microcracks and voids developing around interfacial regions between nanoparticles and epoxy resins.

4.6 Scalability challenges

Although laboratory-scale synthesis has demonstrated continuous improvements in material performance, transitioning to industrial-scale production presents significant challenges.

1. *Nanoparticle agglomeration at high volumes:* Industrial mixers usually struggle to achieve uniform dispersion of nanoparticles when working at kilogram to ton scales.
2. *Material synthesis duration and cost:* The extraction of high-purity nanoparticles from complex chemical processes and the use of advanced dispersion methods can extremely increase the overall cost of manufacturing nanodielectrics.
3. *Standardization issues:* There is no universally accepted processing norms for nano filled epoxy systems. This lack of standardization leads to variability in commercial products.
4. *Health and environmental safety:* The handling of nanofillers, curing agents, and epoxy resins requires stringent safety protocols to minimize the risks of inhalation and contamination.

The synthesis and processing of epoxy nanodielectrics require precise control over every stage, from material selection to dispersion and curing. While considerable progress has been made in laboratory-scale procedures, bridging the gap toward scalable, reproducible, and environmentally sustainable processing remains a continuing challenge. The research in interfacial dynamics and industrial-scale dispersion will be central to the future commercialization of these materials.

5. Properties of nanodielectrics

Epoxy-based nanocomposites have attracted overall attention in high-voltage insulation due to their unique ability to blend the promising aspects of polymer matrices with the functional merits of nanofillers. The interaction between polymer chains and nanofillers governs the electrical, thermal, and mechanical properties of these composites [13]. This section delivers a detailed study of the major properties relevant to the performance of epoxy nanodielectrics.

5.1 Electrical properties

The electrical performance of epoxy nanodielectrics is vital for high-voltage systems. The main properties—dielectric constant, dielectric loss, dielectric strength, and resistance to partial discharges—are significantly affected by the type and dispersion of nanofillers.

5.1.1 Dielectric constant and dielectric loss factor

The incorporation of nanofillers changes the dielectric permittivity of the epoxy matrix by introducing additional polarization mechanisms at the filler-matrix interfaces. The polarization mechanism is introduced at varied frequencies and

different temperatures. The inorganic nanofillers often enhance interfacial polarization due to their high surface area, allowing greater charge storage and localized field distribution [38]. At low filler concentrations (<5 wt%), a subtle increase in dielectric constant is typically observed without significantly increasing dielectric loss. However, excessive filler loadings may introduce percolation that elevates dielectric losses [39]. Frequency-dependent dielectric spectroscopy under varying relative humidity (%RH) and temperature conditions helps a thorough analysis of real ϵ' and imaginary ϵ'' permittivity (**Figure 4**), explaining dielectric polarization, relaxation process, and conduction loss mechanisms in dielectric materials [40].

5.1.2 Dielectric breakdown strength

Epoxy nanodielectrics have shown increased dielectric breakdown strength because of reduced space charge buildup and electron scattering within their complex microstructure. The surface treated TiO_2 - SiO_2 core-shell structure in epoxy has achieved more superior breakdown resistance compared to neat epoxy (refer to **Figure 5**) [22]. It is also true that further increase in the filler loading attracts particle agglomeration leading to the reduced breakdown strength.

5.1.3 Partial discharge and surface tracking resistance

Partial discharge resistance is important for longevity of solid insulation. It has been observed that the nanofillers increase PD resistance by filling micro-void growth and alleviating the epoxy resin-filler interface under high voltage electric stress. Epoxy nanodielectrics exhibit superior resistance to surface treeing-tracking and erosion as compared to neat epoxy under high-humidity or contaminated conditions [41–44].

5.2 Thermal properties

Since the localized overheating often speeds up the degradation of insulation, thermal functioning is also a critical parameter in determining the fitness of the epoxy nanodielectrics for use in electrical power applications.

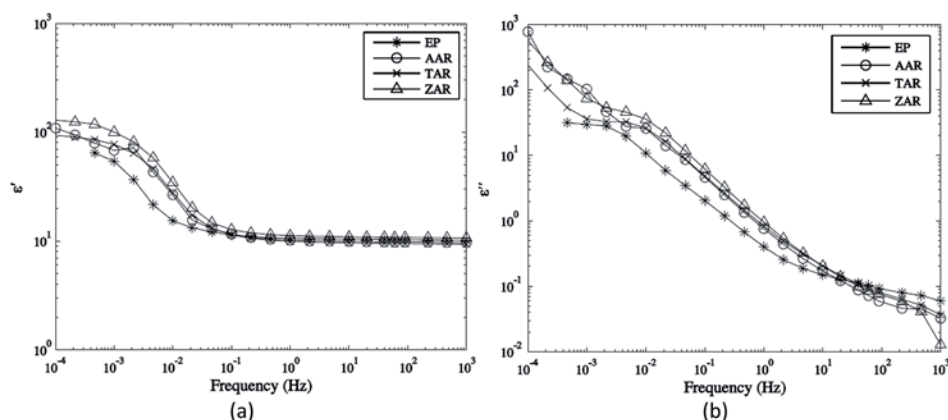


Figure 4. Frequency dependent (a) ϵ' (b) ϵ'' of neat epoxy (EP) and nanocomposites (AAR-alumina as-received, TAR-titanania as-received, ZAR-zinc oxide as-received) with 1% volume fraction at 25% RH and 90°C [40].

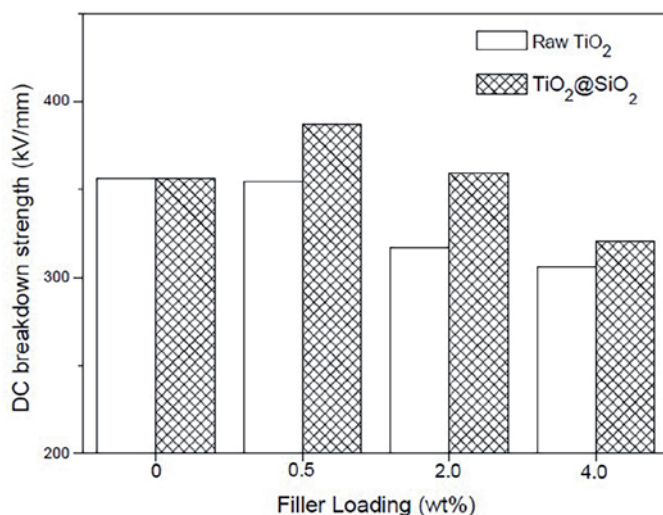


Figure 5.
DC breakdown strength for epoxy filled with raw TiO_2 and $\text{TiO}_2\text{-SiO}_2$ core-shell nanoparticle [22].

5.2.1 Thermal conductivity

Neat epoxy resins show relatively low thermal conductivity ($\sim 0.2 \text{ W/mK}$). The addition of high-thermal-conductivity nanofillers such as BN, ZnO, or graphene derivatives can substantially enhance thermal conductivity. Literature works indicate that epoxy/BN and epoxy/ZnO nanocomposites offer 2-3 times higher thermal conductivity with only moderate filler content [1]. This enhancement reduces hotspot formation and improves thermal management in high-power apparatuses.

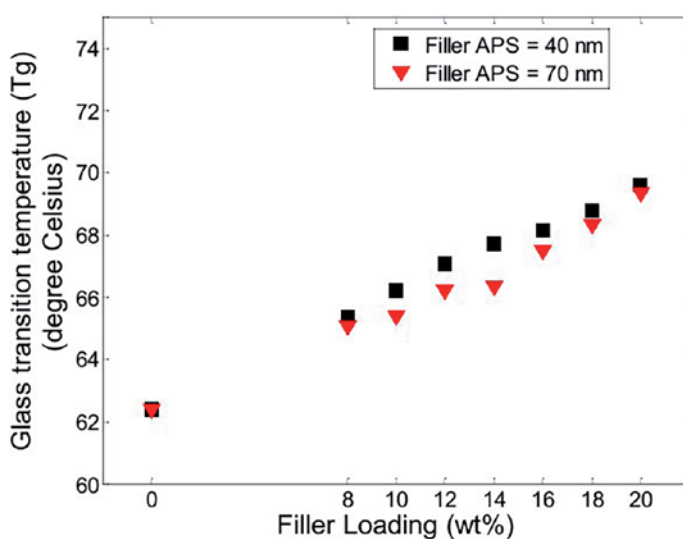


Figure 6.
Glass transition temperature of alumina filled epoxy nanodielectrics [45].

5.2.2 Glass transition temperature (T_g)

The introduction of nanofillers impacts the glass transition temperature by either restricting polymer chain mobility, relying on filler–matrix interactions. Strong interfacial bonding, usually achieved through surface functionalization and silane coupling agents, generally increases T_g by restricting chain relaxation dynamics. A higher T_g ensures thermal stability under elevated temperatures (refer to **Figure 6**) [45].

Thus, epoxy nanodielectrics blend higher dielectric strength with improved thermal conductivity, enabling reliable performance in high-voltage insulation systems.

5.3 Mechanical properties

Mechanical robustness is indispensable for insulation systems subjected to dynamic stresses such as vibration, cantilever loading, and installation-induced stresses.

5.3.1 Tensile and flexural strength

The incorporation of nanofillers, particularly SiO_2 and clay, enhances load transfer efficiency, resulting in improved tensile and flexural strength [46].

5.3.2 Fracture toughness

Nanoparticles reinforce the toughness through mechanisms such as crack path deflection, interfacial energy dissipation, and particle pull-out, collectively delaying crack propagation [47].

5.3.3 Fatigue and creep resistance

Epoxy nanocomposites exhibit excellent resistance to fatigue and creep, thereby assuring long-term mechanical stability in high-voltage insulation applications.

6. Applications in high voltage apparatus

Epoxy nanodielectrics have found growing applications in modern high-voltage technology, where superior dielectric strength, thermal stability, and mechanical robustness are required. A few plausible applications are summarized here [48].

- a. *Transformer bushings*: Epoxy nanodielectrics as oil-end insulator can offer compact, moisture-resistant alternatives to OIP bushings. They withstand impulse stresses and exhibit superior reliability under environmental exposure.
- b. *Instrument transformers*: Nanodielectrics resist partial discharge activity, improving their metering accuracy and long-term stability.
- c. *Switchgear and gas-insulated substation (GIS)*: In compact HV switchgear, epoxy nanocomposites provide high dielectric strength and arc resistance. They also support the move toward SF_6 -free insulation systems.
- d. *Cast-resin transformers*: Nanodielectrics enhance winding insulation, offering better flame retardancy and thermal conductivity compared to neat epoxy.

7. Testing standards and characterization

The qualification of epoxy nanodielectrics depends on standardized evaluations such as IEC/IEEE norms for dielectric strength, permittivity, and tracking resistance, alongside ASTM/ISO methods for thermal and mechanical performance. On the other side, advanced techniques, including dielectric spectroscopy, scanning electron microscopy (SEM), atomic force microscopy (AFM), and transmission electron microscopy (TEM), facilitate accurate insights into surface morphology, filler dispersion, interfacial polarization, and structural integrity.

7.1 Electrical testing standards

Electrical properties form the foundation of insulation performance. The most widely adopted standards are mentioned in **Table 2**.

The partial discharge testing (IEC 60270) and frequency-domain dielectric spectroscopy are the two complementary methods that allow precise monitoring of charge accumulation, interfacial polarization, and relaxation dynamics under varying frequencies and voltages.

7.2 Thermal characterization

The ability of epoxy nanodielectrics to withstand thermal stress is evaluated through established practices:

- a. *Differential scanning calorimetry (DSC) (as per ASTM E1356)* – to determine glass transition temperature (T_g).
- b. *Thermogravimetric analysis (TGA) (as per ASTM E1131)* – to evaluate thermal stability and decomposition temperature.
- c. *Laser flash analysis (as per ASTM E1461)* – for thermal conductivity measurement.

7.3 Mechanical testing

Mechanical integrity is critical for components subjected to cantilever forces, vibrations, and installation stresses. The typical standards include the following:

- a. *ASTM D638* – for the measurement of tensile strength and elongation.
- b. *ASTM D790* – for the determination of flexural strength.

Standards	Purpose
IEC 60243	For measuring dielectric breakdown strength of solid insulation materials.
IEC 60250	For relative permittivity and dielectric dissipation factor.
IEC 60587	For evaluating tracking and erosion resistance under surface stress conditions.
IEC 60112	For the estimation of the comparative tracking indications of solid insulating materials.

Table 2.
IEC standards for electrical testing of solid polymeric insulators.

- c. *ASTM D5045* – for the estimation of fracture toughness.
- d. *ISO 899* – for the assessment of creep and stress relaxation behavior.

These assessments validate the mechanical durability of epoxy nanocomposites, exclusively in applications involving external exposure or mechanical loading.

7.4 Sophisticated diagnostic tools

While standard bulk assessments remain foundational, in context to surface and molecule interaction and dielectric properties, advanced characterization methodologies, facilitate a nuanced (micro- to nanoscale) understanding of epoxy nanodielectrics at finer spatial resolutions:

- a. *Scanning electron microscopy (SEM)* and *transmission electron microscopy (TEM)* – to verify nanofiller dispersion and detect agglomeration.
- b. *Atomic force microscopy (AFM)* – for investigating nanoscale surface topography and mechanical modulus mapping.
- c. *X-ray diffraction (XRD)* – for confirming crystalline structures or intercalation in layered fillers such as clays.
- d. *Fourier transform infrared spectroscopy (FTIR)* – for detecting chemical bonding and surface functionalization effects.
- e. *Dielectric spectroscopy* – to explore interfacial polarization and relaxation processes across a wide frequency range (e.g., 10 μ Hz to 10 kHz) and at different temperatures.

8. Challenges and future perspectives

Despite their promise, epoxy nanodielectrics encounter limitations in large-scale industrial applications. The key challenges include the following:

- a. Achieving uniform nanofiller dispersion at low cost.
- b. Establishing standardized testing protocols tailored to nanocomposites.
- c. Balancing multifunctionality with manufacturability.
- d. Ensuring environmental sustainability by adopting bio-based epoxies and recyclable fillers.

The ongoing research focuses on multifunctional nanodielectrics (carbon allotropes- carbon nanotubes and graphene) that combine electrical, thermal, and mechanical improvements, while also incorporating environmental considerations. In addition, hybrid filler systems comprising 2D and 3D nanostructures may yield

synergistic effects. The inclusion of artificial intelligence and computational modeling is also anticipated to play an increasing role in the design and optimization of such materials.

9. Conclusions

Epoxy nanodielectrics depict a transformative advancement in electrical insulation, offering superior dielectric, thermal, and mechanical properties. Their potential to enable compact, reliable, and sustainable power systems is deducible from emerging research. Yet, the challenges in scalable processing, environmental compatibility, and standardized evaluation persist. Future measures will likely focus on multifunctional formulations, bio-based matrices, and recyclable fillers, guided increasingly by artificial intelligence and computational modeling. As the energy sector evolves, epoxy nanocomposites are poised to become foundational materials in next-generation high-voltage technology.

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Conflict of interest

The authors declare no conflict of interest.

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Synergistic Effect of Flame Retardant Inhibitors and Bamboo Fiber Reinforcement in Epoxy-Based Composites

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Abstract

This chapter investigates the synergistic effect of flame retardant (FR) inhibitors on bamboo fibers used as reinforcement in epoxy-based composites. The chapter explores how chemical treatment of bamboo fibers with various FR agents enhances the fire resistance of the resulting composites while maintaining or improving their mechanical properties. Special focus is given to the modification of fiber surfaces using borax, ammonium polyphosphate, and other FR inhibitors to reduce flammability without compromising the structural integrity of the composites. The impact of these treatments on the thermal stability, wettability, and mechanical strength of the composites is examined using techniques such as DSC, TGA, and tensile tests. This chapter highlights the potential of sustainable, fire-resistant bamboo-epoxy composites for applications in safety panels, construction, and automotive industries, providing an eco-friendly alternative to synthetic fiber-based materials.

Keywords: bamboo fiber, epoxy composites, flame retardant, fiber–matrix adhesion, thermal stability, mechanical properties, sustainable materials

1. Introduction

Epoxy-based composite materials have been widely used in the automotive, construction, and aerospace industries due to their superior mechanical properties, chemical resistance, and ease of processing [1–7]. However, the high level of flammability in epoxy resins is a major challenge in applications that require fire safety [8–10]. On the other hand, increasing awareness of environmental sustainability is driving the search for alternative materials for synthetic fibers, such as fiberglass or carbon, with natural alternatives such as bamboo fiber. Bamboo fibers offer advantages in the form of high specific strength, low cost, and environmentally friendly

properties, but have disadvantages in the form of natural flammability and hydrophilicity [11–16].

To address this problem, a chemical modification approach with flame retardant (FR) inhibitors such as borax, ammonium polyphosphate (APP), or phosphorus-nitrogen compounds is a potential solution [17–19]. Recent research shows that the combination of bamboo fiber surface treatment and incorporation of FR agents into an epoxy matrix can create a synergistic effect, not only improving fire resistance but also maintaining or even improving the mechanical properties of composites. However, the interaction between the fiber-matrix interface, the distribution of FR agents, and their impact on thermal stability and fire performance still needs to be studied more deeply [20–27].

The purpose of this chapter is to comprehensively discuss the synergistic effect between flame retardant inhibitors and bamboo fiber reinforcement in epoxy composites. The main focus includes the effect of chemical treatment with various FR agents on the morphology and wettability of bamboo fibers. This section investigates how the chemical modification of bamboo fibers with various FR agents changes surface morphology and wettability properties. Through SEM and FTIR analysis, it was proven that chemical treatment not only creates an anti-fire protective layer but also increases surface roughness, which implies fiber-matrix adhesion. Further contact angle tests revealed changes in the hydrophobicity properties of the fibers after treatment, which correlated directly with the quality of interfacial bonding in the composite.

The mechanism of increasing fire resistance is through thermal degradation (TGA) analysis and flammability rating (UL-94/LOI). The synergistic effect of FR agents was evaluated through three layers of analysis, namely thermal degradation behavior (using TGA/DSC) to identify decomposition temperatures and stable char residue formation, and flammability rating (UL-94 and LOI), which proved a significant increase from HB (highly flammable) to V-0 (self-extinguishing in 10 seconds) classification.

FR modification impacts the values of tensile strength, impact resistance, and interfacial adhesion between the fiber and the matrix. This section quantifies the trade-off between fire resistance and mechanical performance through a tensile strength and flexural modulus test (ASTM D3039/D790) that confirms the existence of initial strength retention after optimal FR addition. In addition, Impact strength analysis (Izod/Charpy test) was performed to evaluate the toughness of composite shock loads. Interfacial adhesion characterization via SEM revealed that degradation of mechanical properties can be minimized by controlling the distribution of FR at the fiber-matrix interface.

The chapter's main contribution is to provide the latest insights into the design of epoxy-bamboo composites that are refractory yet high-performance, while highlighting their potential as a sustainable material for safety panel applications, transportation interiors, and fire-resistant buildings. This information is expected to be a reference for the development of eco-friendly composites in the future.

2. Bamboo as a fire-resistant composite reinforcer

Bamboo fiber has gained significant attention as a sustainable material due to its rapid growth, low production costs, and eco-friendly properties [28]. These fibers have high tensile strength, even exceeding conventional reinforced concrete in some applications, making them an attractive alternative for composite material reinforcement (**Figure 1**).



Figure 1.
a. Bamboo fiber b. Bamboo plants.

In addition, its flexibility allows for use in various structural forms, such as decorative elements as well as building components [28]. Another advantage is its biodegradable ability and abundant availability, thus reducing dependence on non-renewable resources [29].

However, bamboo fiber has a major disadvantage in the form of high hydrophilic properties, causing excess water absorption and reducing the durability of the material [30]. In addition, its natural flammability poses a serious challenge in applications that require fire resistance [31]. To address this problem, various chemical treatment methods have been developed, such as alkalization with sodium hydroxide, which effectively removes lignin and hemicellulose, thereby improving the adhesion of fibers with polymer matrices [32]. Silanization treatment is also used to increase the hydrophobicity of the fibers and strengthen the interface bonds in composites [30].

The addition of flame retardant inhibitors such as borax and ammonium polyphosphate (APP) after chemical treatment can further increase fire resistance without compromising mechanical properties [31]. The combination of chemical treatment and surface modification with flame retardant creates a synergistic effect that improves the bamboo fiber's weaknesses while maintaining its advantages as a sustainable material [29]. Recent research shows that chemically and physically modified bamboo fibers can improve the performance of composites in structural applications, such as lightweight concrete and automotive materials [33].

3. Fiber surface modification and interaction matrix

In this section, the technique of surface modification of bamboo fiber using FR inhibitors is discussed. This technique aims to improve compatibility with epoxy matrices. The results of the characterization of SEM and FTIR showed changes in the morphology and functional groups of the fibers.

3.1 Mechanical testing (single fiber tensile test and pull-out test)

The research that has been carried out on bamboo fiber is shown in the following stages in **Figure 2**:

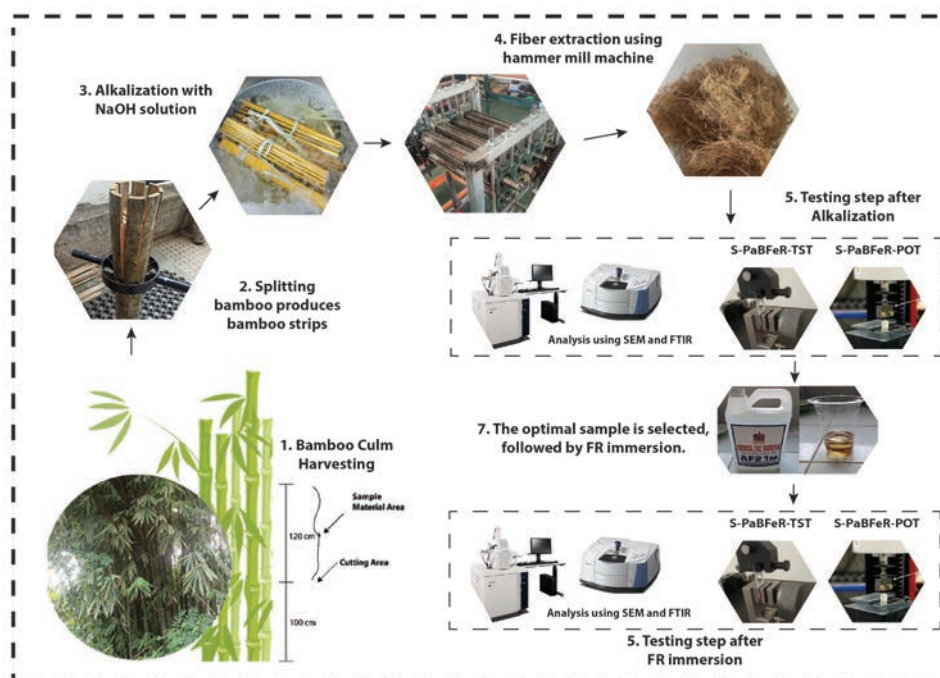


Figure 2.
Stages of mechanical testing.

Figure 2 shows the stages of mechanical testing of bamboo fiber, starting from stem selection, alkali treatment, fiber extraction using machinery, mechanical testing (pull-out test using matrix and single fiber tensile test) before and after FR Inhibitor treatment to SEM and FTIR testing to see the microstructure and functional groups formed.

The research method used was experimental by varying the concentration of NaOH starting from 0%/Normal, 3, 5, 7, and 11% with variations in immersion time of 0, 45, 90, and 135 minutes, respectively, to obtain optimal mechanical strength and watability values as shown in **Table 1** below:

Data obtained from five repetitions for each treatment condition showed consistency of results, with a relatively small standard deviation (<5% for tensile strength at the optimal treatment). The general pattern observed is that the results show that the NaOH5%45 treatment produces optimal performance with a tensile strength of 249.54 ± 0.11 MPa and a shear stress of 14.47 ± 0.69 MPa without pull-out (0%), an increase in mechanical strength in the concentration range of 3–5% NaOH, and a decrease of >7% NaOH. In addition, tensile strain stability is in the range of 3.2–3.4% for all NaOH treatments, and a reduction of pull-out percentage indicates the presence of fiber-matrix adhesions.

Sample Code	Single Fiber Tensile Test		Pull-Out Test	
	Tensile Strength (MPa)	Tensile Strain (%)	Shear Strength (MPa)	Pull-Out (%)
N	224.99 ± 7.88	4.53 ± 0.27	5.57 ± 1.74	100
NaOH3%45	246.16 ± 4.28	3.34 ± 0.004	13.60 ± 0.47	0
NaOH3%90	240.32 ± 8.53	3.40 ± 0.06	11.67 ± 0.27	0
NaOH3%135	237.33 ± 5.62	3.23 ± 0.14	9.62 ± 0.25	0
NaOH5%45	249.54 ± 0.11	3.35 ± 0.01	14.47 ± 0.69	0
NaOH5%90	246.33 ± 2.42	3.37 ± 0.06	13.53 ± 0.21	0
NaOH5%135	245.12 ± 4.97	3.33 ± 0.01	12.16 ± 0.12	0
NaOH7%45	240.36 ± 14.13	3.42 ± 0.16	12.69 ± 0.35	80
NaOH7%90	236.48 ± 14.63	3.31 ± 0.01	11.92 ± 0.82	60
NaOH7%135	222.49 ± 11.43	3.31 ± 0.01	9.63 ± 0.46	60
NaOH11%45	230.43 ± 7.01	3.22 ± 0.12	10.72 ± 1.09	80
NaOH11%90	221.29 ± 2.60	3.28 ± 0.07	7.95 ± 0.88	100
NaOH11%135	212.53 ± 4.52	3.22 ± 0.13	5.32 ± 0.51	80

Table 1.
Results of mechanical testing (pull-out test & single fiber tensile test) of bamboo fiber.

Comparison and validation, and data comparison tests (ANOVA & Tukey HSD) are shown in the following **Figure 3**:

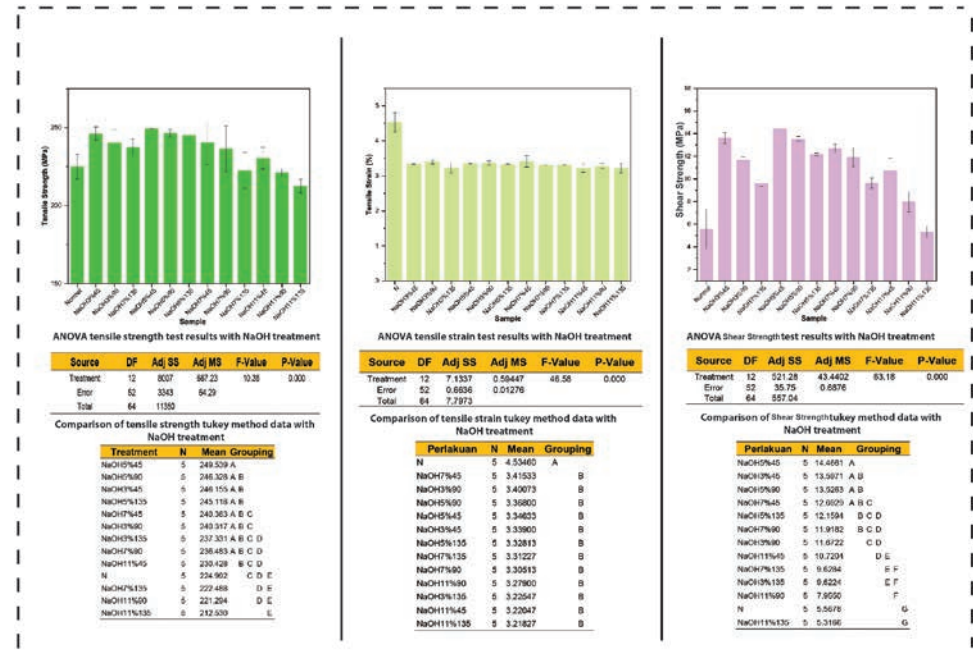


Figure 3.
Comparison of mechanical strength, results of ANOVA, and Tukey HSD statistical tests of NaOH treatment.

In **Figure 3**, the value of mechanical strength is shown as seen from tensile strength, tensile strain, and shear strength. The data was processed and validated using one-way ANOVA and Tukey-HSD comparison tests. The results of the ANOVA statistical test showed that alkalization using NaOH had a significant impact on mechanical strength with a P-value of <0.05 . It was confirmed in the Tukey-HSD comparison test that the 5% NaOH treatment sample for 45 minutes was significantly different from the other samples, even though the tensile strain was not different from other NaOH treatment samples. SEM and FTIR observations were carried out to see the microstructure and functional groups, as shown in **Figure 4** below:

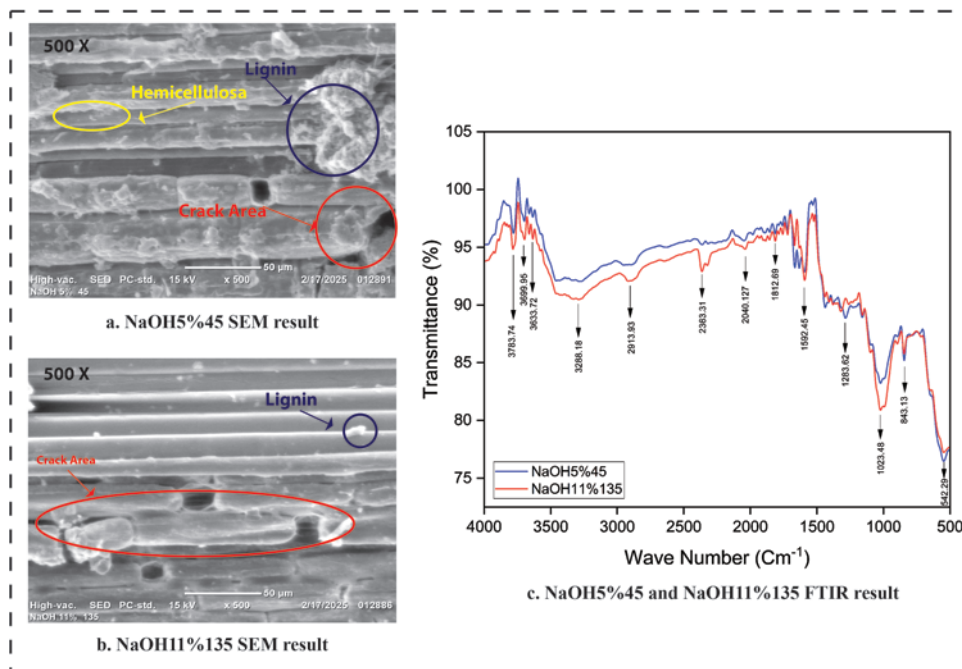


Figure 4.

SEM and FTIR results after 5% alkalization treatment for 45 min and 11% for 135 min.

Figure 4 displays a comparison of bamboo fiber microstructures after chemical treatment with two different NaOH concentrations and durations, observed at 500x magnification. Bamboo fibers treated with 5% NaOH for 45 minutes exhibited a cleaner and more organized structure, indicating effective removal of non-cellulosic components like lignin and hemicellulose without excessive damage. Minor cracks observed in these fibers had a negligible impact on overall integrity, contributing to an enhanced tensile strength of 249.54 ± 0.11 MPa. In contrast, fibers treated with 11% NaOH for 135 minutes displayed more severe degradation, with wider cracks and irregular structures, leading to reduced tensile strength (212.53 ± 4.52 MPa). This suggests that overly aggressive alkali treatment compromises fiber strength.

FTIR analysis of the treated fibers revealed notable changes in functional groups (**Figure 4**). The broad peak at $3600\text{--}3200\text{ cm}^{-1}$, corresponding to hydroxyl (-OH) groups in cellulose, hemicellulose, and lignin, showed reduced intensity after 11% NaOH treatment, indicating partial removal of hemicellulose and

lignin. The weakening of the peak at 3288.18 cm^{-1} , linked to hydrogen bonding in cellulose, suggests increased structural regularity due to amorphous component breakdown. Additionally, the peak at 1592.45 cm^{-1} , associated with aromatic C=C bonds in lignin, diminished more significantly with 11% NaOH, confirming greater lignin degradation compared to 5% NaOH. However, the C-O vibration band at 1023.48 cm^{-1} remained stable, indicating cellulose backbone preservation despite lignin and hemicellulose loss.

SEM and FTIR results demonstrate that NaOH treatment significantly alters PaBFeR bamboo fiber structure and composition. A 5% NaOH treatment for 45 minutes optimally enhances tensile strength with minimal structural damage, whereas 11% NaOH for 135 minutes causes excessive degradation, reducing fiber strength. Therefore, NaOH concentration and treatment duration must be carefully optimized to balance non-cellulosic removal with fiber integrity preservation.

After obtaining the optimal soaking result, namely 5% NaOH treatment for 45 minutes, observation was continued with FR Inhibitor treatment on the sample with a variation of immersion time of 0 minutes (previous results), 30, 45, and 60 minutes, as shown in the following **Table 2**:

Sample Code	Single fiber tensile test		Pull-out test	
	Tensile Strength (MPa)	Tensile Strain (%)	Shear Strength (N/mm ²)	Pull-Out (%)
NaOH5%45	249.54 ± 0.11	3.35 ± 0.01	14.47 ± 0.69	0
NaOH5%45 + FR30	245.81 ± 0.14	4.59 ± 0.23	12.82 ± 0.07	0
NaOH5%45 + FR45	241.87 ± 1.18	3.34 ± 0.003	11.87 ± 0.05	0
NaOH5%45 + FR60	237.26 ± 1.78	3.40 ± 0.06	11.17 ± 0.12	14.8

Table 2.
The results of the single fiber tensile strength and pull-out test results after FR Inhibitor treatment on 5% NaOH treatment samples for 45 minutes.

The initial tests on bamboo fiber samples treated with 5% NaOH for 45 minutes showed a tensile strength of 249.54 MPa with a strain of 3.35%. These values were used as a reference for comparing samples with additional flame retardant (FR) treatments of 30, 45, and 60 minutes. After 30 minutes of FR treatment, the tensile strength slightly decreased to 245.81 MPa while the strain increased to 4.59%, indicating greater deformation capacity before failure. Extending the FR treatment to 45 minutes resulted in a further reduction in tensile strength to 241.87 MPa, with the strain returning close to the original value at 3.34%. The 60-minute FR treatment led to a more substantial decrease in tensile strength to 237.26 MPa, accompanied by a slight strain increase to 3.40%.

In terms of shear strength, the 30-minute FR-treated sample exhibited a value of 14.47 MPa with no fiber pull-out, demonstrating strong adhesion between fibers and matrix. As the FR treatment duration increased to 45 minutes, the shear strength declined to 12.82 MPa while maintaining zero fiber pull-out. Further extension to 60 minutes reduced the shear strength to 11.87 MPa, still with no fiber detachment. However, with prolonged FR exposure, the shear strength continued decreasing to 11.17 MPa, and fiber pull-out reached 14.8%, indicating weakening interfacial bonding and potential compromise of the composite's mechanical integrity. These trends

are comprehensively compared in **Figure 5**, illustrating the effects of FR treatment duration on the mechanical properties of the composite material.

The ANOVA analysis revealed significant effects of FR treatment on the mechanical properties of PaBFeR NaOH5%45 fibers ($p < 0.001$). For tensile strength ($F = 40.31$), the between-group variation substantially exceeded within-group variation, with the Tukey test indicating no significant difference between untreated samples and 30-minute FR treatment. However, significant reductions occurred at 45 minutes, becoming more pronounced at 60 minutes.

Tensile strain showed even stronger treatment effects ($F = 149.52$), peaking at 30 minutes of FR exposure before decreasing to levels comparable to untreated samples at longer durations. Shear strength demonstrated similar trends ($F = 102.81$), with untreated samples exhibiting maximum values that progressively declined with increasing FR treatment time.

These results demonstrate that while brief (30-minute) FR exposure enhances deformation capacity without compromising strength, extended treatments beyond 45 minutes systematically degrade both tensile and shear properties. The optimal balance between flame retardancy and mechanical performance appears to occur at approximately 30 minutes of FR treatment.

Figure 6 presents SEM images and FTIR spectra of FR-treated PaBFeR NaOH5%45 fibers at 500 $\times$ magnification, revealing progressive structural and chemical changes with increasing treatment duration.

In the 30-minute FR-treated sample, the fiber structure remained largely intact, though minor cracks were visible. FTIR analysis confirmed the persistence of hemi-cellulose and lignin, evidenced by hydroxyl group peaks at 3741.45 and 3275.42 cm^{-1} , indicating limited chemical alteration. The aliphatic C-H vibration band at 2898.76 cm^{-1} remained stable, while a new peak at 2356.93 cm^{-1} suggested the formation of volatile FR reaction byproducts.

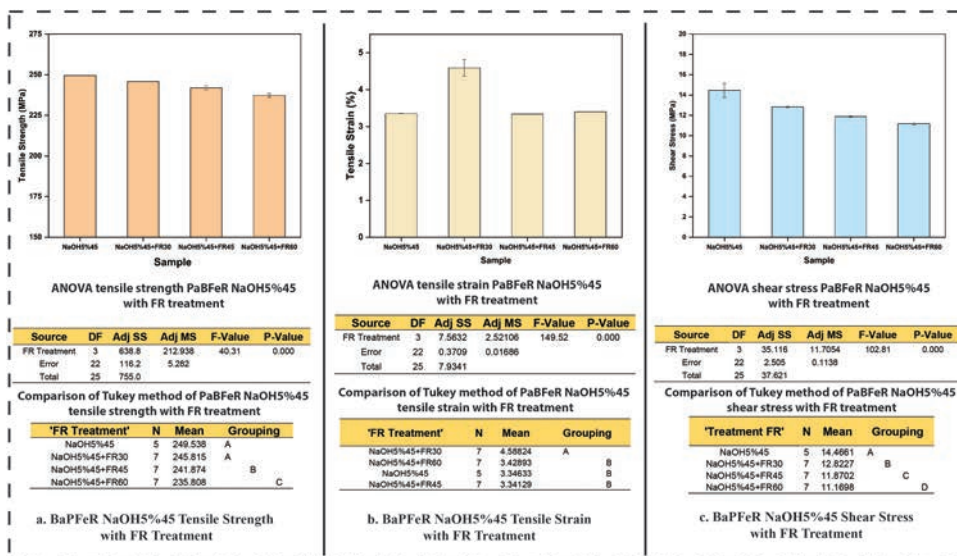


Figure 5.

Comparison of mechanical strength, statistical test results of ANOVA and Tukey HSD FR Inhibitor treatment on 5% NaOH treated fibers for 45 minutes.

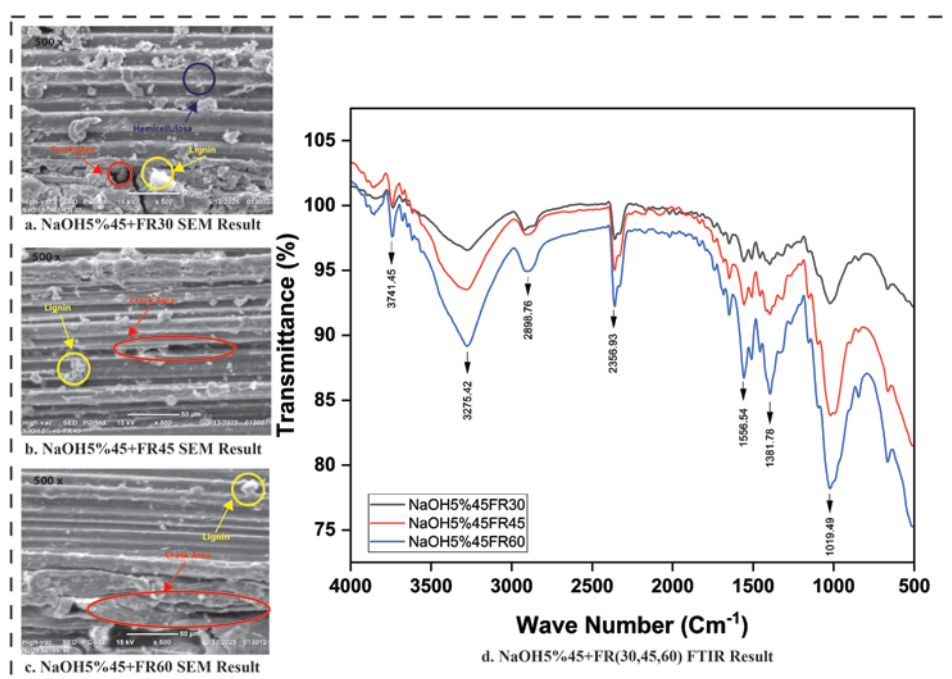


Figure 6.
SEM and FTIR results after FR-Treatment.

Extending treatment to 45 minutes led to more pronounced fiber fragmentation, with deeper cracks and advanced degradation of lignin and hemicellulose. The FTIR spectra reflected these changes: the hydroxyl peaks (3741.45 and 3275.42 cm^{-1}) showed reduced intensity, signaling stronger FR interaction. The lignin-associated band at 1556.54 cm^{-1} shifted slightly, confirming further breakdown, while the aliphatic C-H (2898.76 cm^{-1}) and volatile compound (2356.93 cm^{-1}) peaks persisted.

At 60 minutes, severe fiber degradation occurred, with widespread cracking and near-complete loss of hemicellulose and lignin. The FTIR spectra exhibited sharp declines in hydroxyl group peaks (3741.45 , 3275.42 cm^{-1}), highlighting extensive chemical modification. The 2356.93 cm^{-1} peak intensified, suggesting increased volatile byproducts, while the lignin band (1556.54 cm^{-1}) shifted further. Notably, the cellulose-related C-O vibration peak (1019.49 cm^{-1}) weakened, indicating structural damage to the fiber backbone.

These findings demonstrate that FR treatment duration critically impacts fiber integrity: 30-minute exposure preserves most properties, while 45–60-minute treatments progressively degrade the structure through lignin removal, hydroxyl depletion, and cellulose disruption. This aligns with mechanical data showing strength reductions at longer FR exposures.

3.2 Wettability test

In this study, the contact angle was measured using the sessile drop method following the ASTM D7334-08 (2022) standard, as seen in **Figure 7**.

For each test, a 70 cm-long piece of bamboo fiber was used. Seven matrix drops were dripped on the fibers using dropper pipettes. Each droplet was left for 2 minutes before its contact angle was measured using a $40\times$ magnification macro camera.

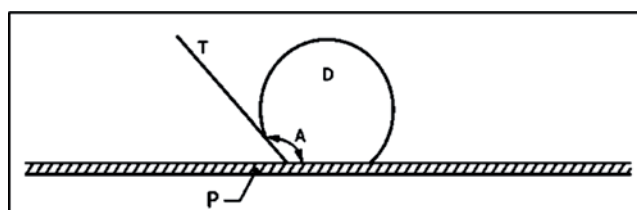


Figure 7.
ASTM D7334-2022 Contact Angle Measurement.

The obtained image is then analyzed with the ImageJ software to calculate the contact angle. Data were collected based on the minimum (x) and maximum (y) contact angle ranges observed at all seven drops ($x^\circ \leq \theta \leq y^\circ$). The optimal value approach is carried out based on the average ($AVG \pm STDEV$), and the results are compared to the ASTM D7334-2022 standard.

The measurement results are shown in the following **Table 3**:

No	Sample Code	Average Contact Angle (θ°)	Range Contact Angle (θ°)
1	N	$36.71^\circ \pm 2.71^\circ$	$32^\circ \leq \theta \leq 41^\circ$
2	NaOH3%45	$33.57^\circ \pm 7.44^\circ$	$21^\circ \leq \theta \leq 46^\circ$
3	NaOH3%90	$36.29^\circ \pm 4.8^\circ$	$26^\circ \leq \theta \leq 46^\circ$
4	NaOH3%135	$35.57^\circ \pm 2.77^\circ$	$31^\circ \leq \theta \leq 41^\circ$
5	NaOH5%45	$37.86^\circ \pm 2.36^\circ$	$33^\circ \leq \theta \leq 40^\circ$
6	NaOH5%90	$43.71^\circ \pm 4.46^\circ$	$39^\circ \leq \theta \leq 50^\circ$
7	NaOH5%135	$40.86^\circ \pm 4.58^\circ$	$34^\circ \leq \theta \leq 47^\circ$
8	NaOH7%45	$37.43^\circ \pm 3.06^\circ$	$33^\circ \leq \theta \leq 43^\circ$
9	NaOH7%90	$37.86^\circ \pm 2.36^\circ$	$34^\circ \leq \theta \leq 41^\circ$
10	NaOH7%135	$33^\circ \pm 3.30^\circ$	$29^\circ \leq \theta \leq 39^\circ$
11	NaOH11%45	$39^\circ \pm 2.51^\circ$	$35^\circ \leq \theta \leq 42^\circ$
12	NaOH11%90	$44.86^\circ \pm 1.88^\circ$	$42^\circ \leq \theta \leq 48^\circ$
13	NaOH11%135	$39.57^\circ \pm 5.15^\circ$	$32^\circ \leq \theta \leq 48^\circ$

Table 3.
Bamboo fibers contact angle measurement result.

Measurement of the contact angle using the sessile drop method showed that NaOH treatment with different concentrations and durations exerted varying influences on the surface properties of bamboo fibers. In the untreated control sample, an average contact angle of 36.71° was obtained with variations between 32° and 41° . Treatment with 3% NaOH for 45 minutes lowered the average contact angle to 33.57° , but with considerable variation (21° – 46°), showing inconsistency in surface modification.

Increased NaOH concentrations result in an interesting pattern of changes. At 5% NaOH, the contact angle tends to increase with the immersion time, reaching a high value of 43.71° after 90 minutes, before dropping back down at 135 minutes of immersion. Meanwhile, the 7% NaOH treatment showed a different tendency where the contact angle actually decreased significantly to 33° after 135 minutes. The highest concentration of NaOH (11%) resulted in the greatest contact angle (44.86° at 90 minutes), indicating the most significant change in the fiber surface.

The results of these measurements revealed that both the concentration and the duration of immersion in the NaOH solution affected the hydrophilic/hydrophobic properties of the bamboo fiber surface. This change in contact angle is closely related to the modification of fiber surface components such as lignin and hemicellulose during the chemical treatment process. To see the comparison between each of the concentration variations and the contact angle, the following **Figure 8** is shown:

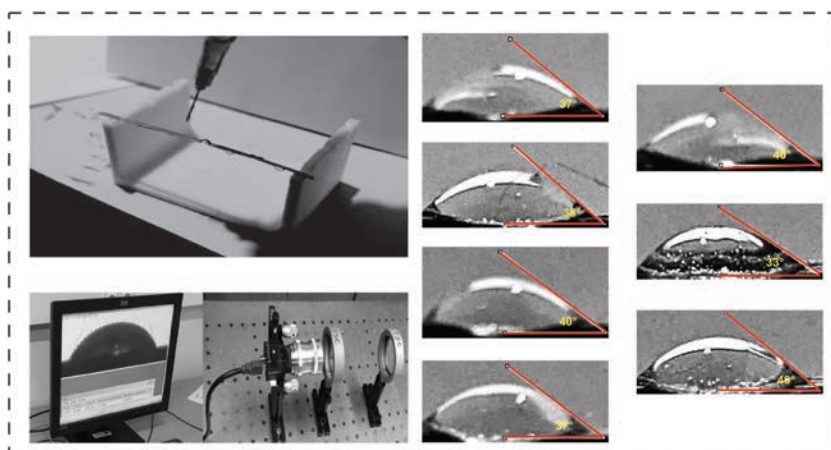


Figure 8.
Testing of bamboo fiber wettability.

Figure 9a shows that 45 minutes of immersion at various concentrations of NaOH resulted in a relatively small change in the angle of contact, with most treatments showing values below 45°. Exceptions occurred in 3% NaOH samples that showed considerable variation, with some measurement points exceeding 45°. This indicates that bamboo fibers still retain good wetness properties in a short soaking time.

More significant changes were observed when the immersion duration was extended to 90 minutes (**Figure 9b**). A concentration of 5% NaOH causes an increase in the angle of contact exceeding 45°, while 11% NaOH is close to the stated t-value. This pattern became more pronounced at 135 min immersion (**Figure 9c**), where 7% NaOH showed a noticeable decrease in wetness, while 11% NaOH resulted in a contact angle of up to 48°.

Figure 10 shows the effect of immersion time on the angle of contact at various NaOH concentrations. At a concentration of 3% (**Figure 10a**), the contact angle value is relatively stable despite the extended immersion time. The 5% concentration (**Figure 10b**) showed a consistent increase in wetness, but began to decrease after 135 minutes. The most significant change was seen at a concentration of 7% (**Figure 10c**), which reached optimal wetness after 135 minutes. Meanwhile, the concentration of 11% (**Figure 10d**) provides a marked increase in wetness but comes with the risk of fiber damage if the soaking time is too long.

In general, this study shows that both the concentration and duration of immersion in the NaOH solution significantly affect the surface properties of bamboo fibers. The observed change in contact angle reflects the modification of the fibers' hydrophilic properties as a result of such chemical treatments.

Based on the ASTM D7334-08 (2022) standard, a contact angle value below 45° indicates a good level of wetness. Measurements are made at several different points

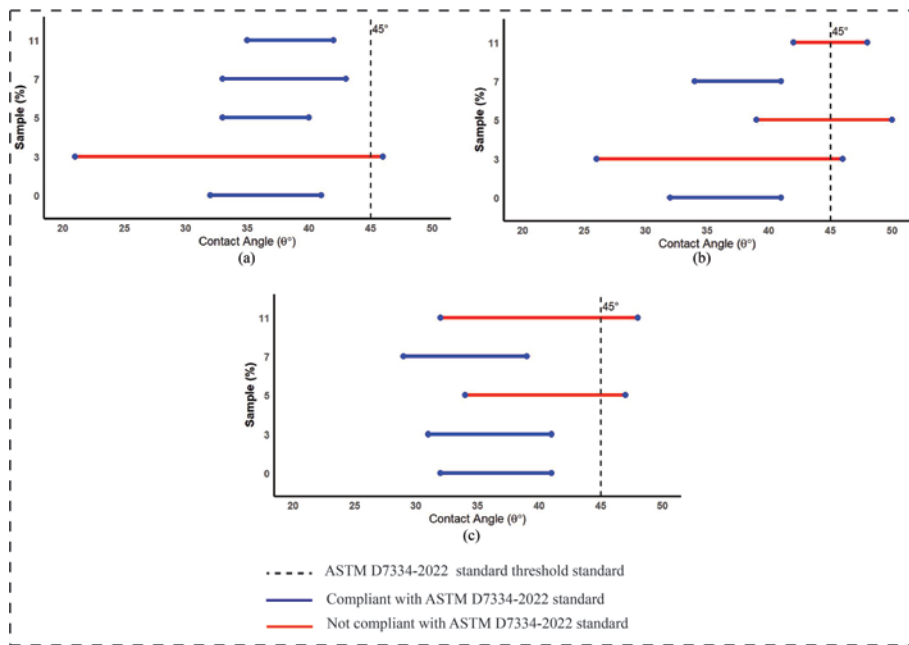


Figure 9.
The range of contact angle of bamboo fibers variations in NaOH concentration for (a) 45 minutes, (b) 90 Minutes, (c) 135 Minutes.

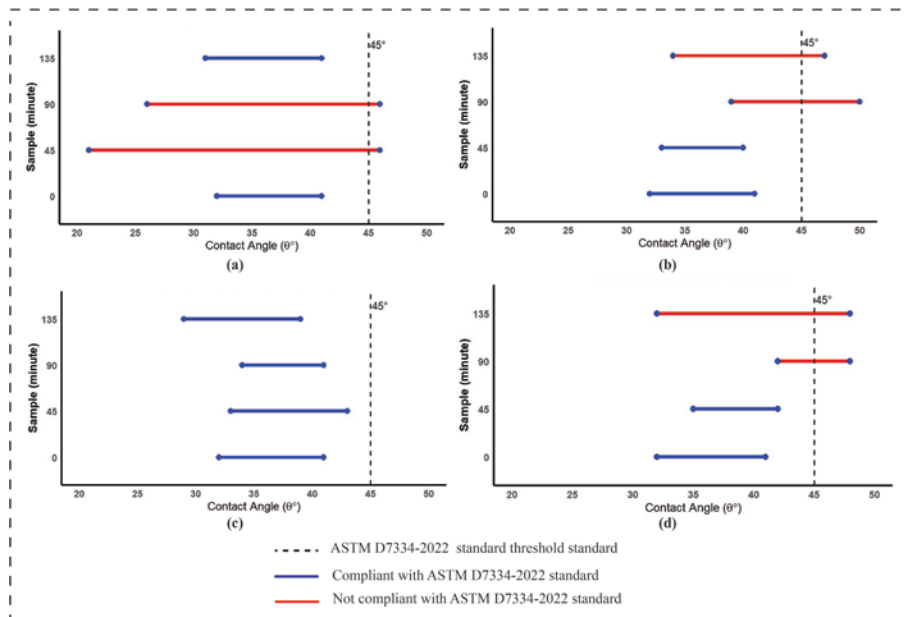


Figure 10.
The range of contact angle PaBFeR variations in different times for (a) 3% NaOH, (b) 5% NaOH, (c) 7% NaOH, and (d) 11% NaOH.

on the surface of the fibers to minimize local variations. The results showed that NaOH treatment with variations in concentration and duration significantly affected the wetness value of bamboo fibers. Although the NaOH7%135 sample recorded the lowest average contact angle (33°), the high standard deviation ($\pm 3.30^\circ$) and wide value range ($29\text{--}39^\circ$) made it less than optimal for practical applications.

The NaOH11%90 samples showed more stable characteristics, with an average contact angle of 44.86° (close to the 45° limit), the smallest standard deviation ($\pm 1.88^\circ$), and a narrow range ($42\text{--}48^\circ$). These results indicated better treatment consistency than the NaOH3%45 sample, with a large standard deviation (± 7.44) and a very wide range ($21\text{--}46^\circ$). These findings highlight the importance of balancing NaOH concentration and immersion duration to achieve optimal wetness.

Research reveals that low to moderate NaOH concentrations can increase fiber wetness, while high concentrations can potentially reduce fiber surface quality. Heat treatment with a long duration also needs to be taken into account because it can modify the chemical composition of the fibers. Overall, surface modification with NaOH is effective in improving the interaction of bamboo fibers with FR-P resin in composite applications, especially in improving fire resistance. Further optimization is required to test the performance of composites under real-world application conditions.

Nonetheless, fiber degradation in alkaline environments and sensitivity to moisture remain challenges that need to be addressed through optimization of chemical treatment and selection of appropriate flame retardants [33]. The development of hybrid treatment methods, such as a combination of alkalization with nanoparticle encapsulation, is also being explored to improve the durability and thermal stability of bamboo fibers in composites [34]. With the continued development of fiber modification technology, bamboo fiber has great potential to become a sustainable alternative to synthetic fiber [35].

Further studies are needed to evaluate the long-term performance of bamboo fiber in various environmental conditions and optimize production costs to compete with conventional materials [36]. In addition, standardization of extraction and treatment methods needs to be carried out to ensure consistency of fiber quality on an industrial scale [37]. Thus, the use of bamboo fiber can contribute to the development of green materials that are not only strong and flexible but also environmentally friendly.

Bamboo fiber has great potential in a wide range of structural and non-structural applications as an epoxy-matrix composite reinforcer. Epoxy was chosen as a matrix because of its excellent adhesion properties, dimensional stability, and high chemical resistance [30]. When chemically treated bamboo fibers are incorporated into the epoxy matrix, there is a significant improvement in the mechanical properties of the composite, such as tensile strength and impact resistance, compared to pure epoxy [29]. This is due to the ability of bamboo fibers, which have a high tensile strength, to withstand loads and distribute stress evenly throughout the matrix [38].

The advantage of bamboo fiber-epoxy composites is their excellent strength-to-weight ratio, making them suitable for applications requiring lightweight yet strong materials, such as automotive components and building panels [29]. In addition, these composites offer environmental advantages because they use renewable natural materials and have a lower carbon footprint than synthetic fiber composites [28]. However, flammability is still an issue that can be overcome by adding flame retardants such as ammonium polyphosphate or borax that work synergistically with bamboo fibers to increase fire resistance without significantly reducing mechanical properties [31].

3.3 Bamboo fibers burning test

Bamboo fiber has become one of the natural materials that has attracted attention in the development of refractory components due to its superior mechanical properties and promising thermal characteristics, as discussed in the previous discussion. This study thoroughly evaluated the fire resistance of bamboo fibers through a series of controlled combustion tests. Bamboo fibers are processed through the delignification stage using a 5% NaOH solution to remove lignin, then dried at 60°C for 24 hours to reduce moisture content. After drying, the fibers are processed into a thin layer 1 mm thick using a splitter machine. Bamboo fiber samples are prepared with length $\times$ width $\times$ thickness sizes of 300 $\times$ 15 $\times$ 1 mm. Furthermore, the dried fibers were given FR Inhibitor treatment with time variations of 0 minutes (control), 30, 45, and 60 minutes. For easy identification, the sample is coded FR-X, where X represents the length of the FR treatment (e.g., FR-30 for a 30-minute treatment). Control samples that were not given FR treatment were coded Normal. This process aims to study the effect of FR treatment duration on the properties of bamboo fibers, especially in composite material applications. The testing level is shown in **Figure 11**:

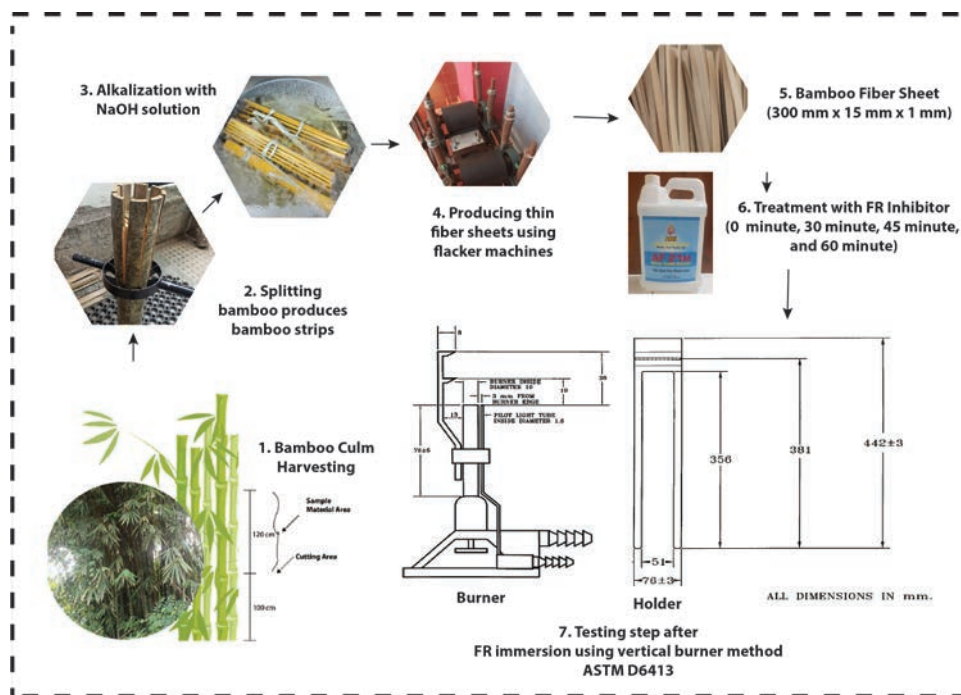


Figure 11.
Stages of bamboo fiber burning testing.

The combustion test equipment is prepared according to ASTM D6413/ D6413M-15 standard, which consists of a Tirrill burner with a barrel diameter of 10 mm and a length of 76 $\pm$ 6 mm. The burner is equipped with a needle valve to regulate the flame height, which is stabilized at 38 mm using a flame height gauge (metal plates marked 19 and 38 mm) and monitored by a flame pilot (flame height 3 mm, diameter 1.5 mm, 3 mm away from the burner edge). About 99%

methane gas is delivered at a pressure of 17.2 ± 1.7 kPa through a solenoid valve that operates in <0.2 seconds.

Bamboo fiber samples measuring 300×15 mm are clamped vertically on a 76×305 mm metal sample holder. The burner position is set so that the bottom edge of the sample is 19 mm above the burner tip. The entire set of test devices can be seen in **Figure 2**. The system is designed to accurately measure the fire resistance of materials, including the time of combustion, the rate of fire spread, and the char formation characteristics of bamboo fibers. The combustion testing apparatus is shown in **Figure 12**:



Figure 12.
Flame testing apparatus.

The test was carried out by placing the sample vertically on the specimen holder, precisely 19 mm above the burner tip. The flame is then applied to the bottom of the sample for 12 seconds. During the test, three main parameters were carefully recorded: (1) after-flame time (the duration of the flame after the fire source is extinguished) measured using a digital stopwatch with an accuracy of ± 0.2 seconds, (2) after-glow time (the length of the embers remaining after the fire is extinguished), and (3) the char length measured after the sample has cooled. For char length measurement, the burned area is folded and given a weight of 100 grams before being measured using a digital caliper with an accuracy of ± 3 mm. These procedures are designed to comprehensively evaluate three fundamental aspects of material fire resistance: self-extinguishing capability, coal persistence, and the extent of material damage due to controlled fire exposure. Precise char length measurements are crucial to determine how much material integrity is degraded by exposure to heat and fire.

Table 4 shows that the control sample without FR treatment had a longer After-Flame Time and After-Glow Time than the sample with flame retardant treatment, and produced the largest Char Length. The longer the duration of the FR immersion,

Sample Code	After-Flame Time (s)	After-Glow Time (s)	Char Length (mm)	Information
Normal	6.5 ± 0.3	5.2 ± 0.2	295 ± 16	Flammable, flame, and old embers
FR-30	4.2 ± 0.2	3.1 ± 0.1	289 ± 12	Significant decrease in flame and embers
FR-45	2.8 ± 0.1	1.7 ± 0.1	210 ± 13	Real fire resistance improvement
FR-60	1.3 ± 0.1	0.8 ± 0.05	182 ± 9	Best performance, almost extinguished the flame.

Table 4.
Bamboo fiber burn test result.

the shorter the After-Flame Time, After-Glow Time, and Char Length that will be produced. The difference in fire resistance performance between the control and FR-treated samples can be clearly observed in **Figure 4**. These results prove that flame retardant treatment effectively increases the resistance of bamboo fibers to fire.

Figure 13 shows a significant decrease in After-Flame Time and After-Glow Time as the duration of FR immersion increases. The value of After-Flame Time was reduced from 6.5 ± 0.3 seconds (Normal sample) to 1.3 ± 0.1 seconds (FR-60), while After-Glow Time decreased from 5.2 ± 0.2 seconds to 0.8 ± 0.05 seconds in the FR-60 treatment. These results show that soaking for 60 minutes effectively increases the fire resistance of bamboo fibers. The graph confirms the linear relationship between the duration of the FR treatment and the improvement of the fire retardant performance of the material, where the longer the duration of immersion, the better the material's ability to resist the spread of fire and maintain its structural integrity. A comparison of the char lengths produced after flame testing is shown in **Figure 14**:

Figure 14 shows a significant decrease in the char area length as the FR treatment duration increases. The Normal sample recorded the longest char length

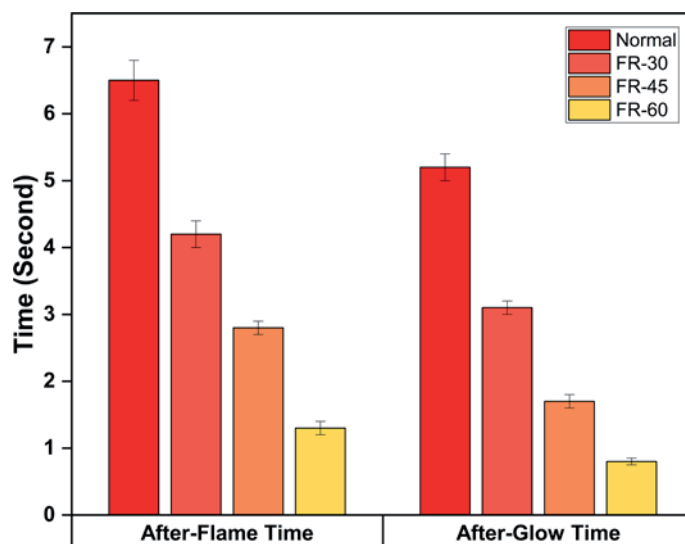


Figure 13.
After-Flame Time and After-Glow Time.

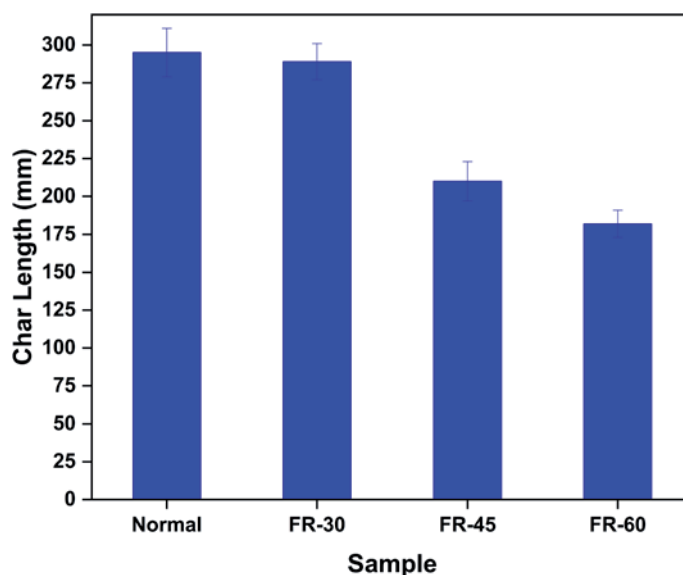


Figure 14.
Bamboo fiber.

(295 ± 16 mm), while FRP-60 reached the lowest value (182 ± 9 mm). The visual differences in combustion results between samples are clearly shown in **Figure 15**, which visualizes the combustion test results on bamboo fibers.

Figure 15 visually compares the results of burning bamboo fibers with four treatment variations. Untreated samples (Normal) showed the most extensive burned area with a deep black color and severe material damage. At the 30-minute treatment (FR-30), the charred area begins to shrink with more regular boundaries. The 45-minute treatment (FR-45) showed significant shrinkage of the burnt area, accompanied by a brownish color in the transition area. In contrast, the 60-minute treatment (FR-60) produced the smallest burnt area with a lighter color and the most preserved material structure. This visual change proves that the longer the duration of the flame

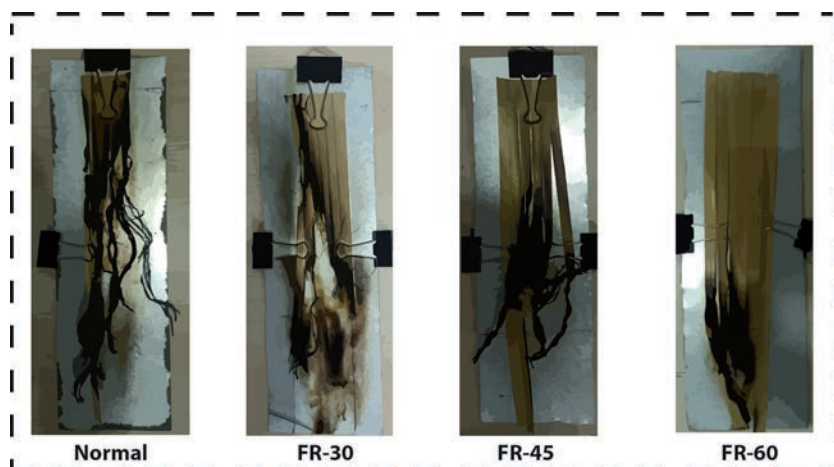


Figure 15.
Visualization of the bamboo fiber flame test result.

retardant immersion, the more effective it is in limiting the expansion of the burn area and maintaining the integrity of the material.

The significance of the effect of FR Inhibitor treatment on After-Flame time, After-Glow time, and Char Length was analyzed using the one-way ANOVA test, followed by the Tukey-HSD test to identify the significant differences between the treatments. The statistical analysis results confirm increasing flame retardant performance as the immersion duration increases. The results of data validation are shown in **Figure 16**.

Statistical analysis using one-way ANOVA and Tukey HSD follow-up test showed a significant effect of flame retardant treatment on all flame resistance parameters ($p < 0.001$). At after-flame time, there was a marked difference between treatment groups ($F = 733.00$), with a gradual decreasing pattern as the duration of immersion increased. The FR-60 treatment produced the lowest after-flame time (1.30 ± 0.1 seconds), significantly different from the control (6.50 ± 0.4 seconds). A similar pattern was seen in after-glow time ($F = 1301.04$), where FR-60 provided the best value (0.79 ± 0.1 seconds), or 84.9% lower than control (5.23 ± 0.3 seconds).

Char length analysis showed a more complex pattern ($F = 279.33$). Although the 30-minute (FR-30) treatment had not shown a significant difference from control ($p > 0.05$), the 45-minute (FR-45) and 60-minute (FR-60) immersions gradually reduced the char length significantly. The FR-60 recorded a best char length of 182.14 ± 9 mm, or 38.3% lower than the control (295.00 ± 16 mm). These results show that a minimum immersion duration of 45 minutes is required to significantly increase fire resistance, with maximum optimization at 60 minutes.

These findings are statistically consistent and support the use of flame retardants with a duration of 60 minutes for applications of bamboo fiber-based composite materials that require optimal fire resistance. The analysis was conducted with a confidence level of 95% ($\alpha = 0.05$), indicating the robustness of the results. However, several aspects need to be considered in the manufacture of composites, such as

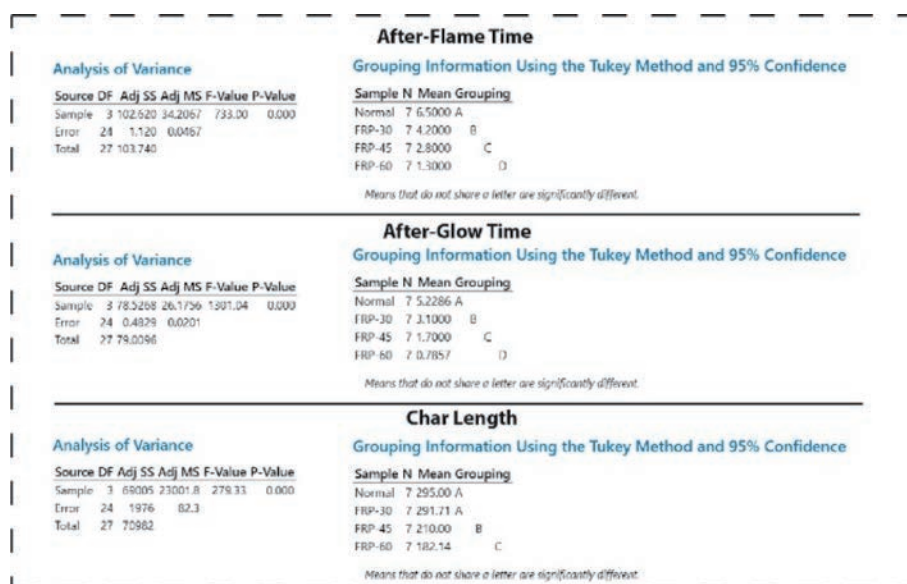


Figure 16.
ANOVA and Tukey HSD result.

mechanical strength and compatibility with the matrix used. The study has not tested the thermal stability (TGA/DSC) or post-treatment mechanical properties, which is an opportunity for further research.

These findings reinforce the potential of bamboo fibers as a sustainable fire retardant material, although further validation is needed for large-scale applications. Combining with nanomaterials or performing performance testing in composite systems can be the next step to achieving higher safety standards.

The combination of bamboo fibers with matrices has the potential to produce composites with superior performance in fire resistance and mechanical properties. The treatment of bamboo fiber using 5% NaOH for 45 minutes improved adhesion with the matrix, reflected in the tensile strength of 249.54 MPa and shear strength of 14.47 MPa without fiber pull-out. The addition of flame retardant (FR) for 60 minutes significantly reduces the after-flame time from 6.5 seconds to 1.3 seconds and the char length from 295 to 182 mm, meeting the UL-94 standard with V-0 classification.

The effectiveness of FR in forming a protective layer can be seen from the burn test, where the modified fibers showed a smaller burnt area and a more preserved material structure than the control sample. Although the TGA analysis has not yet been performed, the results of the UL-94 test prove an increase in fire resistance without compromising mechanical integrity. This combination makes bamboo fiber-based composites suitable for construction and automotive applications that require lightweight, strong, and fire-resistant materials.

Optimizing the duration of FR immersion is key, where the 60-minute treatment shows the best performance. Further research is needed to test thermal stability and durability under real environmental conditions. Still, these findings have opened up opportunities for developing sustainable materials as alternatives to synthetic fibers. Next, the role of epoxy in strengthening fire-resistant composites will be discussed.

4. Role of epoxy in bamboo fiber-reinforced flame retardant composite

Epoxy matrices play a crucial role in bamboo fiber-reinforced composites, especially in forming synergistic flame retardant systems. As the main binder, epoxy not only optimally transfers the load to the strong bamboo fibers but also serves as a homogeneous flame retardant (FR) carrier medium throughout the composite structure. The uniqueness of epoxy lies in its ability to form a stable char layer when exposed to fire, while protecting bamboo fibers from thermal degradation (**Figure 17**).

The epoxy resin shown in the picture is the chemical structure of diglycidyl ether of bisphenol A (DGEBA), one of the most widely used types of epoxy resin in the industry. Its main structure consists of three important parts: a highly reactive epoxy group, a rigid bisphenol A nucleus, and a flexible ether connecting chain. An epoxy group with its three-member ring is the reaction center during the hardening process,

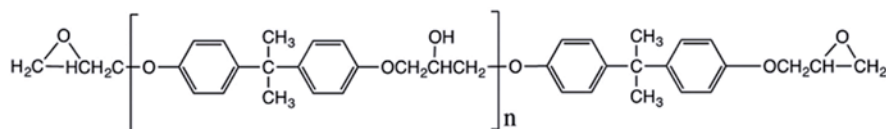


Figure 17.
 Chemical structure of epoxy resin.

while the bisphenol A core provides thermal stability and rigidity to the material. The ether chain between the bisphenol A core and the epoxy group gives the resin a certain flexibility.

Naturally, this DGEBA resin is amorphous or does not form a regular crystal structure. This is due to its asymmetrical and branched molecular structure, as well as the presence of large phenyl rings that block the tightly packed molecules. This amorphous property is advantageous in composite applications because it allows the resin to fill in the gaps and adhere well to the modified bamboo fibers. When this epoxy resin is combined with bamboo fibers, its amorphous structure facilitates the formation of a strong interface between the fiber and the matrix, which is essential for obtaining good mechanical properties.

Despite its amorphous nature, epoxy resins still have good strength and durability, thanks to the presence of aromatic rings in the bisphenol A core. However, its amorphous properties limit the thermal conductivity of resins. In composite applications with bamboo fibers, this epoxy resin provides a good balance between strength, thermal resistance, and the ability to bond with natural fibers. Its unique chemical structure makes it a well-suited material for the development of high-performance, eco-friendly composites (**Figure 18**).

The interaction between the surface of the chemically modified bamboo fiber and the epoxy matrix creates an interface capable of resisting the spread of fire, while maintaining the structural integrity of the composite. This combination results in a material that meets fire resistance standards such as UL-94 V-0 and maintains the mechanical performance required for industrial applications. Recent studies have shown that epoxy formulations with specific FR content can improve the self-extinguishing properties of composites by up to 80%, opening up opportunities for the innovation of versatile materials with superior mechanical properties, yet flammable, so that they require modifications to improve fire resistance. The addition of phosphorus compounds such as epoxyphosphene has been proven to increase impact resistance while reducing the rate of fire spread and resistance to petroleum products [39]. The combination of phosphorus and silicon in the DPSi-ED additive can achieve an LOI of 35.3% and a V-0 rating, with a significant decrease in heat and smoke emission by forming a stable char layer [23]. Lignin modified with APP and melamine showed a 15.3% increase in carbon residue and an LOI of up to 36.1%, while reducing smoke production [40].

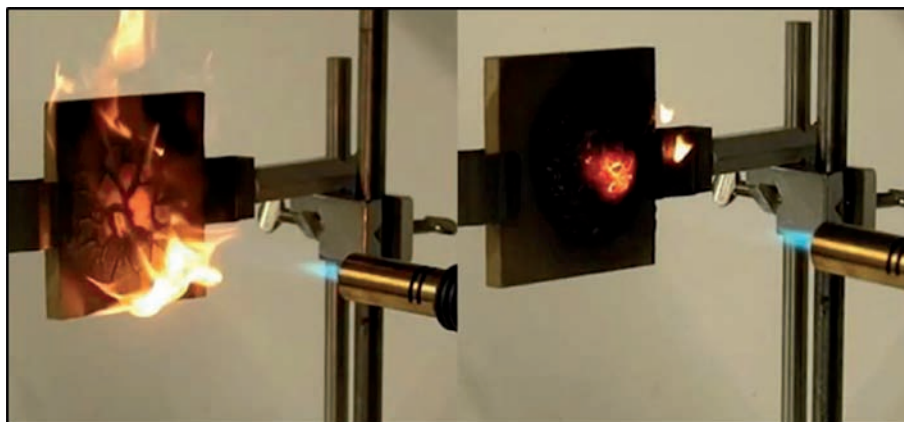


Figure 18.
Illustration of epoxy resin flaming test.

CNT and LDH-based composite structures combined with DOPO successfully suppress total heat dissipation by 19% and smoke by 25.56%, with an LOI of 31% [10]. Epoxy phenol aralkyl resin offers high flame retardancy without harmful halogen compounds, relying on forming a stable foam layer when burning [41]. Although ATH increases fire resistance, its use can reduce the fracture toughness of epoxy resin [42].

Recent developments show that P-Si additives with a phosphorus content of 1% weight can achieve an LOI of 33% and a V-0 rating while increasing tensile strength by 48.41% and impact resistance by 130% [43]. Approaches based on natural materials, such as lignin and a synergistic combination of phosphorus and silicon, are sustainable solutions to improve material performance. Formulation optimization and additive compatibility with resin matrices remain key challenges for wider industrial applications.

To see the characterization of the influence of temperature on materials, measurements were made using Thermogravimetric Analysis (TGA) and Differential Scanning Calorimetry (DSC) techniques, which are critical methods to evaluate the thermal properties of epoxy-based composite materials. DSC measures thermal transitions such as glass transition temperature (T_g), melting point, and crystallization, which are essential for understanding the thermal stability and degree of hardening of polymers [43, 44]. In epoxy, DSC is also used to monitor hardening reactions and detect thermal instability through changes in heat flow [45, 46].

TGA focuses on analyzing thermal decomposition and material composition by measuring changes in mass with temperature. Adding fillers such as nanosilica has been shown to increase the thermal stability of epoxy, as demonstrated by the increase in maximum degradation temperature [47]. TGA can also quantify the filler and resin content in composites and study degradation kinetics for material recycling optimization [48].

The combination of DSC and TGA provides a comprehensive understanding of thermal resistance and material aging. For example, in glass fiber-reinforced epoxy laminates, these techniques identify structural changes due to thermal aging through T_g shifts and degradation profiles [46]. DSC modifications such as temperature-modulated DSC (TMDSC) and TGA paired with FTIR allow a more in-depth analysis of phase transitions and degradation mechanisms [49].

This technique's main applications include optimizing material formulations, such as graphite oxide-reinforced epoxy composites that show a 64% increase in fracture toughness and thermal stability [50]. In the electronics industry, DSC and TGA guarantee the quality of PCB laminates by analyzing thermal expansion and hardening degrees [44].

Recent developments include using fast-scanning DSCs to study polymers under non-equilibrium conditions and local thermal analysis techniques to characterize nanocomposites [43, 49]. Thus, DSC and TGA support cutting-edge materials research and ensure the reliability of epoxy applications in the aerospace, automotive, and electronics fields.

Bamboo-epoxy fiber composites require thorough thermal characterization to ensure optimal application performance. DSC is important in analyzing epoxy matrices' glass transition properties (T_g), where high T_g values indicate good thermal resistance to extreme temperature environments [44]. Furthermore, DSC can monitor the degree of epoxy hardening, which directly affects the composite's mechanical strength, and reveal the effects of the chemical treatment of bamboo fibers on the adhesion of the interface through the heat profile changes detected [46, 51].

Meanwhile, the TGA complements the analysis by measuring the material's thermal stability by observing temperature, degradation, and composition. Natural

bamboo fibers that tend to degrade below 300°C can be increased in resistance to 400°C by combining epoxy matrices and adding flame retardants such as ammonium polyphosphate (APP) or nanosilica [40, 47]. TGA data also allows the identification of the mass contribution of each component as well as the prediction of the material's lifespan in hot environments, which is crucial for structural applications [48].

Integrating these two analysis techniques offers a comprehensive understanding of composite behavior. DSC detects changes in polymer properties due to additive additions, while TGA quantifies improvements in thermal stability and charcoal residues. For example, the use of lignin-based flame retardant not only increases carbon residues by up to 15.3% [40], but can also affect the observed glass transition properties through DSC. This combination analysis is an important cornerstone in the development of materials for the construction industry that requires thermal stability >300°C and optimal Tg values [44], as well as for automotive and aerospace components that require fire resistance certification [39].

The main challenge in developing bamboo-epoxy composites lies in the thermal degradation of bamboo fibers and the compatibility of additives with the matrix. Innovative solutions such as fiber chemical modification and the addition of nanosilica fillers [47] need to be continuously developed, with DSC and TGA acting as tools to verify their effectiveness. In the future, this integrated thermal analysis approach will be increasingly vital in balancing performance, sustainability, and economics, opening up wider application opportunities for environmentally friendly bamboo-epoxy composites (**Figure 19**).

The glass-carbon/epoxy composite thermal analysis graph provides an overview of the functions of TGA and DSC in material characterization. TGA measures the change in material weight with temperature, where the decrease in weight indicates the degradation stages of components such as epoxy matrix and fibers. DSC complements the analysis by detecting energy changes through heat flow measurements, identifying important transitions such as glass transition points and exothermic or endothermic reactions.

A similar analytical approach can be applied to natural-epoxy fiber composites such as bamboo-epoxy, although they have different characteristics. Bamboo fibers tend to degrade at lower temperatures than carbon/glass fibers, so their TGA profile will show an early weight loss. DSC data can reveal how chemical treatment of bamboo fibers affects epoxy matrices' interface adhesion and thermal stability.

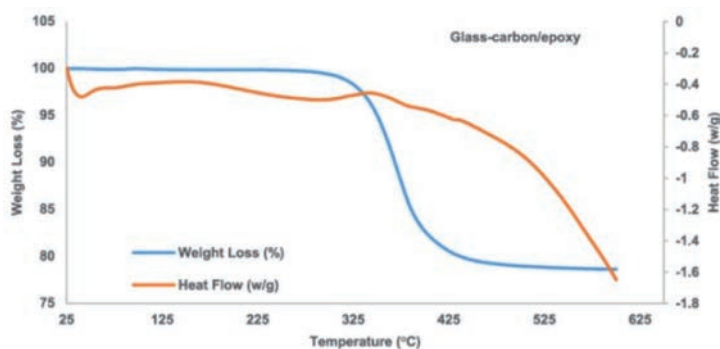


Figure 19.
TGA/DSC Glass-carbon/epoxy.

Figure 20a shows the TGA graph of the bamboo fiber composite, and **Figure 20b** shows the graph of the DSC of the bamboo fiber composite. The TGA graph presents the thermal degradation profiles of bamboo fiber composites under different chemical treatments in a nitrogen atmosphere. The untreated control samples (TP series) with varying concentrations (45, 55, 65, and 85%) exhibit distinct degradation patterns, where TP 45 and 55% demonstrate superior thermal stability compared to TP 65 and 85%. The NaOH 5%-treated samples at 45 and 65% concentrations display significantly different thermal behaviors from the untreated samples, highlighting the substantial impact of chemical modification on thermal resistance. The reference sample (Rearn FRP 100-S) shows optimal performance with minimal weight loss, indicating the highest thermal stability among all tested specimens.

Three characteristic degradation phases are observed: moisture/volatile release below 150°C, organic component decomposition between 200 and 400°C, and stable residue formation above 400°C. The varying degradation profiles among samples clearly reflect differences in fiber-matrix interactions and treatment efficacy. These findings provide critical insights for optimizing bamboo fiber composite formulations, particularly in selecting appropriate chemical treatment types and concentrations for specific applications requiring defined thermal stability thresholds. The comparative analysis reveals that while chemical treatments can enhance certain properties, the untreated reference composite maintains superior thermal performance, suggesting potential trade-offs between treatment benefits and inherent material stability.

The DSC thermogram presents the thermal characteristics of chemically treated bamboo fiber composites analyzed under a nitrogen atmosphere. Samples treated with 5% NaOH at varying concentrations (45, 55, 65%) exhibit markedly different heat flow profiles compared to TP control samples (45, 55, 65%), demonstrating the significant impact of alkaline treatment on the material's thermal behavior. The reference FRP 100-5 resin displays distinct thermal properties, highlighting how bamboo fiber incorporation modifies the composite's characteristics.

The observed exothermic peak patterns reveal variations in thermal degradation mechanisms across samples, with reaction energies and temperatures differing according to treatment type and concentration. These differences clearly illustrate how chemical modifications influence the composite's thermal stability while revealing the complex interfacial interactions between natural fibers and polymer matrices.

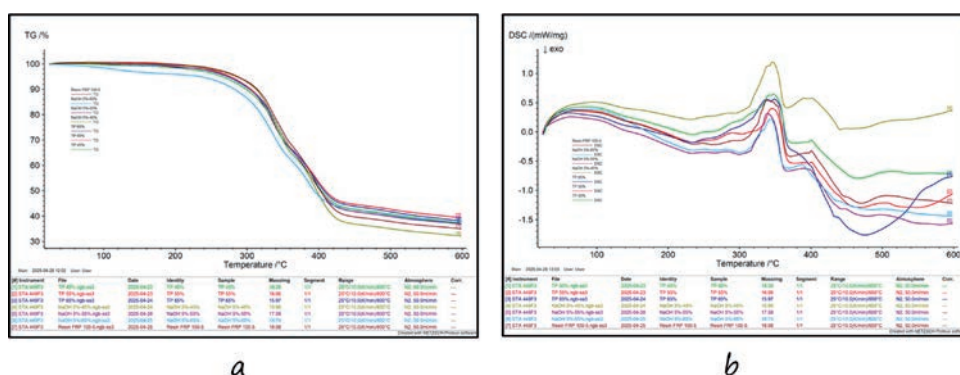


Figure 20.
a. TGA b. DSC Bamboo Fiber Composite.

This DSC analysis provides critical insights for developing bamboo fiber-reinforced composites, particularly in predicting end-product thermal performance and evaluating the effectiveness of various chemical treatment methods. The data serves as a fundamental reference for optimizing material formulations that meet specific thermal stability requirements while preserving the eco-friendly advantages of natural fiber composites. The comprehensive thermal profiles enable researchers to balance performance needs with sustainable material development goals.

The integration of TGA and DSC data is crucial to optimizing composite formulations. In bamboo-epoxy composites, the addition of a flame retardant or coupling agent will increase the degradation temperature in the TGA and change the heat profile in the DSC. This analysis helps balance mechanical performance, thermal resistance, and material sustainability for construction or automotive applications. Together, the TGA and DSC provide a scientific basis for developing superior natural fiber-based composite materials while ensuring compliance with stringent industry standards.

5. Conclusions

It is concluded that there is a synergistic effect between modified bamboo fiber and flame retardant (FR) in epoxy-based composites, which simultaneously improves fire resistance and maintains the material's mechanical properties. Optimal alkaline treatment (NaOH 5%, 45 minutes) increases the tensile strength of bamboo fibers to 249.54 MPa with perfect adhesion (0% pull-out). In contrast, FR impregnation for 60 minutes lowers the after-flame time from 6.5 to 1.3 seconds and char length from 295 to 182 mm, achieving UL-94 V-0 classification. Microscopic analysis revealed that chemical modifications created a coarser fiber surface with better matrix-fiber bonding, while FR formed a protective char layer during combustion. Despite a 4.9% decrease in mechanical strength at the 60-minute FR treatment, the final grade still meets the requirements of structural applications. These results suggest that the modified bamboo-epoxy fiber composite has great potential as a sustainable construction material with superior flame retardant performance. However, further research on long-term thermal stability and optimization of FR formulations for industrial applications is required.

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Conflict of interest

The authors declare no conflict of interest. There are no financial, professional, or personal relationships that could influence the objectivity of the research or its presentation. All data and findings reported herein represent unbiased academic research outcomes without external influence. This book chapter presents original research on the synergistic effects of flame retardants and bamboo fiber reinforcement in epoxy-based composites. The content, including methodologies, data analysis, and conclusions, has not been previously published in identical form. Any referenced materials have been properly attributed in accordance with academic ethics standards. The authors take full responsibility for the authenticity and accuracy of all presented data. This work was specifically developed to contribute to the advancement of sustainable, fire-resistant composite materials in materials science and engineering applications.

Notes/thanks/other declarations

The authors confirm that this book chapter presents original, unpublished research with proper attribution to all referenced works, and declare no competing interests that could influence the study's objectivity.

Nomenclature

Abbreviations

FR	flame retardant
APP	ammonium polyphosphate
SEM	scanning electron microscopy
FTIR	Fourier Transform Infrared Spectroscopy
TGA	thermogravimetric analysis
DSC	differential scanning calorimetry
UL-94	underwriters laboratories flammability standard
LOI	limiting oxygen index

Symbols

θ°	contact angle (degrees)
$^\circ\text{C}$	glass transition temperature (Celcius)
F	F-statistic (ANOVA)

Units

MPa	Megapascals (tensile/shear strength)
s	Seconds (combustion time)

mm Millimeters (char length)
cm⁻¹ Wavenumber (FTIR)

Material codes

NaOHX%Y X% NaOH concentration, Y minutes immersion.
FR-Z Z minutes of flame retardant treatment.

Appendix A: Detailed experimental procedures

A.1 Bamboo fiber preparation

- Selection and Cleaning: Bamboo stems were selected, cleaned, and cut into 70 cm lengths.
- Alkali Treatment: Fibers were immersed in NaOH solutions (3, 5, 7, 11%) for varying durations (45, 90, 135 minutes), followed by rinsing and drying at 60°C for 24 hours.
- Flame Retardant (FR) Treatment: Optimal NaOH-treated fibers (5%, 45 minutes) were further treated with FR inhibitors for 30, 45, and 60 minutes.

A.2 Mechanical testing

- Single Fiber Tensile Test: Conducted per ASTM D3039, with five repetitions per condition.
- Pull-Out Test: Measured interfacial adhesion between fibers and epoxy matrix.
- SEM/FTIR Analysis: Fiber morphology and functional groups were analyzed post-treatment.

A.3 Combustion testing

- UL-94 Standard: Samples (300 × 15 × 1 mm) were exposed to a methane flame (38 mm height) for 12 seconds. Parameters recorded: after-flame time, after-glow time, and char length.

Appendix B: Raw data tables

- Table B.1: Complete mechanical test results for all NaOH and FR treatment variations.
- Table B.2: Full contact angle measurements for wettability analysis.
- Table B.3: Detailed combustion test data (after-flame, after-glow, char length).

Appendix C: Statistical analysis outputs

- ANOVA and Tukey HSD results for mechanical and combustion tests.
Regression models for optimizing NaOH/FR treatment parameters.

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