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RESEARCH ARTICLE

Studies on Lanthanum Strontium Manganite (LSM) Particle Reinforced Polybenzoxazine/Epoxy Hybrid Composites

Satheeshkumar Ellappan[1] | Selvi Mohan[1] | Yuvarani Nadarajan[1] | Devaraju Subramani[2] | Alagar Muthukaruppan[1,2]

[1]Centre for Advanced Materials, PSG College of Arts and Science, Coimbatore, India | [2]Polymer Engineering Laboratory, Department of Chemistry, PSG Institute of Technology and Applied Research, Coimbatore, India

Correspondence: Devaraju Subramani (subudeva@gmail.com) | Alagar Muthukaruppan (muthukaruppanalagar@gmail.com)

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ABSTRACT

In this work, lanthanum strontium manganite (LSM)-reinforced polybenzoxazine (PBZ) and PBZ/epoxy hybrid composites have been developed with enhanced thermal, mechanical, dielectric, and flame-retardant performance. LSM particles was synthesized via a microwave combustion method followed by surface-functionalization using 3-glycidyloxypropyltrimethoxy silane (GPTMS). The functionalized LSM was incorporated into bisphenol-F and aniline based benzoxazine (BF-a) and BF-a/Bisphenol-F epoxy (DGEBF) (50/50) blends followed by thermal curing to obtain composites. An incorporation of LSM improved thermal stability, with the initial degradation temperature (T_5%) of poly(BF-a) increased to 383°C from 346°C and the value of char yield to 59.1% from 42.8%. The heat resistance index (HRI) increased from 205°C to 224°C, while the limiting oxygen index (LOI) increased from 34.6 to 41.1. The value of tensile strength increased up to 116 ± 3 MPa for PBZ/DGEBF composites. Shore D hardness increased from 81 ± 2 to 95 ± 1.5, while the coefficient of thermal expansion decreased from $52.5 \times 10^{-6}°C^{-1}$ to $37.7 \times 10^{-6}°C^{-1}$, indicating enhanced dimensional stability. Dielectric analysis indicated an increase in dielectric constant from 3.50 to 5.61 for poly(BF-a) composites and from 3.90 to 7.35 for PBZ/DGEBF composites at 10 wt% LSM loading, while maintaining low dielectric loss (0.002–0.019).

1 | Introduction

In recent years, polymer composite materials reinforced with inorganic fillers have gained considerable attention owing to their superior mechanical strength, thermal stability, dimensional stability, and multifunctionality compared to those of conventional polymer materials [1–3]. Among various filler materials, ceramic fillers have attracted significant interest due to their high hardness, excellent thermal stability, chemical inertness, and superior electrical insulation capability, which make them potential candidates for advanced applications in various fields [4–6].

As a matrix concern, benzoxazine resins represent an emerging class of high-performance phenolic thermosetting materials synthesized through the Mannich condensation of phenols, primary amines, and formaldehyde. Upon thermal curing, benzoxazine monomers undergo catalyst-free ring-opening polymerization to form highly crosslinked polybenzoxazine (PBZ) networks exhibiting excellent thermal stability, low water absorption, near-zero volumetric shrinkage, superior mechanical strength, and outstanding chemical resistance. Blending PBZ with epoxy resins is an effective strategy to develop hybrid thermosets with improved performance. During curing, phenolic hydroxyl groups generated from benzoxazine ring opening can react with

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epoxy functionalities, producing densely crosslinked networks that improve interfacial compatibility and overall material performance [7–11]. Generally, a 50:50 weight ratio of benzoxazine and epoxy was taken to achieve a balanced combination of the excellent thermal stability, flame retardancy, and low dielectric properties of PBZ with the good processability of epoxy resin. At this composition, both thermosets contribute equally to the formation of a homogeneous co-crosslinked network, leading to enhanced thermal and dielectric performance [12–14].

Various ceramic fillers such as alumina (Al_2O_3) [15], zirconia (ZrO_2) [16], boron nitride (BN) [17], silicon dioxide (SiO_2) [6], and silicon nitride (Si_3N_4) [18, 19] have been widely used in the PBZ-based materials. While these fillers have been reported to improve individual properties, most studies primarily focus on either mechanical or thermal stability. Only limited research has addressed the simultaneous optimization of dielectric behavior, flame retardancy, and dimensional stability in a single PBZ-based system. These fillers significantly improve stiffness, thermal stability, dielectric performance, and durability of PBZ composites. Nevertheless, despite extensive development of ceramic-reinforced PBZ hybrid systems, further research is needed. Furthermore, perovskite-type oxide ceramic materials have unique properties such as excellent thermal stability, chemical durability, semiconducting properties, and structural robustness. Particularly, lanthanum strontium manganite (LSM) is a potential candidate as a reinforcement for matrix materials that are suitable for microelectronics. The partial substitution of Sr^{2+} ions into the La^{3+} position generates a charge imbalance within the perovskite lattice, leading to mixed Mn^{3+}/Mn^{4+} valence states and the formation of oxygen vacancies. The Mn^{3+}/Mn^{4+} ratio governs electrical conductivity; the higher the ratio, the lower the conductivity and the higher the resistivity [20]. Thereby improving the dielectric performance and thermal resistance of polymer composites when employed as a reinforcing filler. Owing to its nano- to submicron-sized and uniform dispersion of particles within thermosetting polymer matrices, it can facilitate efficient stress transfer and promote enhanced dielectric, thermal, and mechanical properties. However, compatibility between inorganic ceramic fillers and organic polymer matrices is a general concern; only pristine LSM often affects dispersion stability and filler-matrix adhesion, which leads to reduced efficiency in composite materials. Hence, surface modification of ceramic fillers is a prerequisite to improve their interfacial adhesion (compatibility) with polymer matrices [21–24]. Despite these promising characteristics, the application of LSM as a functional reinforcing filler in thermosetting polymer composites has received very limited attention, leaving significant opportunities for further research and development [25–28]. The potential of LSM as a multifunctional filler in PBZ matrices remains largely unexplored.

In the present work, LSM particles were synthesized and surface functionalized using 3-glycidyloxypropyltrimethoxysilane (GPTMS) to enhance compatibility with PBZ and PBZ/epoxy matrices. The functionalized LSM was incorporated into neat poly(BF-a) and poly(BF-a)/epoxy (PBZ/DGEBF) (50:50) blend systems at filler loadings of 0, 1, 3, 5, and 10 wt%. The influence of LSM reinforcement on the thermal, mechanical, flame, and dielectric properties of the resulting composites was systematically investigated. To the best of our knowledge, this study represents one of the first comprehensive investigations on LSM-reinforced poly(BF-a) and poly(BF-a)/epoxy (PBZ/DGEBF) hybrid composites, highlighting the novelty and potential of LSM as a multifunctional ceramic reinforcement for high-performance polymer composites.

2 | Materials and Methods

2.1 | Combustion Synthesis of LSM

The $La_{0.8}Sr_{0.2}MnO_3$ (LSM) was synthesized by the microwave combustion method using $La(NO_3)_3{\cdot}6H_2O$, $Sr(NO_3)_2$, $Mn(NO_3)_2{\cdot}xH_2O$. The appropriate amounts of nitrate salts were dissolved in deionized (DI) water and stirred well to obtain a clear solution. Glycine (fuel) was added to the previously obtained solution, and a 0.6 M ratio with nitrates was maintained to achieve better combustion [29, 30]. Further, the temperature of the solution was raised to 80°C and stirred continuously using a magnetic stirrer until a gel was formed. The obtained gel was then placed in a microwave oven (ONIDA, Power Barbecue 28, 900 W, 2.4 GHz) for 15 min. to obtain a combusted foam-like product. The resulting foam mass was then ground well into fine powder, which was subsequently subjected to a two-step calcination process. Initially, the powder was calcined at 600°C, followed by post-calcination at 900°C for 5 h with a heating rate of 5°C min^{-1}. After the calcination process, the powder was thoroughly ground to reduce agglomeration and used for further processing.

2.2 | Preparation of GPTMS-Functionalized LSM

The LSM surface was functionalized with GPTMS to improve interfacial compatibility between LSM and poly(BF-a) as well as poly(BF-a)/DGEBF matrices [31, 32]. GPTMS was mixed with an aqueous ethanol/water solution (95:5 v/v) and ultrasonicated for 15–30 min. The ultrasonication was then continued for an additional 1 h to induce hydrolysis of the methoxy groups of GPTMS. Subsequently, 10 g of LSM was dispersed in the GPTMS solution obtained from the previous step, and ultrasonication was continued for 2 h to ensure uniform dispersion. The resulting mixture was refluxed at 80°C for 24 h to promote covalent grafting of GPTMS onto the LSM surface. After completion of the reaction, the surface modified LSM was filtered, washed several times with deionized water, ethanol, and hexane to remove unreacted silane precursor. Finally, the product was filtered and dried in a hot air oven to obtain GPTMS-functionalized LSM.

2.3 | Synthesis of Benzoxazine Monomer (BF-a)

The benzoxazine monomer (BF-a) was synthesized using bisphenol-F and aniline by following a literature procedure [33]. A mixture of 0.4 mol paraformaldehyde and 0.1 mol bisphenol-F was stirred vigorously. Then, 0.2 mol aniline was added to the mixture in a dropwise manner. The reaction mixture was maintained at 110°C under melt conditions. Stirring was continued for more than 2 h, resulting in the formation of a homogeneous solution. The reaction was found to be complete within 2 h, and the progress of the reaction was monitored using thin-layer

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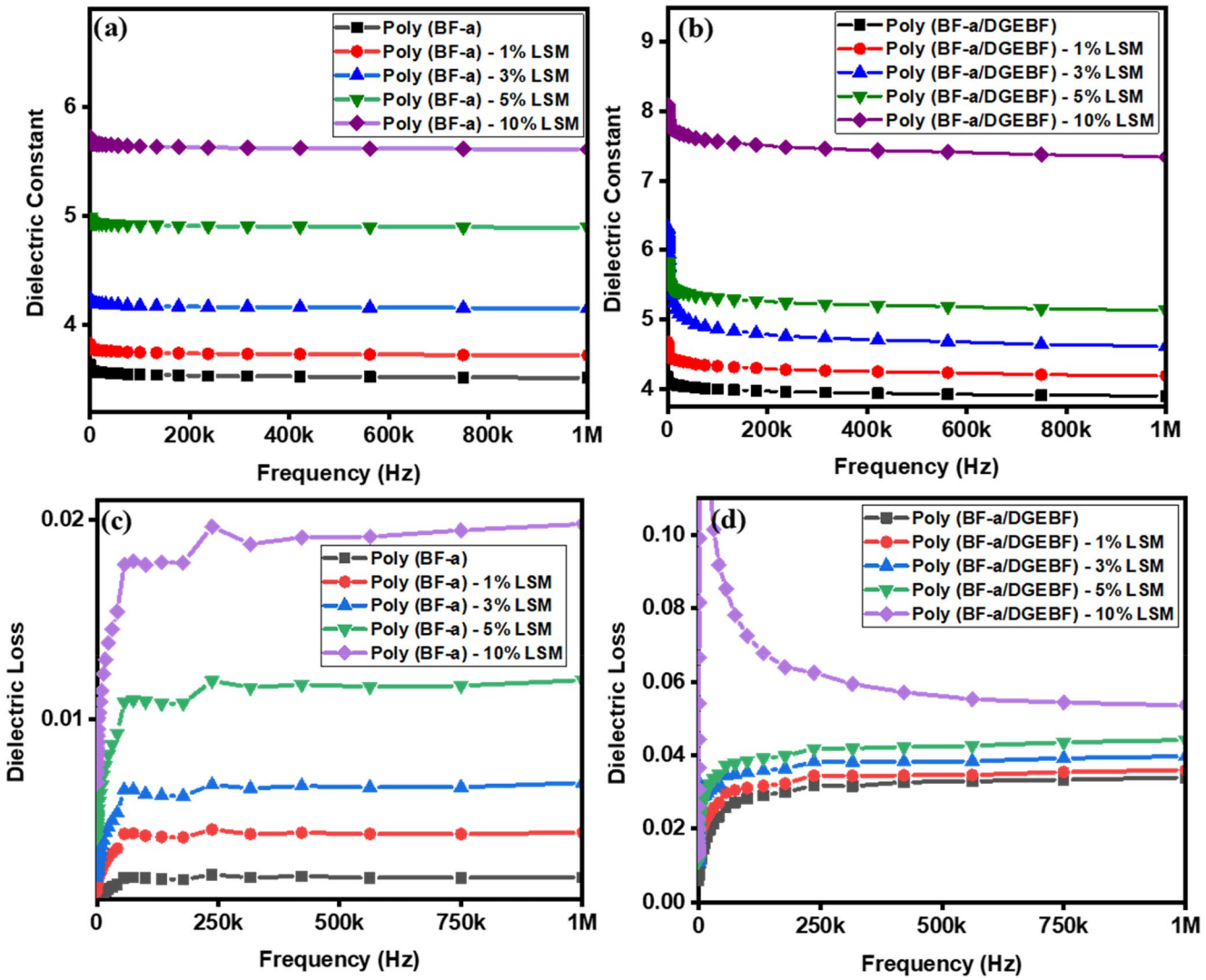


FIGURE 8 | Dielectric properties of poly(BF-a), poly(BF-a/EP), and LSM reinforced polymer composites. [Color figure can be viewed at wileyonlinelibrary.com]

TABLE 4 | Dielectric properties of poly(BF-a), poly(BF-a/EP), and LSM reinforced polymer composites.

	Dielectric constant (k)		Dielectric loss (tanθ)	
Sample	**Poly(BF-a)**	**Poly(BF-a/DGEBF)**	**Poly(BF-a)**	**Poly(BF-a/DGEBF)**
Neat matrix	3.50	3.90	0.0020	0.0343
1% LSM	3.72	4.19	0.0043	0.0356
3% LSM	4.15	4.61	0.0068	0.0392
5% LSM	4.89	5.13	0.0116	0.0473
10% LSM	5.61	7.35	0.0198	0.0532

does not significantly deteriorate the insulation characteristics of the composites.

Overall, GPTMS-functionalized LSM effectively increases the dielectric permittivity of PBZ and poly(BF-a/DGEBF) composites due to enhanced interfacial polarization, while maintaining low dielectric loss. The absence of conductive pathways confirms that the composites preserve good electrical insulating behavior [58]. The balanced combination of improved dielectric, thermal, and mechanical properties makes these materials suitable for advanced insulating and electronic applications.

4 | Conclusion

In this work, GPTMS-functionalized lanthanum strontium manganite ($La_{0.8}Sr_{0.2}MnO_3$, LSM)-reinforced polybenzoxazine (poly(BF-a)) and poly(BF-a/DGEBF) hybrid thermoset composites

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were successfully developed and systematically characterized. LSM synthesized via the microwave combustion method exhibited a phase-pure rhombohedral perovskite structure with good compositional homogeneity. GPTMS functionalization improved interfacial compatibility between the inorganic filler and polymer matrices, enabling uniform dispersion and effective stress transfer within the hybrid networks. Thermal analysis demonstrated improved high-temperature stability and char formation. The initial degradation temperature ($Td_{5\%}$) of poly(BF-a) increased from 346°C to 383°C, while char yield increased from 42.8% to 59.1% at 10 wt% LSM loading. The heat resistance index increased from 205°C to 224°C, and the limiting oxygen index increased from 34.6 to 41.1. All LSM-filled systems achieved UL-94 V-0 classification, confirming excellent flame-retardant behavior. Mechanical performance improved significantly with increasing LSM content. Tensile strength increased from 55 ± 2 MPa to 98 ± 2 MPa for poly(BF-a) and from 70 ± 3 MPa to 116 ± 3 MPa for PBZ/DGEBF composites at 10 wt% loading. Shore D hardness increased up to 96.5, while the coefficient of thermal expansion decreased from 52.5×10^{-6} °C^{-1} to 37.7×10^{-6} °C^{-1}, indicating enhanced dimensional stability. Dielectric analysis showed an increase in dielectric constant from 3.50 to 5.61 for poly(BF-a) composites and from 3.90 to 7.35 for PBZ/DGEBF composites, while maintaining low dielectric loss (0.002–0.053), confirming good insulating behavior. Overall, GPTMS-functionalized LSM effectively enhances thermal, mechanical, dielectric, and flame-retardant performance, making these composites promising materials for heat resistant and electrical insulation applications.

Author Contributions

Satheeshkumar Ellappan: conceptualization, investigation, writing – original draft, formal analysis, data curation, software. **Selvi Mohan:** methodology, conceptualization, formal analysis, data curation, investigation, writing – original draft. **Yuvarani Nadarajan:** formal analysis, data curation, software. **Devaraju Subramani:** validation, resources, writing – review and editing, methodology, project administration. **Alagar Muthukaruppan:** project administration, resources, writing – review and editing, validation, funding acquisition.

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The authors have nothing to report.

Conflicts of Interest

The authors declare no conflicts of interest.

Data Availability Statement

The data that support the findings of this study are available from the corresponding author upon reasonable request.

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Supporting Information

Additional supporting information can be found online in the Supporting Information section. **Figure S1:** Thermogravimetric analysis of unfunctionalized and functionalized LSM. **Figure S2:** EDX profile of functionalized LSM. **Figure S3:** FT-IR spectra of BF-a, DGEBF, GPTMS functionalised LSM reinforced Poly(BF-a), and GPTMS functionalised LSM reinforced poly(BF-a/DGEBF) composites. **Figure S4:** SEM fracture images of poly(BF-a)-10% LSM and poly(BF-a/DGEBF)-10% LSM composites. **Figure S5:** DTG thermogram of (a) poly(BF-a) and LSM (1%, 3%, 5%, and 10%) composites. (b) Poly(BF-a/DGEBF) and LSM (1%, 3%, 5%, and 10%) composites. **Figure S6:** UL-94 test for poly(BF-a), poly(BF-a/EP) and GPTMS functionalised LSM reinforced polymer composites (a) 30 s with flame (b) after 5 s without flame (c) after 30 s without flame.

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