

## RESEARCH ARTICLE

# Thermal, Physical, and Electrical Characterization of Millettia Pinnata Leaf Powder, Alumina, Glass Sheet, Boron Nitride Reinforced Epoxy-based Hybrid Composites: An Experimental Scrutiny

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**Abstract: Aims and objectives:** The aim of this study was to fabricate epoxy resin-based hybrid composites reinforced with biodegradable Millettia pinnata leaf powder in conjunction with glass sheets, boron nitride, and alumina for the production of printed circuit boards.

**Method:** For this application, various thermal, physical, and electrical tests were conducted by the authors. The thermal test results showed that the alumina-based epoxy hybrid composite has more thermal stability than the neat epoxy. Moreover, upon adding BN/Alumina, the flame retarding properties of the epoxy hybrid composites improved. We also observed that with the increase in the content of BN and alumina, the thermal conductivity of the hybrid composite was enhanced. From the water absorption tests, the hybrid composite with 6g BN showed the least amount of water consumption. Particularly, adding BN and leaf powder from 2 to 6 g gave better results for the decrease in water absorption, as compared to adding alumina in the epoxy-based hybrid composite.

**Result:** Lastly, from the electric tests, we observed that with the increase in frequencies, the dielectric constant of the hybrid composite decreases. At a lower frequency range, the hybrid composite having 2g of BN and millettia pinnata leaf powder shows the lowest dielectric constant, whereas, at a higher frequency range, 2g of alumina and millettia pinnata leaf powder shows the lowest dielectric constant.

**Conclusion:** We predict that the results reported in this investigation will aid in accelerating the engineering applications of epoxy resin-based hybrid composite materials and help patent the material compositions for specific purposes.

**Keywords:** Composite, epoxy, alumina, boron nitride, millettia pinnata leaf powder.

## 1. INTRODUCTION

As per the reports of the UN E-Waste Coalition, the total global electronic waste (e-waste) costs about 62.5 billion US dollars, which is comparatively more than the GDP of several countries [1]. However, only 20% of the total e-waste is recycled properly, and the remaining 80% is either dumped or recycled casually, which results in the release of carcinogenic substances [2]. By 2050, the e-waste forecast is expected to be around 120 million tons [3]; specifically, by the

end of 2040, the total carbon emissions for the production of printed circuit boards (PCBs) will reach 14% of the total emissions. In general, properties, such as low dielectric constant, high thermal expansion, low flammability, low thermal conductivity, and low electrical conductivity play an important role in the development and fabrication of PCBs. In the present investigation, a noble approach for the development of PCBs has been made using an epoxy resin-based hybrid composite reinforced with millettia pinnate leaf powder, alumina, glass sheet, and boron nitride. Further, emphasis has been put on different characterizations, such as the behavior of fabricated composite material on thermal stability through thermo-gravimetric analysis, insulation characterization through the dielectric test, flame retarding analysis,

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the thermal conductivity test, and the physical behaviour via the water absorption test.

In the past, applying different novel base materials (specifically composite materials [4-9]) for the development of sustainable and eco-friendly PCBs gained interest among many researchers. In 2008, Kosbar *et al.* [10] used lignin fiber in the development of printing wiring boards (PWB). The fabricated composites passed through the FR4 standards, and their life-cycle assessment approach showed that the developed PWB consumes 40% less energy in comparison to the conventional [10]. FR4 is a grade provided by the NEMA (National Electrical Manufacturing Association) and relates to the laminates that are flame retardants. FR4 commonly signifies the composite materials composed of woven glass fiber-reinforced epoxy composite [11]. In 2016, Deng *et al.* [12] fabricated flax fiber-reinforced epoxidized linseed oil laminate for the development of PCB substrates and performed their life cycle assessment as well. Cradle-to-grave life cycle assessment showed that the fabricated composite focused on resource optimization and reduced environmental impact. Recently, in 2022, Bharath *et al.* [13] incorporated rice husk as a reinforcing material in epoxy resin for the synthesis of PCB. To verify the suitability of the PCB application, various aspects of the fabricated composite material were evaluated, including the dielectric performance, flammability, moisture absorption characteristics, and various thermo-mechanical properties. Some researchers are also utilizing polylactic acid (PLA) for creating electronic devices on the PLA substrates. Currently, PLA is primarily used in food packaging, either in the form of a liquid bottle or a thin film [14, 15]. However, due to its low glass transition temperature, PLA faces challenges in manufacturing electronics products that typically require exposure to higher temperatures [16-18]. Lincoln *et al.* [19] fabricated a PCB that utilized a thermoset matrix consisting of deoxidized linseed oil/melamine polyphosphate and a woven flax fiber as the reinforcement material. The fabricated laminates successfully passed the thermal, mechanical, and electrical tests using the IPC 4101A/24 standards, with the exception of wet conditioning conditions. To enhance the moisture resistance of the flax fibers, sodium hydroxide and octa-decyltrichloro silane were employed. This upgraded design of bio epoxy-flax PCB presents a viable alternative to the current PCBs. Kosbar *et al.* [10] also created a prototype of a PCB using modified polymer laminates reinforced with glass fiber. Their findings revealed that the energy consumption of the bio-based resin was approximately 40% lower as compared to the non bio-based resin. In a similar work, Nägele *et al.* [20] developed a PCB laminate utilizing a thermoplastic polymer that incorporated lignin. They further confirmed the viability of these environmentally friendly composites for PCB-related applications. The rapid advancement of electronic products has resulted in a significant increase in the accumulation of waste-printed circuit boards (WPCBs). These WPCBs consist of both defective products used in the manufacturing process and scrapped products [21, 22].

Consequently, the proper management and treatment of WPCBs have become an urgent matter. WPCBs are com-

posed of approximately 30% metallic elements, such as copper, iron, nickel, antimony, lead, and gold, while the remaining 70% is made up of non-metallic components like thermosetting resins and glass fibers [22-24]. Currently, extensive research works are being conducted on the separation and recovery of valuable metals from WPCBs due to their high value and purity. However, the non-metallic components in WPCBs are often disposed of through incineration or landfilling, resulting in significant resource wastage and environmental damage [23-25]. Thus, the paper provides insights into the effects of different hybrid fillers on the electrical, thermal, and mechanical properties of glass epoxy composites, which can contribute to the development of improved composite materials for various industrial applications.

## 2. MATERIALS AND METHODS

### 2.1. Materials

The glass sheets, Boron Nitride (BN), and alumina used in this study were sourced from the Vimal Mass Finishing Private Limited, a factory located in Peenya, Bangalore, India. These glass sheets varied in thickness from 1 to 2 mm and had a density ranging from approximately 2.5 to 2.8 g/cm<sup>3</sup>. For this investigation, Lapox L-12 resin and K-6 hardener were employed [26], both of which were provided by Yuje Marketing Limited, based in Bangalore, India.

### 2.2. Fabrication of Epoxy-based Composite Material

The hybrid composite was fabricated using a straightforward and simple hand lay-up technique [27, 28], as this approach requires minimal infrastructure [29, 30]. To prevent the polymer from adhering to the surface of the mold, a releasing gel was applied with a brush to coat the base of the mold. Subsequently, 300 x 300 x 5 mm<sup>3</sup> sized glass sheets (having a density of 2.6 g/cc) were positioned onto the surface of the mold. Next, the thermosetting polymer, in liquid form, was meticulously mixed with the required amount of hardener and other specified reinforcements, as detailed in Table 1. The resulting paste was poured onto the surface of the mold to ensure that the glass sheets were already in place. A brush was also used to evenly distribute the polymer across the whole surface, and a roller was then applied with gentle pressure to eliminate any air pockets and excess polymer. The final step involved subjecting the composite to pressure at a specific temperature for curing. Following the curing process, the mold was opened, and the composite product was carefully removed for further treatment. The samples obtained from the mold were allowed to cool to room temperature and subsequently cut in accordance with ASTM standards for the characterization.

### 2.3. Thermogravimetric Analysis Test

Thermogravimetric analysis (TGA) is a widely used technique to determine the thermal stability and composition of materials as a function of temperature [31]. TGA is also used to evaluate the composition of a substance by measur-

**Table 1. Composite samples designation and description.**

S. No.	Sample Designation	Sample Description
1.	0% GF / Control	Epoxy + hardener
2.	2 g BN	Epoxy + hardener + 2 g boron nitride + 2 g leaf powder + 15 glass sheets 6g BN-
3.	6 g BN	Epoxy + hardener + 6 g boron nitride + 6 g leaf powder + 15 glass sheets
4.	2 g AL	Epoxy + hardener + 2 g alumina + 2 g leaf powder + 15 glass sheets
5.	6 g AL	Epoxy + hardener + 6 g alumina + 6 g leaf powder + 15 glass sheets

ing changes in its weight as it is heated [32]. TGA generates various data points based on mass, temperature, and time. In this study, we conducted TGA on synthesized powdered composite materials. The analysis was performed in a nitrogen atmosphere using a PerkinElmer thermogravimetric analyzer. TGA offers a precise representation of how the materials respond to heat and decompose, expressed as a percentage of weight loss relative to temperature. Throughout our investigation, we maintained a constant heating rate of 10°C/min, and the temperature range spanned from room temperature to 600 °C.

#### 2.4. Water Absorption Test

A water absorption test for epoxy-based composite materials is typically performed to assess their resistance to water and/or moisture [33-35]. The water absorption tests for Alumina/Boron nitride reinforced epoxy composites were conducted in accordance with the ASTM 570 standard, involving immersion in distilled water at room temperature. Samples were periodically removed, and after carefully wiping the surface to remove excess water, they were promptly weighed using a precise balance machine to determine the amount of absorbed water. The specimens were weighed at specific time intervals between 0.5 to 24 hours.

To calculate the water absorption, the percentage weight gain of the samples was determined at various time points using the following formula [36, 37]:

$$\text{Water absorption (\%)} = \frac{(W_2 - W_1)}{W_1} \times 100$$

where  $W_1$  and  $W_2$  represent the weight of the dry samples and wet (absorbed water) samples, respectively.

#### 2.5. Thermal Conductivity Test

Thermal conductivity tests were conducted following the ASTM E-1530 standard. Here, the sample specimen was placed between two polished surfaces, both maintained at different temperatures. The lower surface was an integral component of a precisely calibrated heat flow transducer. Heat flowed from the upper surface, traversed through the sample, and reached the lower surface, establishing an axial temperature gradient within this setup. Once thermal equilibrium was achieved, the temperature difference across the sample was meticulously measured, along with the data from the heat flow transducer. These recorded values, coupled with the thickness of the sample, were subsequently employed to compute the

thermal conductivity of the material. To accurately gauge the temperature drop across the sample, temperature sensors were employed within the highly conductive metal surface layers situated on either side of the specimen.

#### 2.6. Dielectric Constant Test

The dielectric constant test, often referred to as the "high potential" test, is performed to verify the effective functioning of insulation in a component. In this research, specially prepared fat samples were subjected to a brief-duration examination. During the testing process, basic test electrodes were employed to apply a consistent voltage at a frequency of 60 Hz across opposing sides of the samples. A rate of 500 volts per second (V/s) was utilized in this investigation, and the average breakdown time fell within the range of 10 to 20 seconds.

#### 2.7. Flammability Test

A standard UL-94 method was employed to assess the flammability of the fabricated composites. The specimens, with dimensions measuring  $125 \times 13 \times 1.5 \text{ mm}^3$ , were subjected to testing using a laboratory burner. This burner, featuring a tube length of  $100 \pm 10 \text{ mm}$  and an interior diameter of  $9.5 \pm 0.3 \text{ mm}$ , was equipped with various components, including a burner mounting fixture, ring stands, timing devices, wire gauze, a supply of methane gas, and a measurement scale. The burner was then precisely adjusted to produce a consistent  $20 \pm 1 \text{ mm}$  blue flame. The flammability test was carried out as follows:

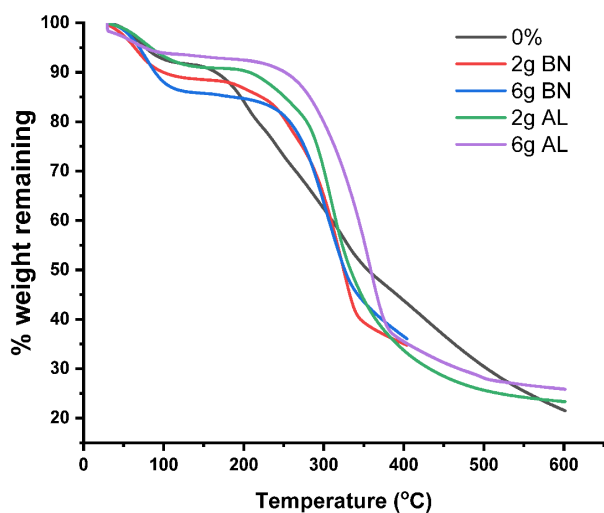
- The flame was applied to the specimen, positioned 10 mm below its bottom end, and maintained for a duration of 10 seconds.
- Subsequently, the burner was withdrawn from the specimen, and the after-flame time was recorded.
- Finally, once the after-flaming of the specimen ceased, the burner was once again positioned below the specimen for an additional 10 seconds, and the after-flame time was noted again.

### 3. RESULTS AND DISCUSSION

#### 3.1. Thermogravimetric Analysis Test

The thermal properties of fabricated hybrid composites were investigated using the differential TGA. Fig. (1) shows

the TGA thermographs of epoxy-based hybrid composites reinforced with millettia pinnata leaf powder, alumina, glass sheet, and boron nitride. According to this thermogram, these epoxy-based hybrid composites decompose or lose mass significantly in three different temperature ranges: 60-150°C, 150-320°C, and 320-560°C. The starting temperature range causes a mass loss of between 0.30 and 2.5 percent due to material evaporation or dehydration. Tsuchiya and Sumi [38] stated that the mass loss in the second range is caused by the backbone chain's thermal degradation, which changes the composite into aldehydes, ketones, and ethanol and releases gaseous byproducts like carbon dioxide (CO<sub>2</sub>) and carbon monoxide (CO). In this range, the epoxy-based hybrid composite has a lower mass loss of 30%, whereas the 0% control epoxy composite has a mass loss of 75%. The formation of carbons and hydrocarbons as a result of the bond breakage characterizes the third temperature range. The structural alteration in epoxy-based hybrid composites results in a significant simultaneous mass loss, as compared to the 0% control epoxy composite. There is a shift in the peak location of 121°C; specifically, the peak in the temperature zone shifts from 170°C (0% control epoxy composite) to 291°C (6g AL hybrid epoxy composite). This movement suggests that the 6g AL epoxy hybrid composite has more thermal stability than the 0% control composite.

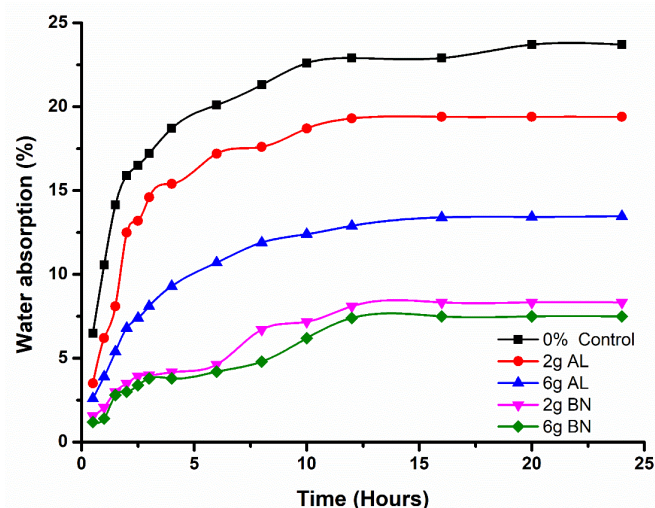


**Fig. (1).** TGA graph of the different hybrid composite materials. (A higher resolution / colour version of this figure is available in the electronic copy of the article).

### 3.2. Water Absorption Test

Fig. (2) depicts the water absorption characteristics of hybrid composites made of epoxy and reinforced with millettia pinnata leaf powder, alumina, glass sheet, and BN. The type and quantity of reinforcement materials, the manufacturing method, and the unique formulation of epoxy resins all affect how much water is absorbed by the composites made of epoxy. Epoxy resins are typically thought to absorb less water than other polymers, but this might change depending on the conditions and other elements added to the resin domain.

The hydrophilic properties of epoxy-based hybrid composite and the vacancies in the composite are responsible for a large portion of the prominent water absorption that occurs in the first 10 hours. Then, over the next 15 hours, the absorption rate decreases as most of the gaps fill up. Water absorption levels stabilize between 10 to 25 hours, suggesting a steady rate for a particular weight ratio of the hybrid composite. It is noteworthy that the 0% control composition shows the most water absorption, while the hybrid composite with 6g BN shows the least amount of water consumption. The addition of BN and leaf powder from 2 to 6g weight gives better results for the decrease in water absorption as compared to the addition of alumina in the epoxy-based hybrid composite.



**Fig. (2).** Water absorption test results of the different hybrid composite materials. (A higher resolution / colour version of this figure is available in the electronic copy of the article).

### 3.3. Thermal Conductivity Test

Table 2 tabulates the information about the thermal conductivity of different composite samples. The control sample consists of epoxy/hardener laminate and possesses a thermal conductivity of approximately 0.274 W/mK, *i.e.*, it is a relatively poor conductor of heat (as it has a lower thermal conductivity). In contrast, the hybrid composite with 2 g of BN and millettia pinnata leaf powder, in addition to the control composition, has a higher thermal conductivity of around 1.074 W/mK. A hybrid composite in which the content of BN and leaf powder increases to 6 g further aids in improving the thermal conductivity to a value of 2.191 W/mK. A similar increasing trend is also seen in the case of alumina-reinforced composite. The hybrid composite, which has 2 g of alumina and leaf powder, has a thermal conductivity of 1.155 W/mK; whereas hybrid composite having 6 g of alumina and leaf powder has a thermal conductivity of 1.708 W/mK.

Thus, the addition of BN as a reinforcing filler significantly enhances the thermal conductivity of the epoxy composite, with higher BN content leading to even better thermal conductivity. Alumina also improves thermal conductivity

**Table 2. Results from the thermal conductivity test.**

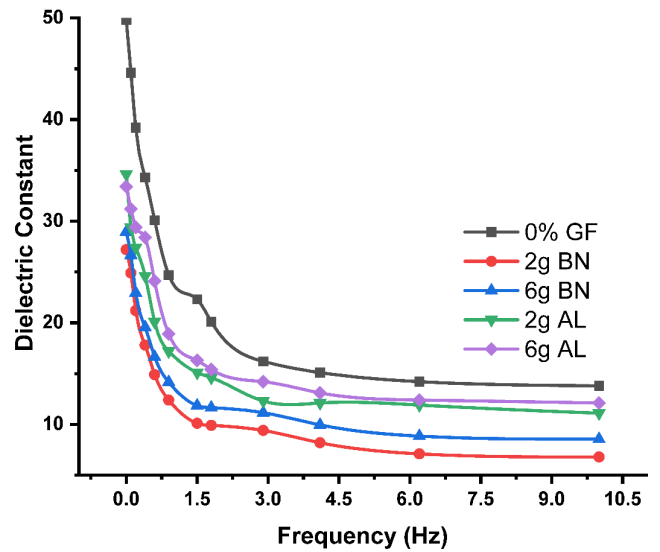
S. No.	Sample Designation	Mean Temp. (°C)	Delta Temp. (°C)	Thermal Conductivity (W/m * K)
1.	0% GF / Control	35	10	0.274 ± 0.02
2.	2g BN	35	10	1.074 ± 0.09
3.	6g BN	35	10	2.191 ± 0.17
4.	2g AL	35	10	1.155 ± 0.09
5.	6g AL	35	10	1.708 ± 0.12

when compared to neat epoxy, although it is not as effective as BN in enhancing heat conduction in these particular composites. Hence, the choice of filler material and its concentration can have a substantial impact on the thermal properties of composite materials.

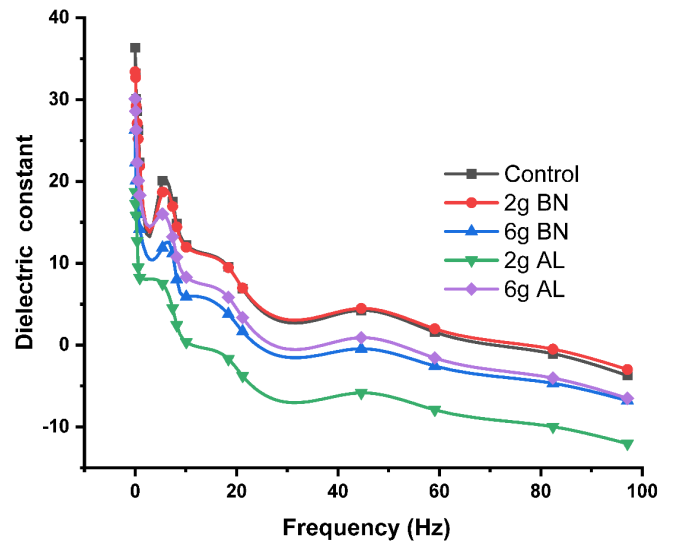
**3.4. Dielectric Constant Test**

The results obtained from the dielectric constant tests are shown in Figs. (3 and 4). Herein, the dielectric constant exhibits a declining behavior as the frequency rises, which aligns with the anticipated pattern observed in most materials. With an increase in the frequency, charges become increasingly disordered and begin to oscillate out of sync with the applied voltage. This phenomenon leads to a reduction in the dielectric constant, contributing to the decrease in its value. Moreover, dispersion can also be attributed to the dielectric relaxation. When the frequency increases, the alignment of polar molecules in the direction of the applied field is disrupted. The charge conveyance in a polymer determines its dielectric properties. Conversely, at higher frequencies, the time available is shorter and insufficient for the molecules to align themselves with the external electric field. The value of the dielectric constant is determined by the combined effect of four types of polarization: electronic, ionic, interfacial, and orientational. However, at higher frequencies, the dielectric constant is primarily influenced by ionic and electronic polarization. These two types of polarization can easily align with the rotation of the external electric field, resulting in a reduction in polarization and subsequently a decrease in the dielectric constant [39].

When subjected to an external electric field, the arrangement of the electrical dipoles is influenced, causing them to align parallel to the electric field. This alignment generates an electric field that opposes the original electric field, resulting in the weakening of the original electric field. Consequently, the electrical insulation constant of the hybrid composite increases. At higher frequencies, hybrid composites act like insulators. This phenomenon can be elucidated by the fact that at the low-frequency range, there is ample time for the dipoles to organize the molecules and orient them parallel to the electric current flowing between the poles [40].



**Fig. (3).** Variations in the dielectric constant test at frequencies between 0 and 10 Hz. (A higher resolution / colour version of this figure is available in the electronic copy of the article).



**Fig. (4).** Result of dielectric constant test at frequencies between 0 and 100 Hz. (A higher resolution / colour version of this figure is available in the electronic copy of the article).

**Table 3. Flammability ratings of the composite materials.**

S. No.	Sample Designation	Time to Self-extinguish After Ignition (s)	Observed Dripping	UL-94 Rating
1	0% GF / Control	< 30	Yes	V2
2	2g BN	< 30	No	V1
3	6g BN	< 30	No	V1
4	2g AL	< 30	No	V1
5	6g AL	< 30	No	V1

### 3.5. Flammability Test

The experimental results of vertical burning of hybrid composites are displayed in Table 3. From the results, it is concluded that with the addition of BN/Alumina, the flame retarding properties of the epoxy hybrid improved. Neat epoxy passed the V2 criteria, which signifies that following the execution of two 10-second combustion trials on the sample, the flame is promptly extinguished within 30 seconds. Moreover, it possesses the capability to ignite the cotton wool positioned at a distance of 30 cm below it. However, with the addition of BN/Alumina, the hybrid composite passed the V1 criteria, which signifies that following the execution of two 10-second burning tests on the sample, the flame is promptly extinguished within a time frame of 30 seconds. Moreover, it is noteworthy that the cotton wool situated 30 cm below remains unignited, indicating the effectiveness of the flame suppression.

### CONCLUSION

The fabrication of epoxy-based hybrid composites reinforced with BN/Alumina was performed using the application for the PCBs. Different thermal and electrical tests were conducted with the following key findings:

1. The peak in the temperature zone shifted from 170°C (0% control epoxy composite) to 291°C (6 g AL hybrid epoxy composite). This movement suggests that the 6 g AL epoxy hybrid composite has more thermal stability than the 0% control composite.
2. Hybrid composite with 6 g BN showed the least amount of water consumption. The addition of BN and leaf powder from 2 to 6 g weight gave better results for the decrease in water absorption, as compared to the addition of alumina in the epoxy-based hybrid composite.
3. With the increase in content of BN and alumina, the thermal conductivity of the hybrid composite was enhanced.
4. With the increase in frequencies, the dielectric constant of the hybrid composite was decreased. At a lower frequency range, the hybrid composite having 2 g of BN and milletia pinnata leaf powder showed the lowest dielectric constant, whereas, at a higher frequency range, 2 g of alumina and milletia pinnata leaf powder showed the lowest dielectric constant.

5. With the addition of BN/Alumina, flame retarding properties of the epoxy hybrid composites improved, and the rating of the hybrid composites changed from V2 to V1.

### AUTHORS' CONTRIBUTIONS

All the authors equally contributed to conceptualization, methodology, writing, reviewing, and editing.

### AVAILABILITY OF DATA AND MATERIALS

The data and supportive information are available within the article.

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### CONFLICT OF INTEREST

The authors declare no conflict of interest, financial or otherwise.

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Declared none.

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