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ORIGINAL ARTICLE

Corncob bio-waste and boron nitride particles reinforced epoxy-based composites for lightweight applications: fabrication and characterization

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Abstract

In the present investigation, corncob waste obtained from local agricultural fields in conjunction with boron nitride particles has been used as the reinforcement material to fabricate epoxy–based composite. The purpose of this research work is to use bio-waste in composite fabrication due to its low cost, non-abrasive, and eco-friendly nature. Aim of this investigation is to provide effective measure for waste disposal and enhance the flexural and tensile strength of the epoxy-based composites to increase its application in day to day life. Fabricated composite material is been characterized through tensile strength, flexural strength, the water up-take test, and fire resistance test. Nine different laminates are prepared with different proportions of corncob and epoxy through the hand layup process. Natural fibers may play important role in developing bio-degradable composite to resolve the current ecological and environmental problems. This report shows that natural water reinforcement also possesses good mechanical properties, and then, fiber composite can also be used in various engineering applications. Results shows that 3.5/3.5 boron nitride and corncob ratio (wt./wt.) composition possess the best flexural strength of about 36.7 N/mm^2, and further characterization has been done only to this composition composite. 3.5/3.5 boron nitride and corncob ratio (wt./wt.) composite shows 2 to 3 times improvement in tensile strength as compared to the neat epoxy resin. Moreover, thermal decomposition of laminate occurs in three phases corresponding to 34%, 16%, and 27% of weight loss.

Keywords Epoxy · Corncob · Composite · Bio-waste · Fabrication

1 Introduction

Due to environmental issues majorly disposal of agricultural bio-wastes such as sawdust, livestock waste crop residues and weeds and forest waste become challenges in front of many countries. To overcome this issue, agricultural bio-wastes are used as filler materials for prepared polymer-based composites by various researchers [1, 2]. Recently in 2022, Partalo et al. [3] used the livestock waste, i.e., sheep wool fibers, as filler material for designing earth-based building material. Sheep wool fibers improved the ductility, strength, toughness, and impact resistance of earth-based building material emphasizing the environmental sustainability. Other than bio-waste, some researchers also used plastic waste as filler material for sustainable development. Sharhan et al. [4] fabricated the polymethyl methacrylate (PMMA)–based composites reinforced with polypropylene and polyacrylonitrile fibers for dental applications. Compressive strength and hardness of PMMA-based composites significantly improved with increase in weight percentage of polypropylene and polyacrylonitrile fibers. Nowadays, application of polymer composites reinforced with agricultural waste also increasing in artificial limbs and denture-based materials. For instance, Fouly et al. [5] studied the sustainability of artificial hip joint prepared from 3D printing. Date pits were used as filler material in PLA matrix. Reinforcement results in enhancement of compressive strength and stiffness in joint whereas elongation and toughness

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decreases. Fouly et al. [6] reinforced with corn cobs and miswak particles to improve the mechanical properties of PMMA. At 8 wt.% of corn cob, the Young's modulus and hardness of PMMA-based composite are the highest, and wear resistance is lowest at all sliding distance against PMMA and steel surface.

Polymer plays an important role in everyday life, and life without polymers is impossible to exist because they provide our daily essentials from clothes to electronics and play a vital role in life processes. For several decades, plastic waste pollution becomes a major challenge for the government, due to which many restrictions have been imposed by the government on the use of toxic plastic (obtained from the petroleum products). For sustainable development, industries have been moving towards eco-friendly products by developing bio-plastic. To fabricate bio-plastics, agriculture-based waste materials play an important role. Bagasse, which is the dry fibrous part of sugarcane, corncob, jute, sisal, hemp, flax, giant cane, etc. are been employed to replace inorganic or synthetic fiber [7]. Agricultural waste bio-plastics have good structural properties and almost have applications in every field such as sports industries, packaging films, interior decoration, civil infrastructure, automobiles, and aerospace [8, 9]. The major building block of agriculture waste material is a semicrystalline polysaccharide (i.e., cellulose) which imparts strength to bio-plastic. On the other hand, they contain hydroxyl groups, resulting in the hydrophilic nature of the overall composite which is one of the disadvantages of using agricultural waste. Both thermoplastic and thermosetting resins are been used by researchers as per the requirement. Thermosetting composites are been characterized as ductile materials, with moderate tensile strength and low hardness. On the other hand, thermosetting composites are been characterized as brittle materials, with good young's modulus and hardness. Among different thermosetting resins, epoxy is most widely used matrix material because of its applications in different fields such as coating material, insulators, structures, automobiles, and aerospace. Interfacial bonding between fiber and matrix plays crucial role in evolving mechanical properties such as tensile strength and hardness [10]. Various researchers have used natural fillers to enhance the mechanical properties of epoxy-based composites [11]. Uppal et al. [12] reviewed the mechanical properties of different epoxy-based composite prepared reinforced with different natural resources. Other than natural fillers, different nano-fillers were also used to improve the thermochemical and tribological behavior of epoxy composites [13]. Low cost, high availability, biodegradability, non-toxic nature, etc. make natural fiber more attractive as compared to synthetic fibers such as glass and carbon [14]. They have been used in applications such as automotive interior linings, upholstery stuffing, egg boxes, and electronics packaging [15]. In the past decade, many researchers fabricated composites based on natural fibers which decrease the dependence on non-renewable fossil–based sources [16]. But still, more research has been required towards natural fiber–reinforced composite field to get a better understanding [17]. Search for new materials to overcome environmental issues becomes the challenging part for researchers, and engineers to develop biodegradable materials for automotive, aerospace, construction, marine, and packaging applications [18].

Recently, the increased application of fiber-reinforced composite in various industrial sectors has been centered on durable and renewable composites. This awareness recognizes the complete assortment of plans and materials positioning from synthetic fiber to natural fiber, about satisfying the demands of yielding composites with desired properties [19]. Sanjay et al. [20] fabricated glass/natural fiber–based hybrid composite to study the inter-laminar and impact strength. Jute and kenaf natural fibers were used with glass fiber to make hybrid epoxy-based composites. The vacuum bagging method was employed to fabricate the composite, and the result shows that jute/kenaf/glass fiber hybrid composite almost has the same strength as compared to glass fiber composite; therefore, natural fiber can be used in place of inorganic fibers. Sarmin et al. [21] used the olive fibers in epoxy composite to improve its mechanical properties for structural application. Tensile and flexural strength of epoxy-based composites are enhanced by 27% and 47%, respectively. Leman et al. [22] studied the possibility of substituting natural fiber (sugar palm fiber) with glass fiber due to the toxic nature of glass fiber such as irritation to eye, skin, and respiratory system. To improve the interfacial bonding between fiber and matrix, sugar palm fiber surface treatment had been done using seawater and freshwater as treatment substances. Mechanical characterization was done by measuring the tensile strength of epoxy composite reinforced with different treated palm fibers at 15% by volume. The result shows that 30 days of seawater-treated palm fiber shows maximum tensile strength and can substitute the glass fiber. Sam et al. [23] prepared the aluminum matrix–based hybrid composite. Reinforcement materials used are 3wt.% boron nitrite, 4wt.% boron carbide, and varying wt.% of corn cob particles to improve the tribological behavior of composites. Hardness and impact strength of composites increases with increase in corn cob up to 4 wt.%. Das et al. [24] fabricated wood and polypropylene bio-composite based on activated biochar with an aim to develop cheaper bio-composite. The reason behind using active biochar is the lack of surface functional groups as compared to conventional amounts while maintaining thermal stability and flammability. The aim of using active biochar is to reduce the amount of compatibilizer (maleic anhydride) which increases the overall cost of the composites. The result shows that adding active biochar compatibilizer can be reduced from 3 to 1% and save the overall cost of composite fabrication by about 18%. Mullen et al. [25] studied the potential of waste corncob by developing bio-oil and bio-char obtained by employing fast pyrolysis. Research shows that co-product of biochar has nutrient minerals and residual carbon, which can increase the soil quantity, sequester carbon, and alleviate environmental problems associated with the removal of crop residues through landfilling. Recently in 2022, Mestry et al. [26] used corn cob ash filler in epoxy-based composite to enhance the tribology behavior. Another application of corncob is studied by Qu et al. [27] by developing porous carbon through thermal treatment as the porous carbon obtained from waste corncob has high surface of 1210 m^2/g after ash-removal. Fouly et al. [28] used corn cob as filler material in PLA matrix for preparing hip joints. Corn cob reinforcement results in enhancement of hardness, wear resistance, compressive strength, and stiffness of PLA-based composites. Anukam et al. [29] studied the gasification potential of the corncob. Investigation shows that the gasification of corncob involves some challenges related to ash fouling, slagging, and sintering effects that may be related to the high ash content recorded for corncob. This may contribute to increasing the concentration of inorganic elements under high-temperature gasification conditions, even though EDX analysis showed a reduced concentration of these elements. The low production cost and density of bio-composites make it a promising potential filler for future bio-composites [30].

The present work aims to utilize the leftovers into a value-added product by using corn cob agricultural bio-waste as filler in epoxy-based composite. This helps in preservation of non-renewable resources with sustainable development; and alternative to synthetic harmful materials that pollute the environment. Moreover, the purpose of using corn cob as reinforcement was to improve the mechanical properties such as flexural strength, tensile strength, and hardness of epoxy composite for structural applications.

2 Materials and methods

2.1 Materials

Boron nitride was bought from “Vimal Mass Finishing Private Limited”, which is a factory situated in Peenya, Bangalore, India, and is specialized in carrying out metal surface finishing job service (refer to Fig. 1). Boron nitride is in particle form which have high low thermal expansion, good, easily machinability, high electrical resistance, non-toxicity, thermal conductivity, low dielectric constant, low loss tangent, thermal shock resistance, microwave transparency, non-abrasive and lubricious, chemical inertness, and non-wetting by most molten metals [31]. Boron nitride has similar properties to that of the wonder material “graphene” [32–38].

Corncob was sourced from local agricultural fields near the Presidency University campus in Bangalore, India. Corncob botanically known as Zea may belong to the Poaceae family. Corncob is a central core of maize. It is the part of the ear on which the kernels grow. Corncob shells were firstly cleaned and washed with water to remove soil and unwanted impurities. Then corncob shells were ground in a hammer mill and required size particles were obtained by using appropriate sieves.

In the present work, Lapox L-12 resin and K-6 hardener were used, and this was supplied by Yuje Marketing Ltd., Bangalore, India. Epoxy is the cured end product of epoxy resins, as well as a colloquial name for the epoxide functional group. Epoxy is also a common name for a type of strong adhesive used for sticking things together.

2.2 Fabrication of composite laminate

Hand layup process was employed to fabricate the composite laminates. Corncob particles and boron nitride were used as reinforcement material for the composite preparation.

Fig. 1 Snapshots of **a** boron nitride and **b** corncob particles

Appropriate amounts of corncob particles and boron nitride along with constant epoxy resin were taken in a plastic container and stirred thoroughly to get a homogeneous mixture. After that, the hardener was poured into the solution and the mixture was stirred again for 10 min. The thoroughly mixed mixture was placed in a mold and compressed uniformly. The mixture was kept for 52 h of time as per the above state and after curing composite board was removed from the mold. The thickness of the composition is limited to 7 mm and the mold size is 210 mm × 110 mm, as shown in the Fig. 2. After curing, the edges of the specimen are neatly cut as per the required dimensions. Nine different composite laminates were fabricated as per the process having different weight percentages of corncob and boron nitride as shown in Table 1.

Based on the basic of flexural test optimization of contents of boron nitride and corncob has been done, which comes out to be at 3.5/3.5. Further studies such as tensile test, impact test, FTIR analysis, TGA analysis, and water absorption test have been done only at optimized composition.

2.3 Flexural test

Flexural strength and modulus of the composites were tested in the universal testing machine (UTM) equipped with a 10 kN load cell, as shown in the Fig. 3. Samples were prepared according to ASTM D790 standard. Samples with a length of 60 mm and width of 12 mm and thickness of 5 mm were used for the flexural studies. Crosshead speed for flexural tests was set at 10 mm/min.

2.4 Tensile test

The tensile properties (strength and modulus) of the composites were tested on a Universal Tensile Tester equipped with a 10 kN load cell. Test samples were prepared according to ASTM D 638 standard with samples having dimensions of 60 mm in length and 12 mm wide at the widest section and thickness of 5mm. A crosshead speed of 18 mm/min was used for the tensile tests.

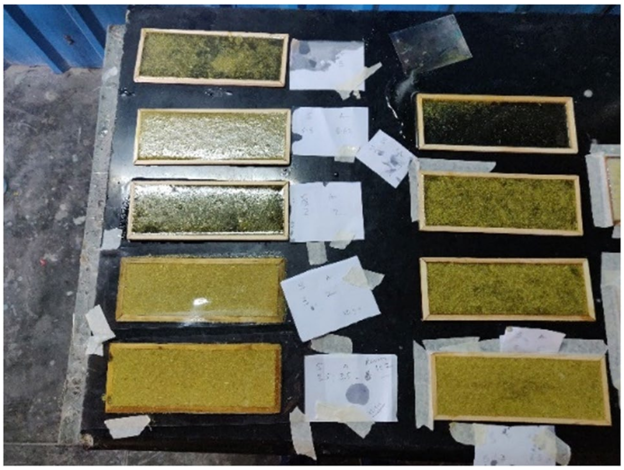

Fig. 2 Fabrication of epoxy-based laminates

Table 1 Nine different types of composites having different boron nitride and corncob amount added into epoxy composite

S. No.	Boron nitride (grams)	Corncob (grams)
1	3.5	3.5
2	5	2
3	2	2
4	2	5
5	3.5	5.62
6	5.62	3.5
7	3.5	1.37
8	1.37	3.5
9	5	5

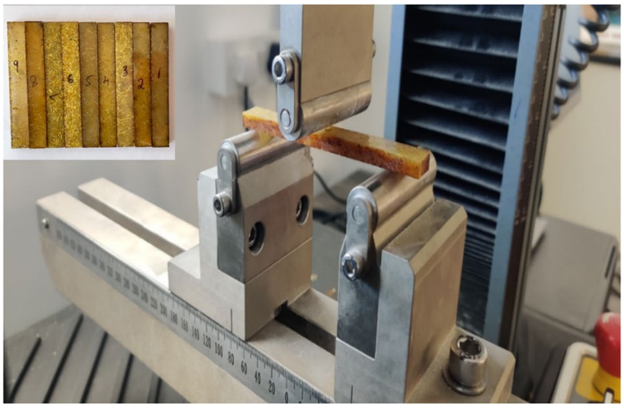

Fig. 3 Flexural testing of specimen

2.5 Vickers microhardness test

The hardness properties of the composites were tested on a micro vickers hardness testing machine equipped with a 2 kN load cell. Test samples were prepared according to ASTM E384 standard with samples having dimensions of 20 mm × 20 mm × 0.5 mm.

2.6 Water absorption

Water absorption of the composites was conducted according to ASTM D570 standards, as shown in the Fig. 4. Samples of dimension 20 × 20 mm were dried in an oven for 24 h at 110°C before the tests. After drying, the samples were weighed and then dipped in a container of distilled water for 24 h at room

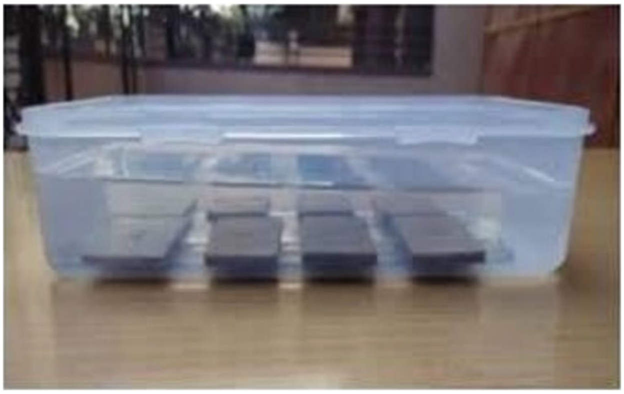

Fig. 4 Set up for the water absorption test

temperature. To study the water absorption behavior over the period of 24 h, the samples were taken out every 2 h until 24 h. Once taken out, the samples were wiped with clean soft tissue and weighed immediately. The percentage of water absorption was given based on the weight difference.

2.7 FTIR analysis

To confirm the presence of epoxy, corncob, and boron nitride Fourier transform infrared spectroscopy (FTIR) was performed. FTIR testing was performed by using ECO-ATR ALPHA, Bruker spectrometer with taking 24 scans at the resolution of 2 cm^{-1}. A spectrum was recorded in transmittance mode varying from 600 to 4000 cm^{-1}.

2.8 TGA analysis

Thermal characterization of fabricated composites has been done through thermo-gravimetric analysis (TGA). TGA is used to determine the degradation of material with respect to material, i.e., weight loss of material at different temperature. This analysis helps in determining the thermal stability of films or temperature working range of the composites. In TGA analysis, specimen mass is monitor as a function of temperature in controlled atmosphere. In the present study, STA7300, HITACHI was employed to study thermal degradation. Sample of weight about 3–6 mg were placed on platinum crucible which is supported by precision balance. The pan is then placed inside the furnace, and analysis was conducted from 30 to 700 °C at heating ramp rate of 10 °C/min. All TGA test was conducted under nitrogen atmosphere by maintaining nitrogen flow rate of 200 ml/min. Differential thermo-gravimetric analysis is another tool for thermal characterization of composite in which rate of change of mass with respect to temperature is measured. It helps in determining the type of reaction, i.e., exothermic or endothermic depending upon the peak. If it is on positive side, it is exothermic, i.e., heat is released in the process; if it is on negative side, it is endothermic, i.e., heat is absorbed during process.

3 Results and discussion

3.1 Flexural test

Flexural strength and modulus of composites having boron nitride/corncob (wt./wt.) have been represented in the Fig. 5. Flexural strength measures the strength of the composite in bending, the result shows that a higher content of boron nitride, and corncob shows lower flexural strength. At higher content of filler material, clustering of corn cob and boron nitride occurred that resulted in overall decrease in the flexural strength of the epoxy-based composites [39]. Moreover, due to this clustering the interfacial adhesion between filler and matrix decreased that further resulted in overall decrement in flexural strength. Maximum flexural strength of about 36.7 N/mm^2 is obtained at 3.5/3.5 boron nitride and corncob ratio (wt./wt.). A similar type of trend has also been reported in Verma et al. [40] where carbon black (CB) obtained from waste tiers is reinforced with epoxy and maximum flexural strength was obtained at 5 wt.% of CB. Hence, for both flexural strength and flexural modulus, no trends were shown. From the flexural test, it was concluded that at 3.5/3.5 boron nitride, and corncob ratio (wt./wt.) best flexural properties were obtained. Thus, further characterization was done only to this particular composition composite. In addition, the authors' main motivation/target was to fabricate a lightweight composite material with best flexural properties.

3.2 Water absorption result

A water uptake test has been performed to determine the interfacial bonding between the corncob and epoxy resin. The result has been presented in the Fig. 6. As corncob is a

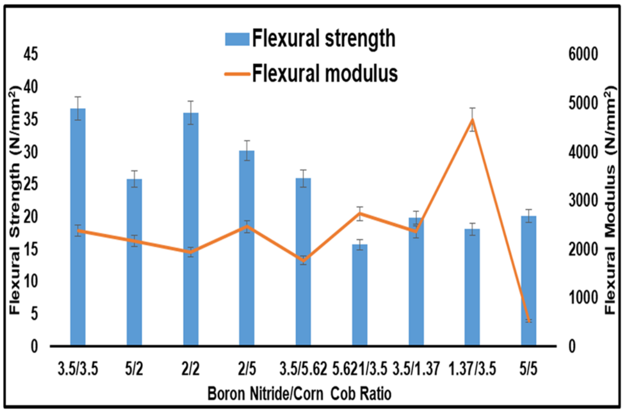

Fig. 5 Flexural properties of an epoxy-based composite at different boron nitride and corncob (wt./wt.) ratio

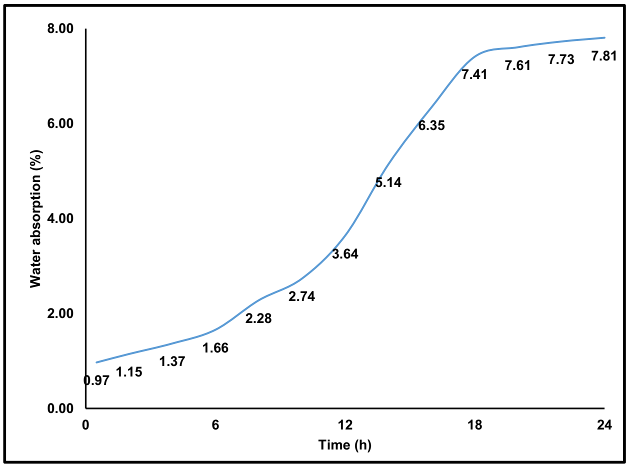

Fig. 6 Water uptake test for 3.5/3.5 boron nitride and corncob ratio (wt./wt.)

bio-waste material (that is hydrophilic in nature), this may result in swelling and micro-cracking in the epoxy matrix. The result shows that the fabricated composite takes a maximum of 7.81 % of water in 24 h. The initial water uptake rate is slower up to 12 h, and then increases to 20 h. After that the curve becomes constant that represents a negligible amount of water uptake. A similar type of result has also been present by Munoz and Garcia-Manrique [41] for flax fiber reinforced bio-epoxy composites.

3.3 FTIR analysis

To investigate the presence of a functional group in the sample FTIR analysis was done. Figure 7 represents the FTIR spectrum of the 3.5/3.5 boron nitride and corncob ratio (wt./wt.) reinforced epoxy composite. From this figure, it is observed that the wider peak from 3421 to 3211cm^{-1} represents the vibration and stretching of a hydroxyl group (-OH), and the peak at 2920 cm^{-1} represents the vibration and stretching of C-H bonds of aromatic rings and CH_2. The sharp peak at 1604 cm^{-1} confirms the presence of aromatic rings (C=C bonds) of epoxy; Vibration and stretching of C-C bonds of an aromatic ring is confirmed by a peak at 1505 cm^{-1}; the peak at 1381 represents the presence of isopropyl group; ether group presence (C-O-C) group is confirmed by the peak at 1026 cm^{-1}, and peak at 823 cm^{-1} showcases the stretching of C-O-C bonds of oxen are a group of epoxies. Moreover, for corncob, following peaks were noted in the FTIR spectroscopy are 3421–3211 cm^{-1}, 2850 cm^{-1}, 1604 cm^{-1}, 1026 cm^{-1}, and 557 cm^{-1}. These peaks confirm the presence of alcohol and phenol (O-H stretching), alkanes (C-H stretching), aromatic compound (C=C stretching), C-O stretching of alcohols, phenols and ethers, and C-H bond bending of aromatic compounds. For boron nitride following peaks absorbed in the samples are 725 cm^{-1}, 1180 cm^{-1}, and 1450 cm. These peaks represent the B-N bond vibration, BH-O bonding, and hexagonal BN structure.

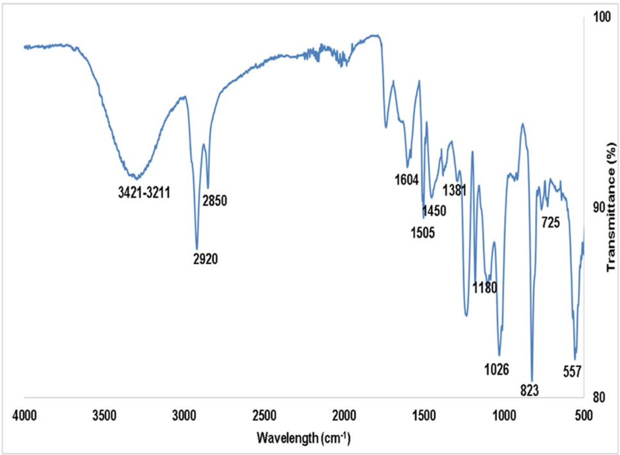

Fig. 7 FTIR spectrum for 3.5/3.5 boron nitride and corncob ratio (wt./wt.) reinforced epoxy composite

3.4 Tensile and hardness test

Mechanical characterization of fabricated composite has been done on the bases of tensile and hardness tests. And the results obtained are being presented in Table 2 for 3.5/3.5 boron nitride and corncob ratio (wt./wt.) reinforced epoxy composite. Other researchers reported that neat epoxy has an ultimate tensile strength (UTS) range of 30 to 40 MPa [40, 42–44]. As per the results, reinforcement of corncob and boron nitride results in an improvement in UTS (which is about 107.1 ± 3.52 MPa) by 2 to 3 times as compared to neat epoxy. Similar result for Young's modulus (YM) is obtained, where neat epoxy has YM of about 1.3–1.5 GPa [45] which is increased to 2.3 GPa by using reinforcing material corn hub and boron nitride. On the other end, micro-hardness of the corncob and boron nitride–reinforced composite decreases with the addition of reinforcement material, and it is about 37.2 MPa as compared to neat epoxy which was about 100–130 MPa reported by someone else's work [46].

Table 2 Young's modulus, ultimate tensile strength, Vicker micro-hardness, and elongation of 3.5/3.5 boron nitride and corncob ratio (wt./wt.) reinforced epoxy composite

Young modulus	2.3 ± 0.87 GPa*
Ultimate tensile strength	107.1 ± 3.52 MPa*
Vicker micro-hardness	37.2 ± 1.3 HV*
% Elongation	4.6 ± 0.97%*

*Mean value ± standard deviation (5 samples were taken)

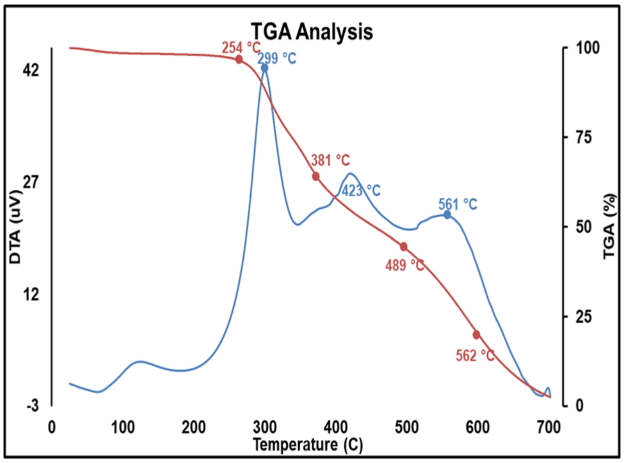

Fig. 8 Thermograph of 3.5/3.5 boron nitride and corncob ratio (wt./wt.) composite composition

3.5 TGA analysis

To investigate the thermal stability of fabricated composite films, TGA analysis has been done from room temperature 30 to 700°C on epoxy-based composite reinforced with 3.5/3.5 boron nitride and corncob ratio (wt./wt.) in the Fig. 8. As per the results decomposition of material has been done in three phases: 254 to 381°C, 381 to 489°C, and 489 to 562°C. Initially, there is about a 2–3% reduction in weight of the overall composite due to the presence of moisture up to 200°C. The first stage of decomposition starts at 254°C and ends near about 381°C with a peak at 299°C. During this phase, total weight loss of near about 34 %, considering the major degradation phase as compared to the other two. The second stage of decomposition starts at 381°C and ends near about 489°C with a peak at 423°C. During this phase, total weight loss was near about 16%, considering a minor degradation phase as compared to the other two. The third stage of decomposition starts at 489°C and ends near about 562°C with a peak at 561°C. During this phase, total weight loss was nearly about 27%. After complete decomposition, a residue of near about 2.5% has been left over which is been char. Neat corncob peaked at about 280°C as reported and discuss in Cheng et al.'s [47] work, which was also the reason for higher weight loss in the first phase reason in which the decomposition of matrix, i.e., epoxy and corncob particles, has taken place.

4 Conclusions

In the present investigation, epoxy based composite reinforced with corncob (obtained from the agriculture waste) was fabricated. With the aim to use a bio-waste into some useful product for the society, this study was performed. The authors also utilized the ultrahigh mechanical properties of boron nitride and used it as a second reinforcement. Through the mechanical characterization it was found that the flexural strength of the composite is maximum at 3.5/3.5 boron nitride and corncob ratio (wt./wt.) having a value of about 36.7 N/mm^2. The tensile test result shows that composite has brittle nature; and the reinforcement result in enhancing the ultimate tensile strength and the young's modulus values as compared to the neat epoxy matrix. Moreover, the composite is found to be almost insoluble in water with a maximum uptake of 7.81 % in 24 hours.

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

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Data availability Not applicable.

Declarations

Ethical approval The authors hereby state that the present work is in compliance with the ethical standards.

Competing interests The authors declare no competing interests.

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