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Bio-Based Epoxies: Mechanical Characterization and Their Applicability in the Development of Eco-Friendly Composites

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Abstract: The combination of awareness of harmful industrial processes, environmental concerns, and depleting petroleum-based resources has spurred research in developing sustainable materials from renewable sources. Natural bio-based polymers have replaced synthetic polymers because of growing concern about environmental sustainability. As a result of heating and distilling cashew nutshell liquid (CNSL), cardanol has emerged as a promising bio-retrieved component that can be used to make bio-based epoxy. The current work intends to investigate the mechanical properties of three kinds of cardanol-based bio-based epoxies in anticipation of widespread use. Vickers hardness, tensile and flexural strength are used to characterize mechanical properties. Additionally, a water absorption test is carried out to examine the weight gain properties of all the bio-based epoxy variants selected. FormuLITE 2 (FormuLITE 2501A + FormuLITE 2401B) exhibited the highest Vickers hardness, tensile and flexural strength among the three variants. Moreover, it exhibited a water absorption rate nearly equivalent to that of the conventional LY556/HY951, and thus, FormuLITE 2, the bio-based epoxy resin having 34% of bio-content blended with conventional epoxy, proves to be the best option out of the selected bio-based epoxies to be used further as the matrix material for the fabrication of biocomposites.

Keywords: bio-based polymer; characterization; eco-friendly composites; amine-cured epoxy system; phenolic resin

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1. Introduction

Epoxy resins are widely used crosslinked polymers that offer significant physical, mechanical, and thermal properties used in a variety of industries as adhesives, coatings, insulations, and high-performance composites and have occupied an indispensable role in various industries such as food, packaging, pharmaceutical, and therapeutic sectors. Epoxy-based composite materials are widely utilized in load-bearing applications, such as automotive, aerospace, construction, oil and gas, and marine, due to their superior mechanical qualities, high specific strength, super adhesiveness, and strong resistance to heat and solvents [1].

Polymer composites are polymer materials reinforced with fibers, with the polymer acting as a matrix that penetrates and adheres to the fibers [2]. As a result, there is an increasing demand for research into and prediction of the deformation behavior of epoxy-based composites under general loading circumstances [3].

On the other hand, epoxy is non-biodegradable, requires high concentrations to achieve desired qualities, and is relatively expensive. Their synthesis from chemical monomers paved the way for their eventual demise as a possibly hazardous non-biodegradable waste. The growing research interest in a sustainable environment has replaced synthetic polymers with natural bio-based polymers. Plant oils and starches, which are renewable natural resources, have gained interest as polymer building blocks due to their low cost, environmentally friendly nature, and ease of epoxidation, which results in bio-based epoxy resins. A bio-based polymer serves as the matrix for eco-friendly composites, consisting of a bio-based polymer and natural or synthetic reinforcements. Bio-polymers are synthesized from monomeric chemical sources, which paved the way to become a potentially harmful non-biodegradable waste in the environment. [4].

Cashew nutshell liquid (CNSL) is a viscous dark brown liquid contained within the delicate honeycomb structure of the cashew nutshell. It is a significant agricultural byproduct of cashew nut and cashew apple production [5]. Figure 1 illustrates the chemical structure of CNSL and its major constituents.

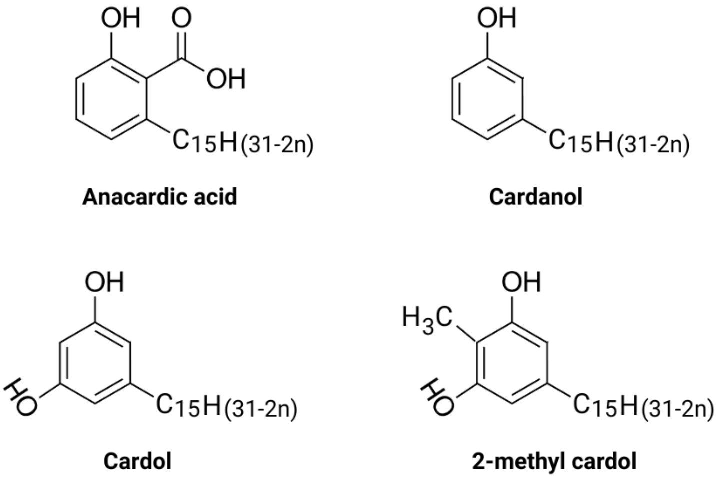


Figure 1. Chemical structure of CNSL with its components.

CNSL manufacturing on a global scale approaches one million tons annually [6]. As a result, CNSL established itself as one of the few significant and economically viable sources of naturally occurring phenols. Cardanol is a bio-substance created when CNSL is heated and then distilled, resulting in the decarboxylation of anacardic acid [7]. Due to their hydrophobic characteristics, organic coatings using cardanol were developed to protect metals from environmental corrosion caused by acids, salts, and water [8–12].

Additionally, its chemical structure can be changed to create a variety of bio-based surfactants that can be used in place of hazardous petroleum-based surfactants as corrosion inhibitors [13–15]. Cardanol derivatives have been widely employed in epoxy organic coatings as modified epoxy blends with bisphenol diglycidyl ether, anti-corrosive additives in epoxy, epoxy curing agents, and self-cured epoxy coatings [6,16–21]. Moreover, cardanol's phenolic nature and the unsaturation of its hydrophobic alkyl side chain drew attention to its chemical modification as a reactive raw material in manufacturing bio-based epoxy resins [22]. An aromatic ring in a phenalkamine structure confers chemical and fire resistance, while a long aliphatic chain gives the resin hydrophobicity and boosts its water resistance. A phenolic hydroxyl is added, which increases the resin's reactivity at low temperatures and accelerates curing; finally, an amine chain is added, which improves the resin's mechanical properties by raising the cross-linking density [23–25]. With the advent of new commercially available cardanol, it is required to study the mechanical and physical properties of the cardanol-epoxy-based bio-based polymer as products derived from this would find widespread use, particularly in synthesizing composites with an unusual combination of characteristics.

To summarize the literature, bio-based epoxy resins show a promising alternative to conventional epoxy. Cardanol is a bio-substance created by heating and distilling cashew nutshell liquid (CNSL), causing the decarboxylation of anacardic acid. Cardanol is a relatively inexpensive and readily available adaptable industrial raw material with considerable potential as a substitute for certain manufactured products. With the advent of commercially available cardanol-based-epoxy resins, the study focuses on researching their mechanical properties to better understand their potential use as bio-resins in eco-friendly composites.

2. Materials and Methods

2.1. Material Fabrication

Cardolite Specialty Chemicals India LLP supplied the FormuLITE amine-cured epoxy systems and Hindusthan Specialty Chemicals Limited, India, supplied conventional infusion resin and hardeners. The specimens were made by thoroughly mixing the resin and hardeners according to the mixing ratios in Table 1, obtained from commercial suppliers' data sheets. The mixtures were poured into molds to create 250 × 250 × 4 mm dimension bio-based polymer sheets. The switchboard cutting in the CNC Router machine was used to cut the specimens as per the ASTM standards for tensile test, flexural test, hardness test, and water absorption test.

Table 1. Composition of conventional epoxy and FormuLITE amine-cured bio-based epoxy systems.

Part A + Part B	Conventional Epoxy LY556 + HY951	FormuLITE 1 FormuLITE 2500A + FormuLITE 2401B	FormuLITE 2 FormuLITE 2501A + FormuLITE 2401B	FormuLITE 3 FormuLITE 2501A + FormuLITE 2002B
Calculated bio-content (wt.%)	0.0	36.60	34.00	45.40
Suggested cure cycles	24–32 h at RT 4 h at RT + 4 h at 60 °C	4–8 h at RT 2–4 h at 50–70 °C 2–3 h at 80 °C	4–8 h at RT 2–4 h at 50–70 °C 2–3 h at 80–100 °C	4–8 h at RT 2–3 h at 70–80 °C
Viscosity 25 °C (cPs)	10,000–12,000	700	905	1100
Pot life (min)	35 at 23 °C	105 at 25 °C	95 at 25 °C	58 at 25 °C

2.2. Experiment and Equipment Details

2.2.1. Experimental Setup for Tensile Testing

The ultimate tensile strength (σ_t) and Young's modulus (E) of the material were determined using the ASTM D638-14 'standard test method for tensile properties of plastics'. Five dumbbell-shaped, 4 mm thick samples of each type were subjected to uniaxial tensile load with their ends gripped in the universal testing machine (UTM). A gauge length of 50 mm was maintained. They conformed to the dimensions of Type 1 specimens specified in ASTM D638-14 standards. Figure 2 illustrates the schematic representation of a typical dumbbell-shaped specimen.

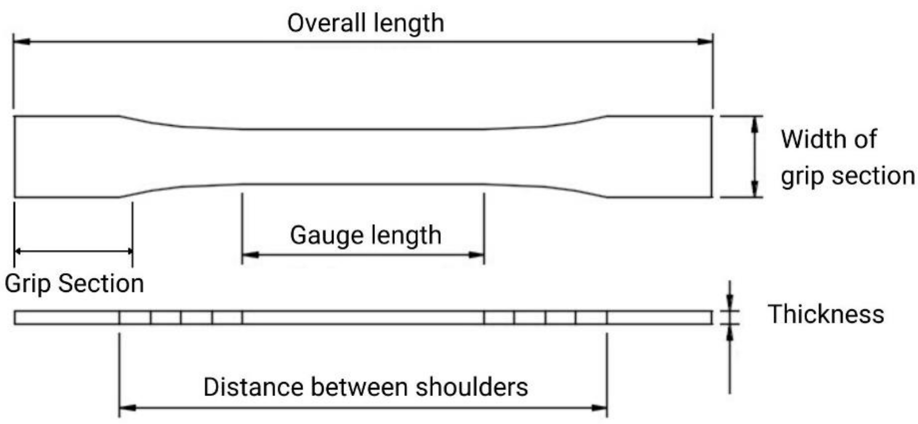


Figure 2. Schematic representation of tensile test specimen dimension as per ASTM D638-14 Standard.

Figure 3 illustrates the equipment setup used in the present study, wherein each specimen was mounted in the grips of a computerized universal testing machine from DAK system incorporation, having a capacity of 50 kN, which was monotonically loaded in tension while recording the applied load. The maximum load carried before failure determines the specimen's ultimate strength. The stress–strain responses of each specimen were recorded to determine its Young's modulus.

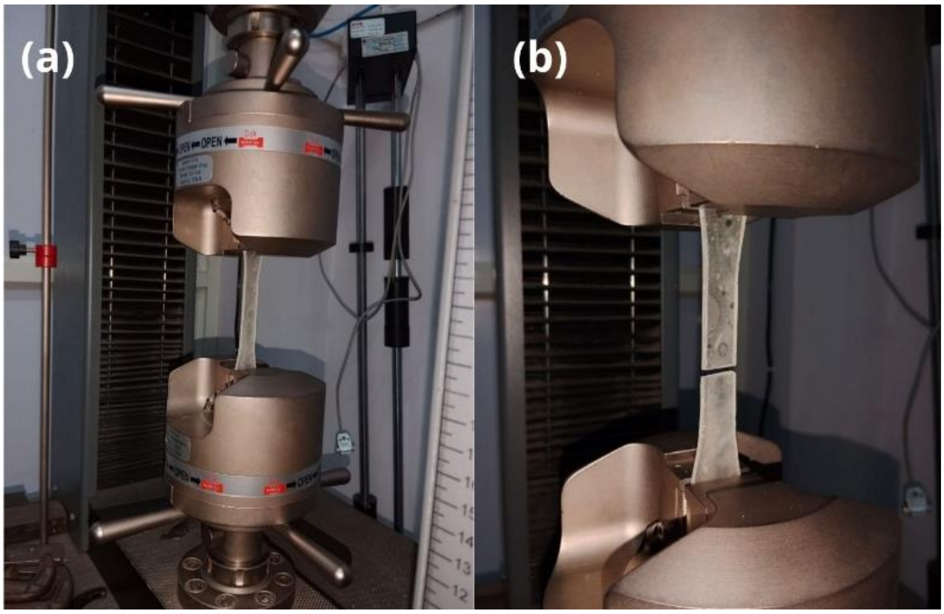


Figure 3. Equipment setup for tensile test specimen in a computerized universal testing machine from DAK system incorporation, (**a**) tensile specimen in a fixture, (**b**) fractured tensile specimen.

2.2.2. Experimental Setup for Flexural Testing

The ASTM D790-17 standard test methods for flexural properties of unreinforced and reinforced plastics and electrical insulating materials were used to determine the material's maximum flexural strength (σ_f) and tangent modulus of elasticity (E_B).

Five flat strips of 4 mm thickness were subjected to three-point bending on the DAK system incorporating computerized UTM for each type. All samples met the dimensions specified for thermoset specimens mentioned in ASTM D790-17 standards. The span was set to 70 mm to avoid shear stresses with a span-to-depth ratio of no less than 16.

The displacement rate during the tests was set to 3 mm/min, and Figure 4 shows the experimental setup as per the ASTM D790-17 standards. The maximum load determines the maximum flexural strength of the specimen carried before failure. Each specimen's stress–strain response was recorded to determine its tangent modulus of elasticity.

2.2.3. Experimental Setup for Hardness Testing

Hardness is a property of a material that determines its resistance to deformation. It is determined using a standard test measuring surface resistance to indentation. The indent's shape or type defines the most frequently used hardness tests, size, and amount of load applied. The hardness values referenced here represent an arbitrary, un-dimensioned scale, with increasing numbers representing harder surfaces [26]. Ductility, elastic stiffness, plasticity, strain, strength, toughness, viscoelasticity, and viscosity affect hardness [27].

The indentation hardness test is a type of hardness measurement used to determine the ability of the materials to resist mechanical damage when the indent diagonal or diameter is equal to or lesser than 1 micron. Among the various types of available tests, the Vickers hardness test is known [28,29]. Vickers hardness (*HV*) is defined as the ratio of the force '*F*' acting on the diamond indenter to the indentation's surface *A*, as defined in Equation (1). The load (N) is denoted by '*F*' in Equation (1), and '*d*' is the average measured value of the indentation's diagonal in (N-mm). The coefficient 1.854 pertains to the Vickers indenter's geometry. The *HV* test in the present study was conducted using the Matsuzawa MMT-X machine:

$$HV = \frac{F}{A} \cong 1.854\frac{F}{d^2} \quad (1)$$

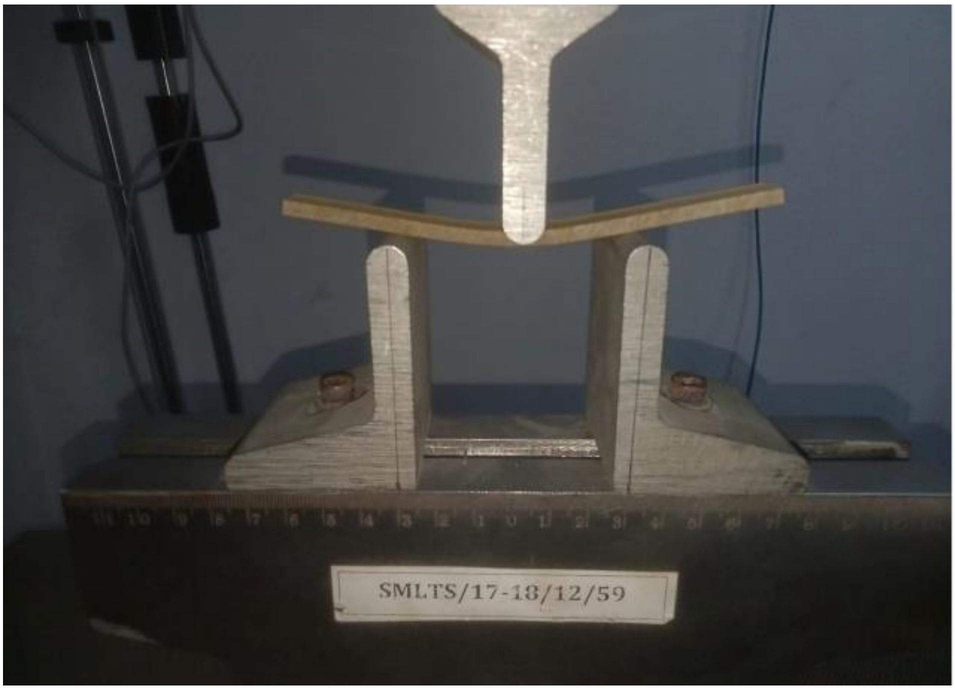


Figure 4. Equipment setup for flexural test specimen in a computerized universal testing machine from DAK system incorporation.

The indentation hardness characterized by the Vickers hardness of the prepared samples (Figure 5) is determined according to the ASTM E92-17 standard test method for Vickers hardness and Knoop hardness of metallic materials.

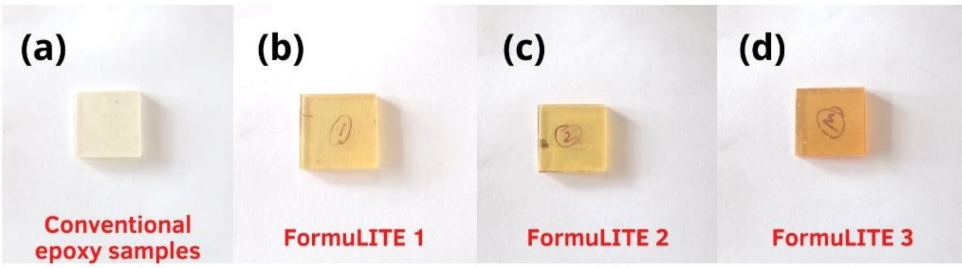


Figure 5. Hardness test specimen dimension as per ASTM E92-17 standard, (**a**) conventional epoxy, (**b**) FormuLITE 1, (**c**) FormuLITE 2, and (**d**) FormuLITE 3.

2.2.4. Experimental Setup for Water Absorption Test

The relative rate of water absorption by plastics when immersed is determined using ASTM D570-98 (reapproved 2018) 'standard test method for water absorption of plastics'. The test is important because the moisture content of plastic affects attributes including electrical insulating resistance, dielectric losses, mechanical strength, appearance, and dimensions. The prepared specimens as per the standard requirement for the water absorption test are shown in Figure 6.

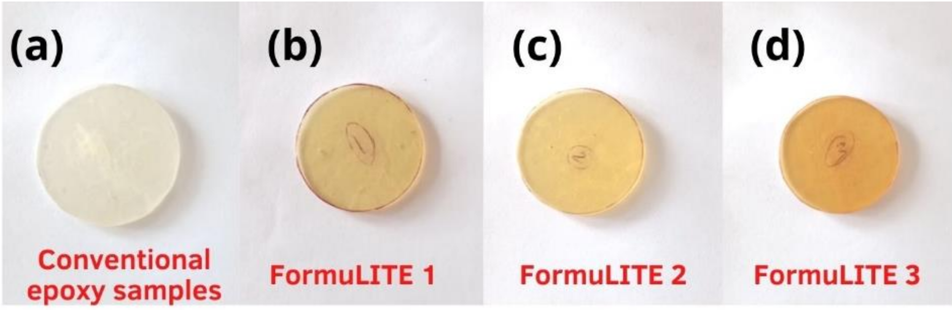


Figure 6. Water absorption test specimen dimension as per ASTM D570-98 standard, (**a**) conventional epoxy, (**b**) FormuLITE 1, (**c**) FormuLITE 2, and (**d**) FormuLITE 3.

Changes in moisture content due to water absorption have various effects on these properties, depending on the type of exposure (immersion in water or exposure to high humidity), the part's geometry, and the plastic's intrinsic qualities.

The 24 h immersion method is utilized in the present work wherein the conditioned specimens resting on the edge are entirely immersed in a container of distilled water maintained at a room temperature of 23 $\pm$ 1 °C. At the end of 24 + 1/2 h, the specimens were removed from the water one at a time, all surface water wiped off with a dry cloth, and weighed immediately using the digital weighing machine (Figure 7).

Figure 7. Water absorption specimens immersed in a container of distilled water.

The percentage increase in the mass during immersion is calculated to the nearest 0.01% using Equation (2), wherein Δm is the increase in mass, m_{wet} is the wet mass and m_{cond} is the conditioned mass:

$$\Delta m\ \% = \frac{m_{wet} - m_{cond}}{m_{cond}} \times 100 \tag{2}$$

2.2.5. Statistical Methods Used

Ronald Fisher proposed one-way ANOVA, the fundamental model that specifies a common mean value for all observations in a group [30,31]. It is used when a categorical factor and a continuous response exist to determine whether the population means of two or more groups differ. Since the present work aims to ascertain differences in mechanical properties between various types of matrix (resin + hardener) materials, one-way ANOVA was used as the statistical analysis tool. Additionally, the Games–Howell comparison is used to compare all pairs of groups while simultaneously controlling the confidence level in the existence of unequal variances.

3. Results

3.1. Results for Tensile Test

Tensile stress–strain diagrams for conventional LY556/HY951 and bio-based epoxies (FormuLITE 1, 2, and 3) specimens are shown in Figure 8. The diagram depicts the maximum tensile strength sample of each type. At low strains, the initial portion of the curve is linear, followed by a change in the slope of the curve, indicating the material's nonlinear behavior. The onset of nonlinearity in the curve indicates that the matrix has cracked initially [32].

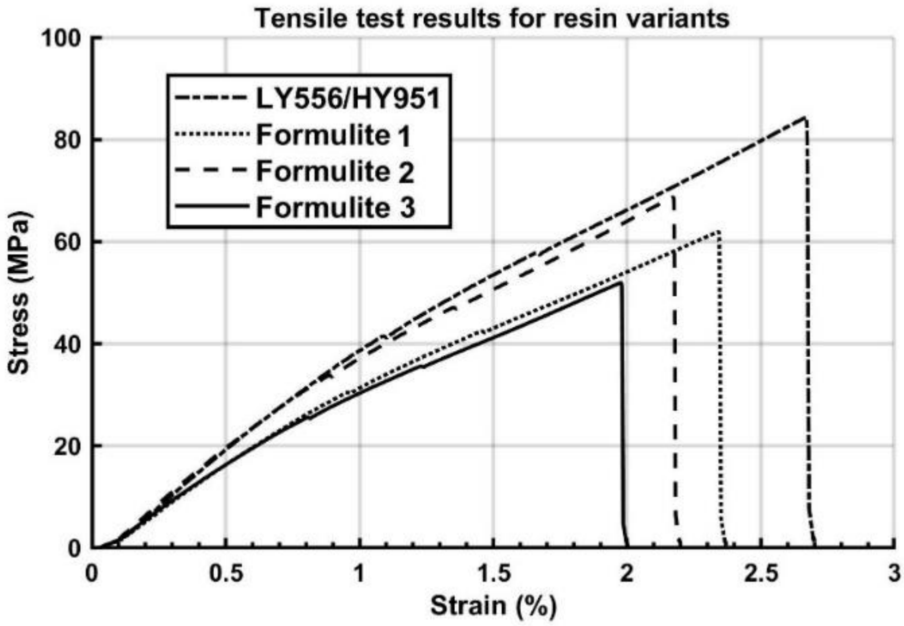


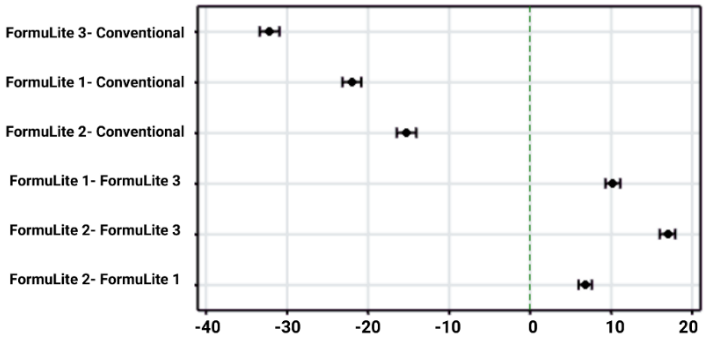
Figure 8. Stress–strain graph for samples tested for tensile properties.

The average ultimate tensile strength (UTS) of the LY556/HY951 specimen was 83.63 MPa, while FormuLITE 1, 2, and 3 had UTS values of 61.57, 68.32, and 51.40 MPa, respectively. FormuLITE 2′s UTS is the closest to that of conventional epoxy samples. Moreover, the tensile modulus of FormuLITE 2 is approximately the same and on the higher side. The ultimate tensile strength, strain at UTS, and tensile modulus at UTS of the specimen are summarized in Table 2. The table shows that the deviation between the true values of elastic properties obtained from supplier datasheets and the experimental values obtained is less than 5%.

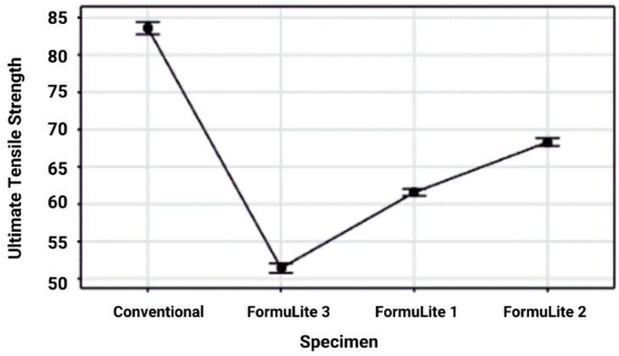
Table 2. Tensile properties of the conventional and bio-based epoxy system.

Tested Tensile Property	Conventional Epoxy Samples	FormuLITE 1	FormuLITE 2	FormuLITE 3
Ultimate tensile strength (MPa)	83.63	61.57	68.32	51.40
Strain at UTS (%)	2.68	2.35	2.18	1.97
Tensile modulus at UTS (GPa)	3.12	2.62	3.13	2.60

The analysis of variance (ANOVA) test conducted along with the Games–Howell comparison (considering unequal variance) indicated that there is a significant difference in the mean values of ultimate tensile strength of each of the tested types of epoxy/hardeners, and the same is depicted using Figure 9a,b represents the interval plot of UTS versus specimen, wherein individual standard deviations are used to determine the intervals.



(a)



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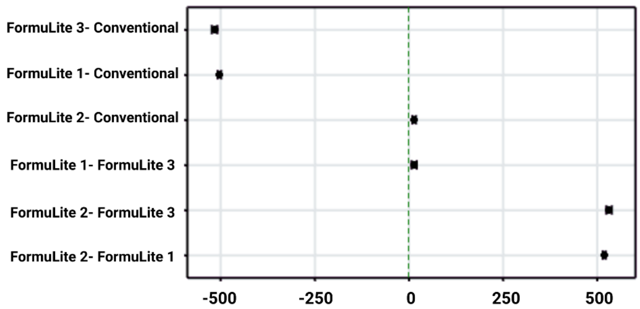
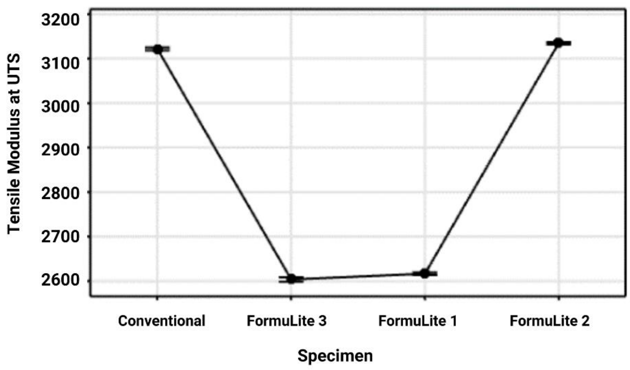


(c)



(d)

Figure 9. (**a**) Differences of means for ultimate tensile strength as per the ANOVA with Games–Howell comparison. (**b**) Interval plots of ultimate tensile strength versus epoxy/hardener specimens. (**c**) Differences of means for tensile modulus as per the ANOVA with Games–Howell comparison. (**d**) Interval plots of means of tensile modulus versus test specimens.

The interval plot indicates only slight variations between the determined samples of each type of specimen. The slight variation is normal due to minor flaws either caused due to stress concentration or because of slight thickness variation [33]. Similarly, the ANOVA with Games–Howell comparison result for tensile modulus is shown in Figure 9c, and the interval plots of the same are shown in Figure 9d.

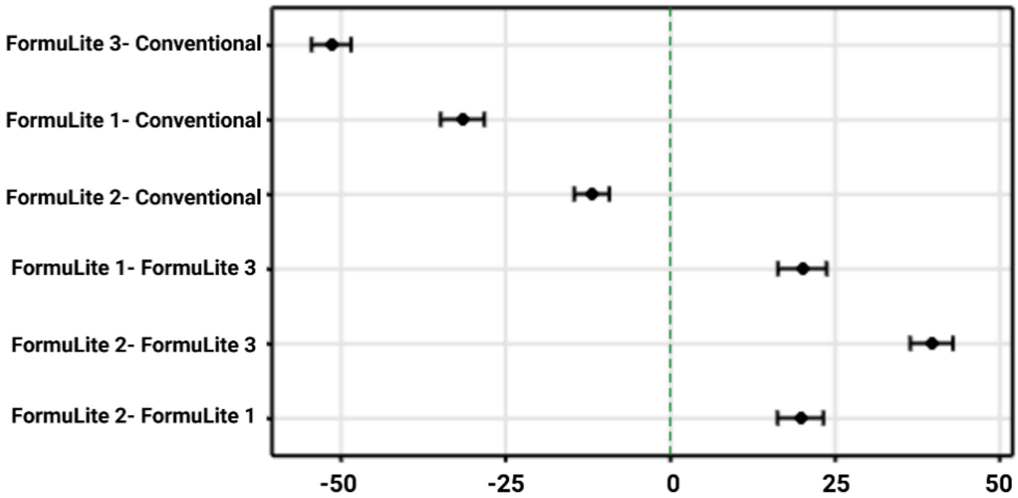
3.2. Results for Flexural Test

The results in Table 3 indicate that conventional LY556/HY951 exhibits an average ultimate flexural strength of 124.162 MPa. The average values of ultimate flexural strengths of bio-based epoxies, namely FormuLITE 1, 2, and 3, are 92.46, 112.13, and 72.55 MPa, respectively. However, similar to the case of tensile strength, FormuLITE 2 has the nearest possible value to the conventional epoxy compared to FormuLITE 1 and 3. Increased flexural strength indicates that the epoxy can tolerate greater loads when flexing. Due to its ability to bend, it is more resistant to fracture [34]. Therefore, FormuLITE 2 seems to be the best bio-based epoxy option among the three choices.

Table 3. Flexural strength of conventional epoxy and bio-based epoxy samples.

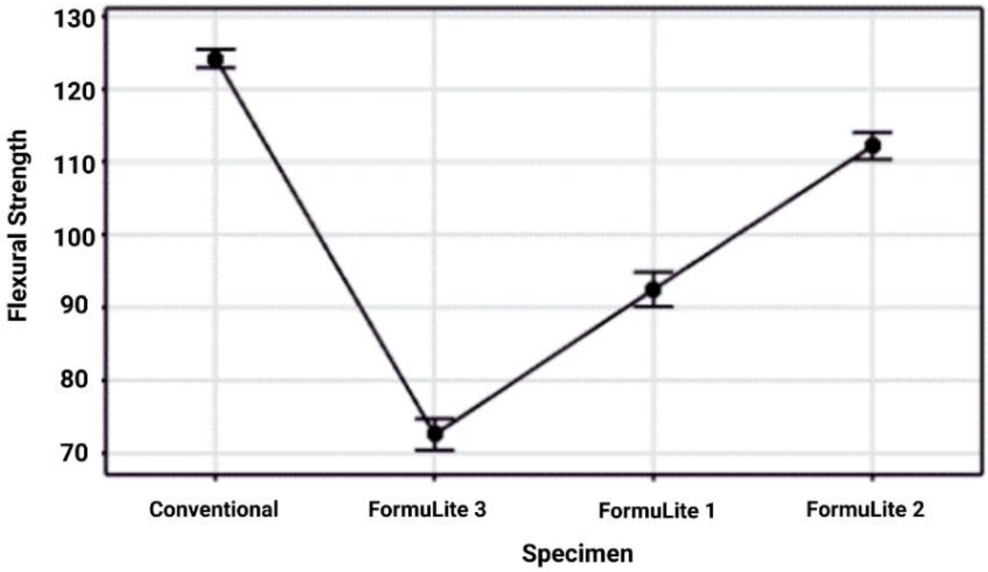
Tested Flexural Property	Conventional Epoxy Samples	FormuLITE 1	FormuLITE 2	FormuLITE 3
Average flexural strength	124.16	92.46	112.13	72.55

The ANOVA test conducted along with the Games–Howell comparison indicated a significant difference in the mean values of flexural strength of each of the tested types of epoxy/hardeners, and the same is depicted using Figure 10a,b represents the interval plot of flexural strength versus specimen, wherein individual standard deviations are used to determine the intervals. The interval plot indicates only slight variations between the determined samples of each type of specimen. The slight variation is normal due to minor flaws either caused due to stress concentration or because of slight thickness variation [33].



(**a**)

Figure 10. *Cont.*



(**b**)

Figure 10. (**a**) Differences of means for flexural strength as per the ANOVA with Games–Howell comparison. (**b**) Interval plots of means of flexural strength versus test specimens.

3.3. Results of Hardness Test

Table 4 indicates the average values of all the test samples (conventional and bio-based epoxies). The hardness was measured at three points for each specimen, and five specimens were tested for each sample as per ASTM E92-17 standard test method for Vickers hardness. Therefore, the average hardness value depicted is the average of 15 readings for each type of resin/hardener combination. The results indicate that the hardness of LY556/HY951 is 17.93 HV, while the values for bio-based epoxies range between 10.953 and 22 HV.

Table 4. Average hardness values of the conventional and bio-based epoxies.

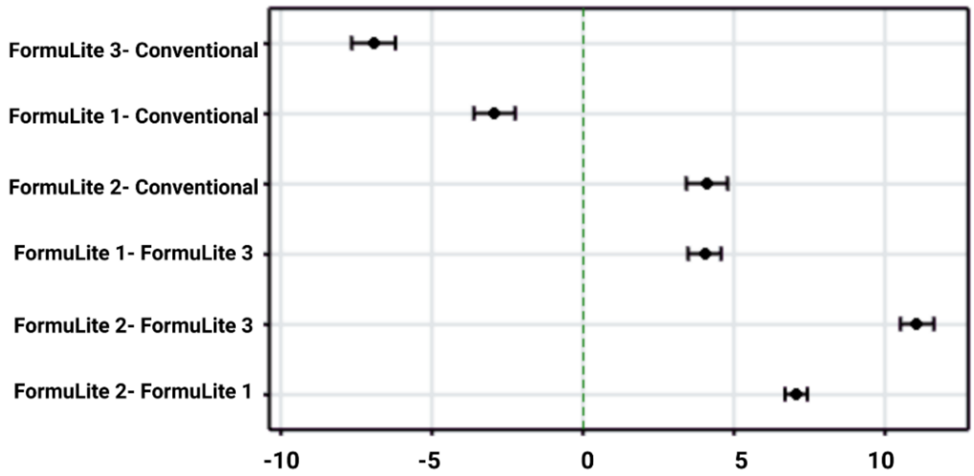
Sample	Average Hardness Values of the Samples (HV)
Conventional epoxy samples	17.91
FormuLITE 1	14.96
FormuLITE 2	22.00
FormuLITE 3	10.95

FormuLITE 2 bio-based epoxy samples exhibited the highest hardness value, even exceeding the values of the conventional LY556/HY951 sample by 18.57% on average. FormuLITE 3 exhibited the lowest hardness value, while FormuLITE 1 samples have the second maximum values and are near the values of conventional LY556/HY951.

The analysis of variance (ANOVA) test conducted along with the Games–Howell comparison (considering unequal variance) indicated a significant difference in the mean values of the hardness of each of the tested types of epoxy/hardeners, and the same is depicted using Figure 11a,b to represent the interval plot of hardness versus specimen, wherein individual standard deviations are used to determine the intervals.

3.4. Results of Water Absorption Test

Table 5 indicates the average values for mass gained due to 24 h of water immersion for all the test samples (conventional and bio-based epoxies). The weight gain in grams was measured for five samples of each specimen. Therefore, the average value depicted is the average of 5 readings for each type of resin/hardener combination. The results indicate that the weight gained by FormuLITE 2 was less than that of FormuLITE 1 and 3 and exhibited a water absorption rate slightly higher than the conventional LY556/HY951.



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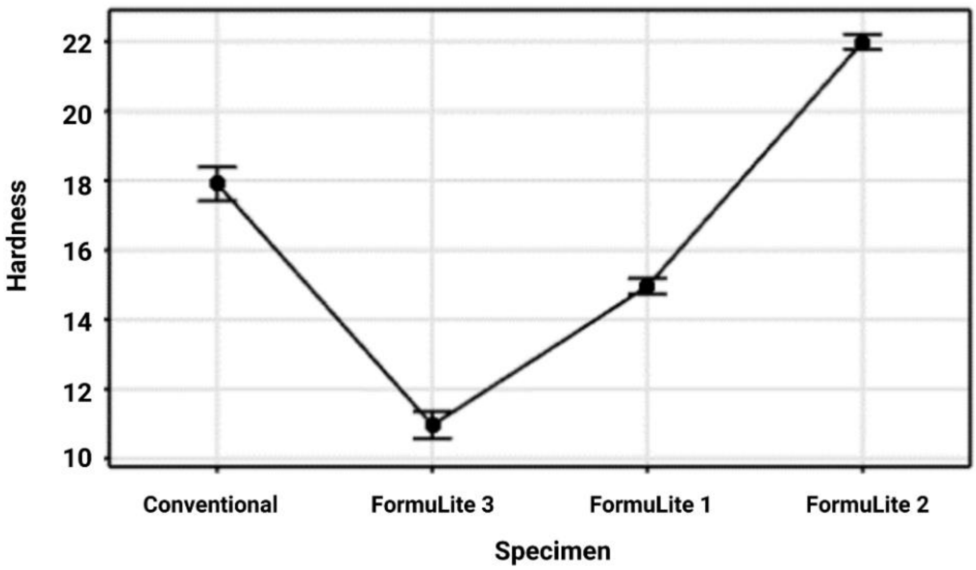

(**b**)

Figure 11. (**a**) Differences of means for hardness as per the ANOVA with Games–Howell comparison. (**b**) Interval plots of means of hardness versus test specimens.

Table 5. Weight gained in 24 h water absorption test by the samples.

Sample	Mass Gained Due to 24 h Water Absorption for the Samples
Conventional epoxy samples	0.08%
FormuLITE 1	0.22%
FormuLITE 2	0.10%
FormuLITE 3	0.28%

4. Discussion

From the results obtained experimentally for tensile, flexural, hardness, and water absorption, it is observed that cardolite-based (bio-based) epoxies could be a great alternative to synthetic epoxies. Compared to its counterparts such as furans, lignin, sugar, and vegetable oil. Cardolite has always proved to be the best option for fabricating bio-based epoxies [35]. It is observed that the mechanical strength characterized by tensile and flexural properties increases with the decrease in the bio element. The inclusion of the bio-component showed a 15–20% reduction in the tensile and flexural properties with the use of these bio-based polymers. This is because of the reduction in the cross-linking ability of the epoxy is reduced due to the addition of bio-content which leads to the reduction of the mechanical properties. The hardness value obtained for Formulite 2 was higher in value in comparison to conventional epoxy samples.

Moreover, hardness, the property which governs the material's wear resistance, is determined to decrease with the increased bio-component of the fabricated bio-based epoxies. The addition of increased bio-content or blending of the bio-based cardanol leads to reduced cross-linking of the polymers. The prepared sample was observed to be less brittle in comparison to the conventional epoxy samples. Hence, the hardness value trend differs in comparison to the mechanical properties.

The strength of bio-based polymers depends on the strength of hydrogen bonding, the strength of hydrogen clusters, and the strength of single molecules [36]. These elements that determine the strength of bio-based polymers might tend to differ from the difference in the composition of bio-based components in the fabricated bio-based epoxies and can be investigated in detail using several material characterization techniques. However, the material characterization using the advanced instruments does not fall into the scope of the present work and can be considered a possible extension by the researchers working in this area. Moreover, future researchers can also investigate the effects of confinement, which is also known to affect the strength of the bio-based polymers for all the three selected bio-based epoxies in the present study.

Observing the weight gain test, it may be concluded that the bio-component's increased concentration increased its affinity for water absorption. However, its right proportion improves water absorption, as shown in Table 5. The improvement in weight gain, in the case of FormuLITE 2 compared to FormuLITE 1 and 3, might be due to certain molecular level interactions and could be further investigated through material characterization techniques such as SEM, FTIR, EDX, and XRD analysis.

5. Conclusions

The mechanical tests characterized by tensile properties, flexural strength, and hardness, and the 24 h water absorption test were conducted for conventional epoxy/hardener and bio-based epoxies, namely FormuLITE 1, 2, and 3. The main aim of the work was to identify the bio-based epoxy out of the selected options for its further usage as the matrix material for the fabrication of biocomposites. The following points could be concluded from the conducted tests and statistical analysis. FormuLITE 2 has the highest ultimate tensile strength and is the closest to a conventional epoxy/hardener among the three bio-based epoxies. While FormuLITE 2′s average ultimate tensile strength is 18.32% lower than the conventional element, its average tensile modulus at UTS is slightly higher. Considering the flexural strength, the FormuLITE 2 indicated a better strength amongst the three selected bio-based epoxies and was nearest to the ultimate flexural strength of the conventional epoxy/hardener. FormuLITE 2 bio-based epoxy samples exhibited the highest hardness value considering the hardness test and even exceeded the values of the conventional LY556/HY951 sample by 18.57% on average. The 24 h water absorption test results indicate that the mass gained by FormuLITE 1 and 3 was more than that of FormuLITE 2, which exhibited a water absorption rate nearly equivalent to that of the conventional LY556/HY951. Thus, FormuLITE 2 proves to be the best option out of the selected bio-based epoxies to be used further as the matrix material for the fabrication of biocomposites. Moreover, the water absorption rate is almost like conventional LY556/HY951. Thus, it can be used as an alternative to creating a sustainable and eco-friendly matrix for eco-friendly composites.

Author Contributions: Conceptualization, N.N.; methodology, N.N.; software, R.B.; validation, B.H.S.T. and Z.G.; formal analysis, R.B.; investigation, N.N.; data curation, R.B.; writing—original draft preparation, N.N. and R.B.; writing—review and editing, B.S., B.H.S.T. and Z.G.; supervision, B.S. and Z.G. All authors have read and agreed to the published version of the manuscript.

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