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Biodegradability of Musa Acuminata (Banana)-Fiber-Reinforced Bio-Based Epoxy Composites: The Influence of Montmorillonite Clay †

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Abstract: The increasing environmental concerns associated with conventional composites, made using glass-fiber-reinforced polymers (GFRP) and carbon-fiber-reinforced polymers (CFRP), have shifted attention to bio-based composites. These environmentally responsible alternatives offer performance without sacrificing biodegradability. The present study examines the biodegradability of a novel bio-based epoxy composite reinforced with Musa acuminata (banana) fibers. Two composite variants were compared: one with 2.5% Montmorillonite (MMT) nanoclay and one without. While previous research has demonstrated an enhancement in mechanical and physical properties of polymer matrix composites with the addition of MMT nanoclay, it was hypothesized in this study that nanoclay addition would not significantly impact the composites' biodegradability. To confirm this, we conducted standard biodegradability tests and an SEM analysis. The SEM results revealed a uniform distribution of MMT nanoclay within the bio-based polymer matrix, in addition to strong interfacial adhesion and decreased void crater sizes. The inclusion of nanoclay did not significantly impact the composites' biodegradability, according to the statistical analysis provided in the present study. The present study also developed regression models to predict biodegradability over time to facilitate the determination of the timespan required for 100 percent biodegradability of the tested bio-based composite. Thus, this study is a significant benchmark for advancing eco-friendly composite materials.

Keywords: Musa acuminata; bio-based epoxy composites; nano clay filler; biodegradability; renewable composites

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1. Introduction

Due to their unique lightweight characteristics and superior mechanical properties, the development and application of composites, especially polymer matrix composites, have been progressively transforming industries ranging from aerospace to construction [1,2]. The most common varieties of these composites are glass-fiber-reinforced polymers (GFRP) and carbon-fiber-reinforced polymers (CFRP). Their unrivaled tensile strength, durability, and thermal resistance have made them indispensable in numerous applications, paving the way for developing innovative solutions in various fields. Despite these advantageous characteristics, GFRPs and CFRPs pose a significant environmental challenge [3,4]. Environmental concerns stem from their limited biodegradability, which makes their disposal difficult and contributes to the growing accumulation of non-degradable solid waste. This circumstance emphasizes the significance of developing new materials combining conventional composites' benefits with enhanced environmental compatibility. In response to this global issue, there is a growing scientific interest in investigating the potential of bio-based composites. These composites, derived from natural or renewable materials, can reduce environmental impact without significantly impairing material performance [5–7].

Bio-based epoxy composites reinforced with natural fibers have attracted considerable interest among bio-based alternatives [8–10]. The current research study concentrates on bio-based epoxy composites reinforced with banana plant fibers, also known as Musa acuminata. In several nations, these fibers are regarded as a waste product. However, they offer prospective sustainability and cost advantages [11]. Due to their high cellulose content, lightweight nature, and relative mechanical strength, banana fibers are a promising candidate for composite reinforcement [12]. This investigation focuses on the biodegradability of Musa acuminata-fiber-reinforced bio-based epoxy composites. Two variants of the composite have been investigated: one with 2.5% Montmorillonite (MMT) clay as infill and the other without. It has been demonstrated by previous research studies [13–17] that adding MMT clay to composites improves their mechanical, tribological, and physical properties. However, its effect on the biodegradability of these composites is currently unknown. According to the present work, it is hypothesized that the addition of MMT clay may enhance the performance of composites, but its effect on their biodegradability would be minimal. The confirmation or refutation of this hypothesis will play a crucial role in advancing our knowledge of bio-based composites, particularly their environmental implications. By investigating this hypothesis, the present study attempts to establish a foundation for developing and applying bio-based composites that are favorable to the environment. This contribution may inform future research in this field, thereby accelerating the development of sustainable composite materials.

2. Materials and Method

2.1. Materials

The present study utilized three primary materials to create the bio-based composite. The reinforcement material consisted of fibers from the Vruksha Composite, Andhra Pradesh, India, derived from the pseudostems of mature Musa acuminata (banana) plants. These fibers, a byproduct of banana cultivation, provided a sustainable alternative for composite reinforcement. The bio-based FormuLITE™ amine-cured epoxy system supplied by Cardolite LLC, United States, was selected as the composites' matrix. The epoxy system was synthesized using bio-derived monomers, particularly Cashew Nutshell Liquid (CNSL). It is characterized by low viscosity, efficient fiber hydration, an extended pot life, and balanced mechanical properties. Montmorillonite (MMT) demonstrated strong biocompatibility and biodegradability as the nano clay filler material. MMT was combined with aminopropyl triethoxy silane and octadecylamine to form MMT Nanoclay [18], which enhanced the composite matrix's mechanical and thermal properties.

2.2. Composite Fabrication

Several steps were involved in the fabrication of composites: resin and fiber preparation, composite lamination, compression molding, demolding, post-curing treatment, and abrasive water jet machining. Initially, the procured banana fibers were saturated with either ordinary bio-based epoxy resin or nanoclay-infilled bio-based epoxy resin, and then allowed to rest for optimal absorption. The mixture was then used to fabricate the composite material after the hardener was added and stirred in. A layer of banana fibers was added to a mold during the layup process and overlapped to assure uniformity. The epoxy resin was applied methodically to the fibers, and this process was repeated until the desired thickness (4 mm) of the composite was attained. Vacuum bags and polyvinyl alcohol-based release agents were employed to eliminate air pockets and prevent the composite from adhering to the mold during curing. Following the hand layup, the mold was sealed, initiating the compression molding process in which heat and pressure were applied to assist in curing. After the composite material had cured, it was carefully demolded and cut to the desired dimensions and shape.

For precision cutting, water jet machines with high-pressure water jets and fine abrasive sand were utilized to cut through the composite material without damaging it.

After the curing process, the composite was sanded or polished for a clean finish, then coated with an additional layer of epoxy, and cured in a hot air furnace for enhanced strength and aesthetics.

2.3. Scanning Electron Microscopy Analysis

Scanning electron microscopy (SEM) was utilized to conduct a detailed microstructural analysis of the fabricated bio-based composites with and without MMT nanoclay. The composite samples for the SEM analysis were prepared as per the standards, and prior to observation, they were coated in a vacuum with a thin conductive layer of gold-palladium (Au-Pd) to prevent charging under the electron beam. The coating procedure guarantees high resolution and prevents the buildup of static electricity on the specimen's surface during electron bombardment [19]. A high-resolution ZEISS scanning electron microscope was utilized to conduct the SEM examination. From the obtained SEM images, characteristics such as the distribution of MMT nanoclay within the polymer matrix, the quality of the interface bonding between the matrix and the nanoclay, and any potential morphological changes caused by the addition of MMT nanoclay were analyzed and discussed.

2.4. Biodegradability Testing

Assessing the biodegradability of the Musa acuminata (banana)-fiber-reinforced bio-based epoxy composite necessitated a methodical procedure inspired by the ASTM D5338 standard test method for determining the aerobic biodegradation of plastic materials under controlled composting conditions [20,21]. The bio-based composite material test specimens were cut into uniformly sized and shaped samples with a constant weight. The mud with compost was selected as the testing medium in the present study. The test containers were prepared by filling them with the testing medium, and the composite samples were completely submerged. As necessary, water or other nutrients were introduced to promote microbial growth and aid the biodegradation process. Throughout the experiment, the weight loss in the samples was monitored at regular intervals of 10 days for a total period of 60 days. The results were recorded, providing insight into the progression of biodegradation. Using Equation (1), the biodegradability percentage was determined for each sample. A total of 10 samples were tested for each variant, and the average values were considered while recording the weights.

$$Biodegradability\ (\%) = \frac{\left(W_i - W_f\right)}{(W_i)} \times 100 \tag{1}$$

2.5. Statistical Analysis

In the present study, a series of statistical analyses were conducted to comprehend the data, determine the relationship between variables, and predict the biodegradability of composite materials. Primarily, the two-sample *t*-test and regression modeling techniques were utilized. The two-sample *t*-test, also known as the independent samples t-test [22,23], was utilized to compare the mean values of the two groups: composites with and without the addition of MMT clay infill, using the independent sample size. This test determined the chances of substantial differences in biodegradability between these two groups. The t-test presupposes that the data are normally distributed and that the variances of the two groups being compared are equal. The t-test results included the t-value, degrees of freedom, and p-value. If the p-value is less than the predetermined significance level (normally 0.05), the null hypothesis is rejected, and it is concluded that the two groups differ significantly [22].

A predictive model of the composites' biodegradability was developed using regression modeling techniques. The primary objective was to investigate the relationship between the variables and use this relationship to predict the amount of time required for complete biodegradation. The dependent variable for the regression models in the present study was the percentage of biodegradability, while the independent variable was the incubation time. The procedure involved constructing a linear regression model, analyzing its residuals to ensure that they satisfy the assumptions of linearity, independence, homoscedasticity, and normality, and then utilizing the model to make predictions. The R-squared value was utilized to determine how well the model suits the data. The regression equation facilitated the biodegradability prediction of the prepared bio-based composite variants for a given incubation time.

3. Results and Discussion

3.1. Morphological Result

The analysis using scanning electron microscopy (SEM) revealed that the MMT nanoclay was evenly distributed throughout the polymer matrix, indicating a high level of dispersion. The absence of observed agglomeration suggests the effective exfoliation of nanoclay layers within the polymer. In addition, the absence of discernible spaces demonstrated robust interfacial adhesion between the MMT nanoclay and polymer matrix. Another significant finding was that the reduced void crater sizes in the polymer matrix indicated enhanced compaction and packaging within the composite. The composites containing MMT nanoclay had a morphologically coarser surface than those without nanoclay.

The obtained SEM results may have implications for the mechanical properties of the composite, but this is beyond the scope of the current study. Figure 1 shows SEM images of the tested composites without and with MMT nanoclay infill. The successful and uniform distribution of the MMT nanoclay throughout the polymer matrix is important for attaining desirable properties in the resulting nanocomposite materials. The absence of observed agglomeration indicates that the nanoclay was effectively exfoliated and distributed within the polymer matrix. This is particularly important because agglomerates can function as defects, decreasing the composite's overall performance. The SEM analysis revealed that the strong interfacial adhesion between the nanoclay and polymer matrix could contribute to the improved performance of the material, particularly in terms of its biodegradability. A strong interfacial adhesion may restrict degrading agent access to the polymer matrix, thereby enhancing the composite's biodegradation resistance.

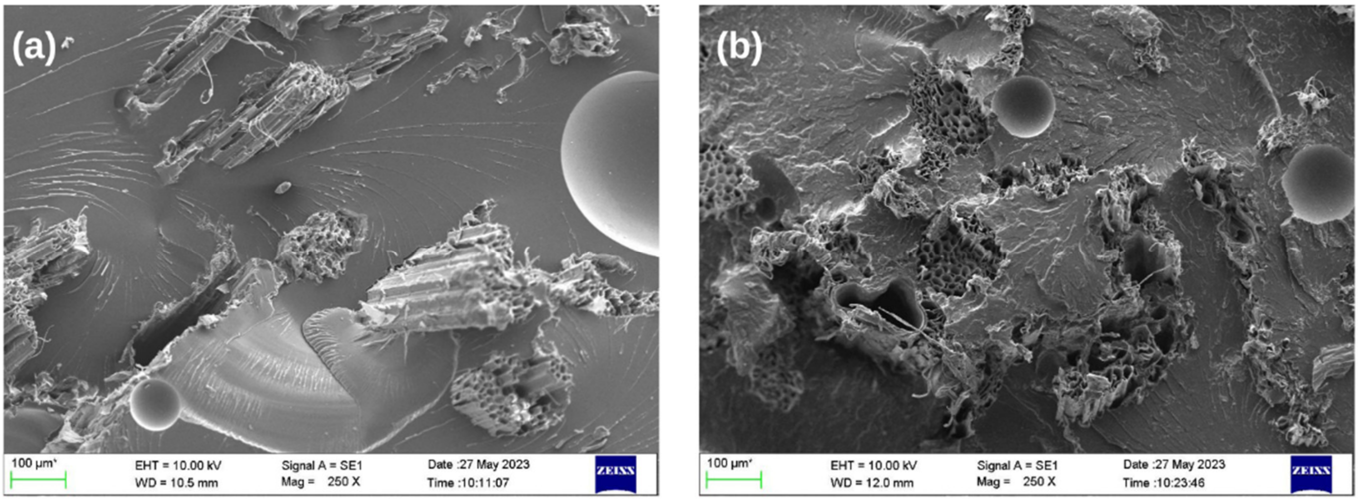

Figure 1. Scanning electron microscopy (SEM) images at 250× magnification and 100 microns showing a comparison between (**a**) a composite without nanoclay and (**b**) a composite with 2.5% MMT nanoclay.

The reduction in the size of the void craters is also a positive finding, as voids in composites can serve as sites of initiation for degradation or failure under stress. Reduced void sizes suggest enhanced packing and increased density, which can result in a more robust and durable material. The increased roughness observed on the surface of the composites containing MMT nanoclay is a significant morphological change that may result from the interaction between the polymer matrix and nanoclay. Understanding the exact implications of this change on the mechanical properties and surface characteristics of the composites, which were not investigated in this study, would necessitate additional research.

3.2. Biodegradability Result

The biodegradability experiments for the bio-based epoxy composites, one with a 2.5% MMT nanoclay filler and one without, were conducted over 60 days. Both composites demonstrated a distinct tendency for weight loss, indicating biodegradation. Tabulated in Table 1 are the experimental results.

Table 1. Experimental data for the biodegradability test.

Sample	Incubation Period	Initial Weight, W_i (g)	Final Weight, W_f (g)	Weight Loss (g)	Biodegradability (%)
Bio-based composite without nanoclay	0	5	5	0	0
	10	5	4.83	0.17	3.4
	20	5	4.69	0.31	6.2
	30	5	4.48	0.52	10.4
	40	5	4.27	0.73	14.6
	50	5	4.13	0.87	17.4
	60	5	4.04	0.96	19.2
Bio-based composite with 2.5% MMT nanoclay	0	5	5	0	0
	10	5	4.85	0.15	3
	20	5	4.71	0.29	5.8
	30	5	4.51	0.49	9.8
	40	5	4.3	0.7	14
	50	5	4.16	0.84	16.8
	60	5	4.07	0.93	18.6

The statistical two-sample t-test was performed to compare the mean biodegradability of both variants of the bio-based composites. The population mean of biodegradability for the composites containing MMT nanoclay is denoted by 1, while the population mean of biodegradability for the composites lacking nanoclay is denoted by 2. This analysis was primarily concerned with the difference between these means, denoted by 1–2. As part of the testing procedure, it was not assumed that the two groups had comparable variances.

Table 2 provides descriptive statistics for the composites' biodegradability (%) with and without MMT nanoclay. As indicated by 'N,' each variety is characterized by seven observations (0 to 60 days). The average biodegradability of the composites containing MMT nanoclay is 9.71%, while that of the composites without nanoclay is 10.17%. This mean is accompanied by a standard deviation, quantifying the variability among composites within each group.

Table 2. Descriptive statistics: biodegradability (%).

Composite Type	N	Mean	St Dev	SE Mean
With MMT Nanoclay	7	9.71	7.10	2.7
Without Nanoclay	7	10.17	7.29	2.8

The standard deviations of the composites with and without nanoclay are 7.10% and 7.29%, respectively. In addition, the standard error of the mean indicates the precision of these mean estimates, with values of 2.7 and 2.8 for the composites with and without nanoclay, respectively.

The estimated difference between the means of the two composite groups and the 95% confidence interval for this difference is presented in Table 3. The difference of −0.46 indicates that, on average, composites without nanoclay have a marginally higher biodegradability than those with MMT nanoclay. However, the confidence interval ranging from −8.92 to 8.00 indicates that this difference is subject to substantial uncertainty.

Table 3. Estimation of the difference.

Difference	95% CI for Difference
−0.46	(−8.92, 8.00)

The results of the two-sample t-test are shown in Table 4. The null hypothesis states that there is no difference in the mean biodegradability of the two categories of composites, whereas the alternative hypothesis states that there is a difference. The test statistic, or t-value, is −0.12, and the degrees of freedom, which indicate the number of values that are free to vary, are 11. If the null hypothesis is true, the p-value is the probability of obtaining the observed data (or more extreme data). In this instance, the p-value is 0.907, which is substantially higher than the standard significance level of 0.05. Therefore, we do not reject the null hypothesis, indicating insufficient evidence to conclude that the two composite types differ in biodegradability.

Table 4. Two-sample t-test result.

Null Hypothesis	Alternative Hypothesis	T-Value	DF	*p*-Value
H_0: $\mu_1 - \mu_2 = 0$	H_1: $\mu_1 - \mu_2 \neq 0$	−0.12	11	0.907

This observation was evaluated further using regression modeling to determine the effect of incubation time on biodegradability and to determine whether the composite type plays a significant role. The regression equations for the two composites with and without nanoclay are represented by Equations (2) and (3), respectively.

$$Biodegradability\ (\%) = -0.229 + 0.33143 * Incubation\ Time\ (Days) \tag{2}$$

$$Biodegradability\ (\%) = 0.229 + 0.33143 * Incubation\ Time\ (Days) \tag{3}$$

Consistent with the obtained biodegradability test findings, the positive coefficient of the incubation time indicates that as the incubation time increases, the biodegradability percentage also increases.

Table 5 displays the regression analysis results for predictors highlighting the incubation time (Days) as significant, positive relationship (coefficient = 0.33143, $p < 0.001$) with the dependent variable. However, composite type exhibits non-significance (coefficient = 0.457, $p = 0.259$), suggesting limited impact.

Table 5. Regression analysis results.

Predictor	Coefficient	Std. Error	T-Value	*p*-Value	VIF
Intercept	−0.229	0.396	−0.58	0.576	
Incubation Time (Days)	0.33143	0.00961	34.48	0.000	1.00
Composite Type	0.457	0.384	1.19	0.259	1.00

The regression model exhibits a high R-squared value of 99.08%, indicating that the incubation duration and composite type can explain 99.08% of the biodegradability variance. The p-value for the incubation time is statistically significant (0.000), supporting the notion that the incubation time is a crucial factor influencing biodegradability as shown in Table 6.

Table 6. Regression model summary.

S	R-sq	R-sq (adj)	R-sq (pred)
0.719307	99.08%	98.92%	98.47%

However, the p-value for the composite type is relatively high (0.259) as shown in Table 7, indicating that the composite type, particularly the addition of MMT nanoclay, does not contribute substantially to biodegradability, as the *t*-test analysis also suggested.

Table 7. ANOVA results.

Source	DF	Adj SS	Adj MS	F-Value	*p*-Value
Regression	2	615.863	307.931	595.15	0.000
Incubation Time (Days)	1	615.131	615.131	1188.88	0.000
Composite Type	1	0.731	0.731	1.41	0.259
Error	11	5.691	0.517		
Total	13	621.554			

These findings provide a firm basis for estimating the biodegradability of bio-based composites over time. Further investigation of the interactions of MMT nanoclay with the epoxy matrix and biodegradation microorganisms could lead to the creation of even more sustainable and effective bio-based composites.

4. Conclusions

The present study examined the biodegradability of two varieties of Musa acuminata (banana)-fiber-reinforced bio-based epoxy composites: one containing 2.5% MMT nanoclay and the other without nanoclay. The objective was to determine if the addition of nanoclay, which is frequently associated with enhanced mechanical and physical properties, affects the biodegradability of the composites. The SEM analysis determined that the MMT nanoclay was uniformly disseminated within the polymer matrix, exhibiting strong interfacial adhesion and decreased void crater sizes. These characteristics may contribute to enhanced composite efficacy. Despite these favorable morphological changes, our statistical analysis revealed that nanoclay had no significant effect on biodegradability, as measured by the weight loss percentage. Both types of composites demonstrated a progressive increase in biodegradability over time, confirming our initial hypothesis that while nanoclay addition may improve the overall performance of the composites, it does not significantly impact their biodegradability. This finding contributes to the current understanding of biodegradable composites, particularly those reinforced with banana fibers and bio-based epoxy, and offers beneficial direction to manufacturers and researchers for developing high-performance, environmentally sustainable composite materials. During the analysis, regression models highlighted the strong correlation between the incubation time and biodegradability, highlighting the potential of these composites as eco-friendly substitutes for non-biodegradable materials. As different materials may yield varying results, future research should evaluate the impact of various bio-based fibers, resins, and fillers on biodegradability and mechanical performance. Optimizing the nanoclay content of bio-composites to obtain the optimal balance between mechanical properties and biodegradability remains an intriguing area for future research.

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