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The effect of the presence of nanoclay and basalt fibers on the tensile and impact mechanical properties of PLA/NR polymer composites using the response surface method (RSM)

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Abstract

This study experimentally investigates the influence of nanoclay and basalt fibers on the tensile and impact properties of polylactic acid/natural rubber (PLA/NR) biocomposites using response surface methodology (RSM). The investigated variables included nanoclay (0–6 wt%), basalt fibers (0–20 wt%), and natural rubber (0–30 wt%). Composite samples were prepared via internal mixing and hot pressing in accordance with standard protocols. Tensile tests evaluated tensile strength and elastic modulus, while Charpy impact tests assessed impact resistance. Results indicated that low nanoclay loading (≤3 wt%) enhanced tensile strength and elastic modulus by 10% and 21%, respectively, whereas higher loadings led to agglomeration and reduced performance. Basalt fiber incorporation significantly improved tensile strength (14%), elastic modulus (37%), and impact strength (39%). Conversely, increasing natural rubber content enhanced impact strength by up to 44% but reduced tensile strength and modulus by 11% and 23%, respectively, due to its soft elastomeric nature. Scanning electron microscopy (SEM) was employed to examine the dispersion of nanoclay and basalt fibers within the PLA/NR matrix, revealing good interfacial adhesion at optimal loadings and agglomeration at higher nanoclay concentrations. These findings provide valuable insights for tailoring the mechanical performance of sustainable PLA/NR composites through balanced hybrid reinforcement.

Keywords: Biodegradable nanocomposites, nanoclay, basalt fibers, mechanical properties, scanning electron microscopy

1. Introduction

In recent decades, composites and nanocomposites have emerged as among the most significant advancements in materials science. These materials consist of two or more distinct phases, each contributing uniquely to enhance the overall performance of the final product. Owing to their exceptional properties such as high strength-to-weight ratios, lightweight nature, and design flexibility composites have been widely adopted across diverse industries, including aerospace, automotive, biomedical, and sustainable packaging [1]. Biodegradable nanocomposites have gained significant traction in advanced industries such as aerospace and automotive, owing to their exceptional combination of properties—including low density, high strength-to-weight ratio, impact resistance, and biocompatibility. In the aerospace sector, where minimizing weight while maintaining structural integrity is paramount, these materials offer a compelling solution. Similarly, in the automotive industry, biodegradable nanocomposites serve as sustainable alternatives to conventional materials, enabling the production of lightweight components such as body panels, interior trims, and structural parts. By reducing vehicle mass, they contribute to lower fuel consumption and decreased greenhouse gas emissions. Beyond performance enhancements, the inherent biodegradability of these composites helps mitigate the environmental burden associated with persistent plastic waste, aligning industrial practices with principles of circular economy and sustainable development [2–5]. Trivedi et al. [6] provided a comprehensive review of polylactic acid (PLA)-based composites reinforced with natural fibers and nanoparticles for sustainable product development. They highlighted PLA composites as a promising alternative to conventional petroleum-based plastics, owing to their biodegradability, environmental compatibility, and tunable mechanical properties when combined with natural reinforcements such as plant fibers, biodegradable elastomers, and nanofillers. The authors also identified key challenges related to the limited toughness, impact resistance, and thermal stability of these materials and proposed several strategies to address them, including fiber surface modification, blending with flexible polymers, and optimization of processing parameters.

The primary objective of this study is to optimize the tensile strength and impact resistance of biodegradable nanocomposites reinforced with basalt fibers and nanoclay. To achieve this, the Box–Behnken design of experiments is employed to systematically evaluate the influence of varying reinforcement concentrations on the mechanical performance of the composites. Complementary structural analyses, conducted via scanning electron microscopy (SEM), provide insights into the reinforcement mechanisms and interfacial interactions between the constituent phases. The findings of this research can serve as a foundational reference for the development of advanced, eco-friendly materials tailored for diverse industrial applications.

2. Methodology

In this study, four primary materials were employed: polylactic acid (PLA) as the base polymer, natural rubber (NR) as a toughening agent, nanoclay as a nanoscale filler, and basalt fibers as a microscale reinforcement. The PLA was supplied by NatureWorks LLC, the NR was sourced from Rubberex Corporation (Malaysia), the nanoclay was obtained from Southern Clay Products, and the basalt fibers were procured from Basalt Fiber Co. (China).

Sample preparation was carried out via melt blending using an internal mixer (Model HBISYS90) operated at 200 °C and 60 rpm. The process began by melting PLA as the matrix material, followed by the incorporation of NR at three weight percentages (0, 15, and 30 wt%). Subsequently, nanoclay (0, 3, and 6 wt%) and basalt fibers (0, 10, and 20 wt%) were added to the blend. Each formulation was mixed for a total of 10 minutes to ensure homogeneity.

Following melt mixing, the composites were compression-molded into standard specimens using a hot press for mechanical characterization, specifically tensile and impact testing. To ensure reproducibility and statistical reliability, five replicate specimens were fabricated for each test condition. Based on a Box–Behnken experimental design generated using Design-Expert® software, a total of 15 distinct material combinations were formulated, as detailed in Table 1. Tensile testing was conducted using a Zwick Roell Z100 universal testing machine in accordance with the ISO 527-1 standard. The crosshead speed was set to 5 mm/min, and specimens were loaded in tension until fracture. Impact resistance was evaluated using a Charpy impact tester following the ISO 179 standard at ambient temperature. To investigate fracture morphology and interfacial interactions, scanning electron microscopy (SEM) was performed on the fractured surfaces of selected samples post-impact. To prevent surface charging during SEM imaging, the specimens were sputter-coated with a thin (~10 nm) layer of gold.

Table 1. Weight percentage of compounds designed by Design Expert software

Run	Nanoclay (wt%)	Basalt Fiber (wt%)	NR (wt%)	PLA (wt%)
1	0	0	15	85
2	6	0	15	79
3	0	20	15	65
4	6	20	15	59
5	0	10	0	90
6	6	10	0	84
7	0	10	30	60
8	6	10	30	54
9	3	0	0	97
10	3	20	0	77
11	3	0	30	67
12	3	20	30	47
13	3	10	15	72
14	3	10	15	72
15	3	10	15	72

3. Discussion and Results

In this phase of the study, the combined effect of two reinforcing agents basalt fibers and nanoclay was investigated within a polymer matrix composed of polylactic acid (PLA) and natural rubber (NR). Following tensile and impact mechanical testing, the average results for impact strength, tensile strength, and elastic modulus are summarized in Table 2.

Table 2. Results of tensile and impact tests

Sample Code	Tensile Strength (MPa)	Elastic Modulus (GPa)	Impact Strength (J/m)
1	57	1.3	21
2	58	4.3	15
3	65	3.5	30
4	66	6.5	24
5	65	8.4	18
6	66	5.1	12
7	59	0.4	33
8	60	4.1	28
9	65	4.3	10
10	75	6.5	19
11	59	3.5	25
12	67	5.7	35

13	67	5.2	23
14	67	5.2	23
15	67	5.2	23

The incorporation of up to 3 wt% nanoclay into the PLA/NR matrix results in a ~10% increase in tensile strength. This enhancement can be attributed to two primary mechanisms: (i) the high surface-to-volume ratio of clay nanoparticles and their strong interfacial interactions with the polymer chains significantly improve stress transfer efficiency at the matrix nanoparticle interface [7]; and (ii) at low loadings (≤3 wt%), the nanoparticles disperse uniformly, promoting the formation of an intercalated or partially exfoliated nanostructure that reinforces the composite [8]. However, further increasing the nanoclay content beyond 3 wt% leads to an ~8% reduction in tensile strength. This decline is attributed to nanoparticle agglomeration at higher concentrations, which generates stress concentration sites and compromises interfacial adhesion, thereby diminishing reinforcement effectiveness. The addition of basalt fibers up to 15 wt% enhances the tensile strength of the PLA/NR nanocomposites by approximately 14%. Under applied load, the basalt fibers effectively carry a significant portion of the stress, thereby restricting matrix deformation. Their inherently rough and chemically active surface promotes strong interfacial bonding with the PLA matrix. At 15 wt%, the fibers are well-dispersed and exhibit favorable alignment along the loading direction, minimizing the formation of fiber clusters that could otherwise act as stress concentrators. Moreover, the presence of well-dispersed nanoclay further enhances interfacial adhesion by occupying interstitial spaces between the fibers and the matrix, thereby improving load transfer [9]. Conversely, the incorporation of natural rubber (NR) reduces tensile strength by approximately 11%. This reduction stems primarily from the inherent incompatibility between the hydrophobic, semi-crystalline PLA matrix and the elastomeric NR phase, which leads to weak interfacial adhesion and limited stress transfer across the phase boundaries.

The incorporation of up to 3 wt% nanoclay into the PLA/NR matrix enhances the elastic modulus by approximately 21%. This notable improvement can be attributed to the high surface-to-volume ratio of well-dispersed clay nanoparticles, which effectively restrict polymer chain mobility and increase matrix stiffness. However, further increasing the nanoclay content to 6 wt% results in a 14% reduction in elastic modulus, primarily due to nanoparticle aggregation and the formation of agglomerates, which act as stress concentrators and impair load transfer within the matrix. The addition of basalt fibers also significantly influences composite stiffness. Introducing up to 15 wt% basalt fibers into the PLA/NR nanocomposite leads to a 37% increase in elastic modulus. Basalt fibers possessing a much higher intrinsic modulus than the polymer matrix serve as effective load-bearing reinforcements. Under applied stress, these fibers carry a substantial portion of the load, thereby enhancing the overall rigidity of the composite.

Conversely, the inclusion of natural rubber (NR) in the PLA matrix reduces the elastic modulus by 23%. This softening effect arises from the dispersion of elastomeric NR domains within the rigid PLA structure. These soft rubbery phases act as compliant regions that undergo greater elastic deformation under load, thereby lowering the composite's overall stiffness [10].

The incorporation of nanoclay into the composite formulation leads to a notable reduction in impact strength. This decline in toughness is attributed to the formation of a rigid percolating network by the clay nanoparticles within the polymer matrix, which restricts segmental mobility of the polymer chains and suppresses localized plastic deformation ultimately resulting in matrix embrittlement [11]. In contrast, the addition of basalt fibers up to 15 wt% significantly enhances the impact strength of PLA/NR nanocomposites by approximately 39%. This improvement stems from the high strength and elastic modulus of basalt fibers, which enable them to effectively absorb and dissipate impact energy through multiple mechanisms, including fiber elongation, elastic deformation, and fiber pull-out. Moreover, basalt fibers act as crack arrestors by bridging crack surfaces and hindering rapid crack propagation [12]. Similarly, blending up to 30 wt% natural rubber (NR) into the PLA matrix increases impact strength by about 44%. The dispersed NR particles function as stress concentrators and energy-absorbing domains. Upon impact loading, they promote extensive energy dissipation by inducing multiple sites of plastic deformation and microcracking throughout the matrix, thereby enhancing the overall toughness of the composite.

4. Conclusions

In this study, the mechanical properties of biodegradable nanocomposites based on a polylactic acid/natural rubber (PLA/NR) matrix, reinforced with nanoclay and basalt fibers, were experimentally investigated using the response surface methodology (RSM). The key findings are summarized as follows:

- Tensile Strength: Incorporating nanoclay up to 3 wt% into the PLA/NR matrix enhanced tensile strength by approximately 10%. However, further increasing the nanoclay content beyond 3 wt% led to an 8% reduction in tensile strength, likely due to nanoparticle agglomeration. The addition of basalt fibers up to 15 wt% improved tensile strength by 14%. Conversely, the inclusion of NR (up to 30 wt%) reduced tensile strength by about 11%, consistent with its elastomeric nature.
- Elastic Modulus: The elastic modulus increased by

21% when nanoclay was added up to 3 wt%. At higher loadings (6 wt%), however, the modulus decreased by 14%, attributed to poor dispersion and agglomeration of nanoclay particles. Basalt fiber reinforcement up to 15 wt% significantly enhanced the elastic modulus by 37%. In contrast, blending NR into the PLA matrix reduced the elastic modulus by 23%, reflecting the softening effect of the rubber phase.

- Impact Strength: Impact strength exhibited a marked decline with increasing nanoclay content, indicating embrittlement at higher filler loadings. In contrast, the incorporation of basalt fibers up to 15 wt% improved impact strength by 39%. Most notably, adding NR up to 30 wt% resulted in a substantial 44% increase in impact strength, highlighting its role as an effective toughening agent in the PLA matrix.

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