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FTIR SPECTROSCOPY AND THERMAL ANALYSIS OF NATURAL RUBBER NANOCOMPOSITE SYNTHESISED FROM RSS1 GRADE NATURAL RUBBER AND NANO SILICA DERIVED FROM BAMBOO LEAVES

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ABSTRACT

Natural rubber (RSS1 grade) reinforced with silica derived from bamboo leaves ash (BLA) was developed as a sustainable biocomposite material. The biosilica was obtained through controlled calcination of bamboo leaves followed by purification to yield predominantly amorphous SiO_2. Composites containing varying filler loadings were prepared through two roll mill mixing and subsequent vulcanization. Structural interactions between the rubber matrix and bio-silica were investigated using Fourier Transform Infrared (FTIR) spectroscopy. FTIR spectra revealed characteristic absorption bands of cis-1,4-polyisoprene along with Si–O–Si stretching vibrations, confirming thesuccessful incorporation of silica without altering the fundamental structure of the rubber backbone. Thermogravimetric analysis (TGA) demonstrated improved thermal stability and increased char residue with increasing biosilica content, attributed to the barrier effect and restricted polymer chain mobility. The results indicate that bamboo leaf–derived silica can act as an effective eco-friendly reinforcing filler for RSS1 rubber, enhancing thermal resistance while supporting sustainable material development.

Keywords: Natural Rubber (RSS1), Bamboo Leaves Ash, Bio-Silica, FTIR, TGA, Green Composites.

1. INTRODUCTION

Bamboo leaves are known to contain a significant amount ofsilica, and controlled combustion of the leaves can produce amorphous silica suitable for polymer reinforcement. The incorporation of bio-silica into natural rubber can enhance thermal stability through physical reinforcement and barrier effects that restrict polymer chain mobility. Spectroscopic techniques such as Fourier TransformInfrared (FTIR) microscopy are widely used to study interactions between fillers and polymer matrices, while thermogravimetric analysis (TGA) provides valuable information about the thermal degradation behaviour ofcomposite materials.

Natural rubber (NR), chemically known as cis-1,4-polyisoprene, is widely used in industrial applications due to its excellent elasticity, resilience and mechanical strength. Commercial natural rubber is commonly available as Ribbed Smoked Sheet grade 1 (RSS1), is a high-purity elastomer obtained from Hevea brasiliensis and widely used in tires, vibration isolators, footwear, and other engineering products due [1]. However, despite its excellent mechanical properties natural rubber exhibits limited thermal stability and aging resistance. Therefore, reinforcing suitable fillers is necessary to improve its performance [2].

In recent years, bio-derived silica obtained from agricultural wastes such as rice husk ash, sugarcane bagasse ash, and bamboo leaves ash has attracted considerable attention as a sustainable and environmentally friendly alternative to conventional fillers. In this context, the present work focuses on the preparation and characterization of RSS1 natural rubber composites reinforced with bamboo leaves ash-derived silica.

Traditionally, petroleum-based fillers such as carbon black and precipitated silica have been widely used to improve the performance of natural rubber composites [3]. However, increasing environmental concerns and the demand for sustainable materials have encouraged the development of agro-waste-derived biofillers as environmentally friendly alternatives [4]. Bamboo leaf ash (BLA), produced through the controlled combustion of bamboo leaves, is an agricultural waste material rich in amorphous silica [5]. Previous studies have reported that bamboo leaves can yield approximately 60–70% silica after calcination under optimized conditions [5].

The incorporation of silica-rich BLA into the RSS1 rubber matrix can promote filler–rubber interactions through hydrogen bonding between silanol (Si–OH) groups present on the silica surface and the polyisoprene chains of natural rubber. These interactions improve stress transfer within the composite and contribute to enhanced mechanical properties [6]. In addition, silica particles can restrict the mobility of polymer chains and act as thermal barriers, thereby improving degradation resistance and increasing char formation during thermal decomposition [7].

Spectral characterization techniques such as Fourier Transform Infrared (FTIR) spectroscopy are widely used to investigate interfacial interactions and structural changes in rubber composites [8]. In FTIR spectra, the presence of Si–O–Si asymmetric stretching vibrations in the region of 1000–1100 cm^{-1} confirms the incorporation of silica into the polymer matrix, while variations in C=C stretching vibrations may indicate possible interactions between the filler and the rubber chains [8]. Thermal analysis techniques such as thermogravimetric analysis (TGA) provide valuable information regarding the degradation behavior and thermal stability of polymer composites [9]. The incorporation of silica-based biofillers often results in a slight increase in degradation temperature due to the restriction of polymer chain mobility and the barrier effect of the filler particles [7,9].

In the present study, bamboo leaf ash calcined at 650 °C was incorporated into RSS1 natural rubber at varying loadings (0–3 phr) in order to evaluate its influence on the spectral characteristics and thermal stability of the resulting composites.

2. Experimental

2.1. Material

Ribbed Smoked Sheet grade 1 natural rubber (RSS1) was procured from a licensed rubber supplier, St. Antony S Rubber, Thiruvananthapuram, Kerala, India. The rubber was used as the base elastomer without further purification. Zinc oxide (ZnO) and stearic acid, used as activators in the vulcanization system, were purchased from Central Scientific Company, Nagpur, India. N-Cyclohexyl-2-benzothiazole sulfenamide (CBS), employed as an accelerator, was imported from Malaysia and used as received. Sulphur, used as the vulcanizing agent, was also obtained from Central Scientific Company, Nagpur, India. Bamboo leaves were collected from Tadoba forest and processed to obtain bamboo leaf ash (BLA). The collected leaves were washed, dried, and calcined at 650 °C to produce silica-rich ash, which was used as the biofiller in varying concentrations (0–3 phr).

2.2. Preparation and Compounding of Nanocomposite

The compounding of RSS1 natural rubber with bamboo leaf ash (BLA) silica was carried out using a laboratory two-roll mixing mill located at Visvesvaraya National Institute of Technology (VNIT), Nagpur, India. The formulation of the rubber compounds was prepared on a parts per hundred rubber (phr) basis.

Initially, RSS1 natural rubber (100 phr) was masticated on the two-roll mill for approximately 3–4 minutes to improve its plasticity. Zinc oxide (5 phr) and stearic acid (2 phr) were then added as activators for the vulcanization system. Processing oil (5 phr) was incorporated to improve the processability of the compound and to facilitate uniform dispersion of the filler.

Bamboo leaf ash (BLA) silica was gradually introduced at different loadings (0, 1, 2, and 3 phr) and mixed thoroughly to ensure uniform dispersion within the rubber matrix. Care was taken during mixing to minimize filler agglomeration.

Subsequently, N-cyclohexyl-2-benzothiazole sulfenamide (CBS, 1 phr) was added as the accelerator, followed by sulphur (2 phr) as the curing agent. These curatives were incorporated at the final stage of mixing to prevent premature vulcanization (scorching). The total mixing time was maintained at approximately 12–15 minutes.

The compounded rubber sheets were then removed from the mill and conditioned at room temperature for 24 hours prior to vulcanization.

Table 1. Formulation of Rubber Compound

Ingredient	NR0	NR1	NR2	NR3
Natural Rubber(g)	100	100	100	100
ZnO (g)	5	5	5	5
Stearic Acid (g)	2	2	2	2
CBS (g)	1	1	1	1
|Sulphur (g)	2	2	2	2
BLA Silica (g)	0	1	2	3
Processing oil	5	5	5	5

3.Characterisation

The spectral and thermal properties of RSS1 natural rubber composites filled with bamboo leaf ash (BLA) silica were systematically evaluated. Fourier Transform Infrared (FTIR) spectroscopy confirmed the characteristic absorption bands of polyisoprene, indicating that the fundamental molecular structure of natural rubber remained unchanged after the incorporation of the bio-silica filler. No new absorption peaks were observed, suggesting that the interaction between the rubber matrix and silica-rich BLA is mainly physical rather than chemical in nature. Thermogravimetric analysis (TGA) revealed a typical single-stage thermal degradation behavior associated with natural rubber. An increase in residual ash content was observed with increasing BLA loading, confirming the presence of thermally stable inorganic silica within the composite system.

The Fourier Transform Infrared (FTIR) spectra of bamboo leaf ash–derived biosilica and RSS1 natural rubber composites were analyzed to identify the functional groups and to evaluate the interaction between the filler and the rubber matrix. The FTIR spectrum of biosilica showed a strong absorption band in the region of 1080–1100 cm^{-1}, which corresponds to the asymmetric stretching vibration of Si–O–Si bonds and confirms the presence of silica. A peak observed around 800 cm^{-1} is attributed to the symmetric stretching vibration of Si–O–Si bonds, while the band near 460 cm^{-1} represents the bending vibration of the siloxane linkage. In addition, a broad absorption band around 3400 cm^{-1} can be associated with the stretching vibration of hydroxyl (–OH) groups present on the surface of silica particles.

In the rubber composite samples, the presence of these characteristic bands confirms the successful incorporation of biosilica derived from bamboo leaf ash into the natural rubber matrix. The appearance of silica-related peaks also suggests the existence of interactions between the silica filler and the polyisoprene chains of the rubber matrix.

Thermogravimetric analysis (TGA) was carried out to evaluate the thermal stability of RSS1 natural rubber composites containing different loadings of bamboo leaf ash–derived biosilica (0, 1, 2, and 3 phr). The TGA curves indicate a small weight loss at lower temperatures, which can be attributed to the removal of moisture and other volatile components present in the material. The major weight loss occurs at higher temperatures due to the thermal degradation of the natural rubber polymer backbone.

The incorporation of biosilica slightly improved the thermal stability of the rubber composites. With increasing loading of bamboo leaf ash–derived silica, the degradation temperature shifted to slightly higher values, indicating enhanced thermal resistance. This improvement in thermal stability can be attributed to the reinforcing effect of silica particles, which restrict the mobility of polymer chains and act as a barrier during thermal degradation

4.Result and Discussion

4.1. FTIR Analysis

Fourier Transform Infrared (FTIR) spectroscopy was used to investigate the functional groups present in the silica filler and to confirm the surface modification of silica particles. The FTIR spectra (fig. 1) of natural rubber composites containing bamboo leaf ash were recorded in the range of 4000–500 cm^{-1}. The absorption bands around a frequency range of 2,930 and 2,840 are indication groups of symmetric and asymmetric stretching of the extending vibration of -CH2 group, respectively i.e vibrations of the polyisoprene backbone of natural rubber (10, 11). A peak near 1660 cm^{-1} represents C=C stretching of the rubber chain. The broad band around 3400 cm^{-1} is attributed to O–H stretching, which may arise from moisture and silica present in the biofiller. Additionally, the strong absorption band observed around 1000–1100 cm^{-1} corresponds to Si–O–Si stretching vibrations, confirming the presence of silica derived from bamboo leaf ash. These results indicate the successful incorporation of bio-silica filler into the rubber matrix.

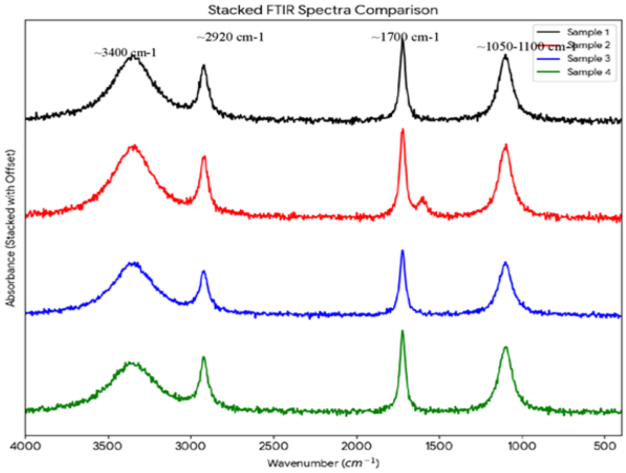

Fig. 1. FTIR of RSS1 grade nanocomposite (a) sample 1-unfilled composite (b) 1phr silica RSS1NR (c)2 phr silica in RSS1NR(d) 3phrsilica RSS1NR

Comparison of FTIR Spectra of Four Samples

Functional Group	Sample 1	Sample 2	Sample 3	Sample 4	Observation
O–H stretching (~3400 cm^{-1})	Present	Slightly broad	Strong	Reduced	Indicates variation in hydroxyl or moisture
C–H stretching (~2920 cm^{-1})	Weak	Moderate	Strong	Strong	Increase in aliphatic groups
C=O stretching (~1700 cm^{-1})	Weak	Clear peak	Strong	Slight shift	Indicates formation/interaction of carbonyl groups
C–O stretching (~1050–1100 cm^{-1})	Present	Strong	Stronger	Slightly shifted	Shows presence of alcohol/ether groups

Main Differences Between Samples

Sample 1

Shows basic functional groups.Peaks are less intense.Indicates initial or untreated material.

Sample 2

Peaks become more pronounced.Slight broadening of O–H peak.Suggests chemical interaction or modification.

Sample 3

Strongest absorption peaks.Indicates maximum interaction or functional group presence. Possibly highest concentration of active compounds.

Sample 4

Some peaks shift slightly.Peak intensity may decrease or stabilize.
Indicates structural rearrangement or stabilization of the material.

4.1. Thermogravimetric Analysis (TGA)

Thermogravimetric examination was performed to assess the thermal stability of the composites in (Fig. 2). The measure of nano silica content in the elastic composites is resolved from lingering weight level of a specific composite. Fig. 2 Thermogravimetric Curves of RSS1grade-Nanocomposite (a)0 phr Nanocomposite in RSS1(1) (b) 1 phr silica in RSS1 (2) (c) 2 phr silica in RSS1(3) (d) 3 phr silica in RSS1 (4) Thermogravimetric bends are comparative for all the elastic composites (1-4). First weight reduction saw at the temperature extend 200 0C to 463 0C is because of debasement of elastic part.

The thermogravimetric analysis showed that NR composites had one step of mass loss as shown in the weight loss curve between 330 C and 455 C [12]. At temperatures below 250 C, no thermal degradation was observed due to the scission and the crosslinking of carbon chains in rubber molecules not causing a loss of volatility in the bulk rubber [13]. From Table 2, NR composites vulcanized using EB radiation and sulfur had higher initial degradation temperatures (Td) than those of the un-vulcanized NR composites. This was because the vulcanization formed a dense crosslink structure in the NR/TSi composites [14].

For the composites containing bio-silica filler, the ash content increased compared to the unfilled rubber. The sample containing 1 g BLA showed 0.747% highly volatile matter, 92.049% medium volatile matter and 5.292% residue at 987.78 °C. Similarly, the 2 g BLA sample exhibited 0.685% highly volatile matter, 91.634% medium volatile matter and 5.736% residue at 986.21 °C, while the 3 g BLA sample showed 0.715% highly volatile matter, 91.596% medium volatile matter with 5.670% residue at 987.77 °C.

The increase in residual ash content with increasing filler loading confirms the presence of thermally stable inorganic silica derived from bamboo leaves ash in the rubber matrix. The decomposition behaviour indicates that the organic rubber components undergo thermal degradation at elevated temperatures, whereas the silica filler remains as stable residue. Therefore, the incorporation of BLA improves the thermal stability and char formation of the rubber composites, demonstrating its effectiveness as a reinforcing bio-silica filler.

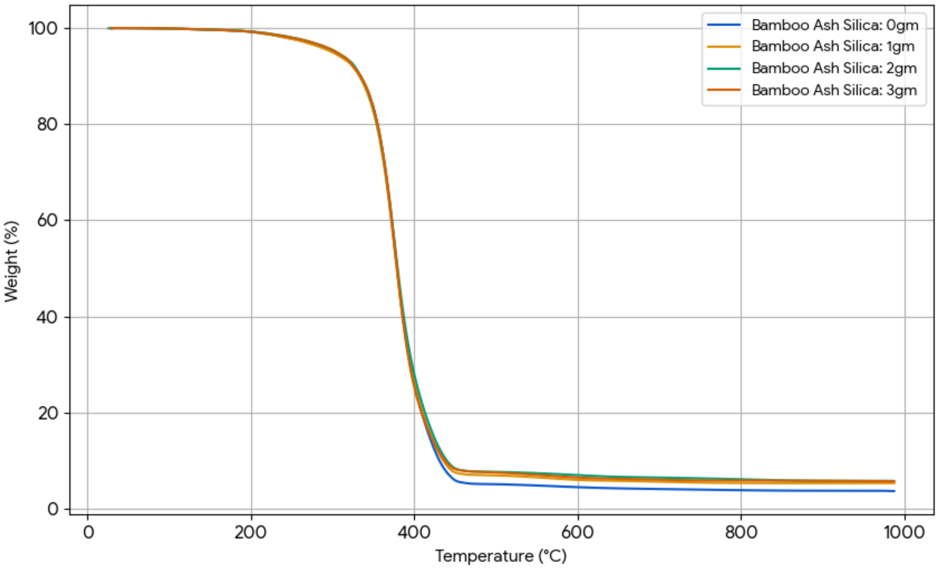

Fig. 2 Thermogravimetric Curves of RSS1 Grade Nanocomposite (a) Unfilled Composites (1) (b) 1phr silica in RSS1 Grade (2) (c) 2 phr silica in RSS1 Grade (3) (d) 3 phr silica in RSS2 Grade (4)

5. Conclusion

The present work investigated the effect of bamboo leaves ash (BLA) derived bio-silica as a reinforcing filler in RSS1 natural rubber composites. The composites were successfully prepared using a two-roll mill mixing process followed by sulfur vulcanization. FTIR analysis confirmed the presence of the characteristic functional groups of natural rubber along with the Si–O–Si stretching vibrations of silica, indicating the successful incorporation of bio-silica into the rubber matrix without altering the basic polyisoprene structure. Thermogravimetric analysis revealed that the addition of bamboo leaf ash slightly improved the thermal stability of the composites and increased the residual ash content due to the presence of thermally stable inorganic silica. The improved thermal behavior is attributed to the reinforcing and barrier effect of silica particles, which restrict the mobility of polymer chains during thermal degradation. Overall, the study demonstrates that bamboo leaves ash derived silica can serve as a promising eco-friendly reinforcing filler for natural rubber composites and provides a sustainable approach for utilizing agricultural waste in advanced rubber materials.

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