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Influence of *Hevea brasiliensis* Leaf Fillers and Clay Reinforcement on the Thermal Stability of Polymer Composites

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Abstract: This study aimed to evaluate the thermal behaviour of *Hevea brasiliensis* leaf (HBL)–polyester composites enhanced with clay and to determine how different clay loadings affect composite weight loss under high-temperature conditions. Natural fibre composites were prepared with 1%, 3%, and 5% clay, and their thermal stability was measured using Thermogravimetric Analysis (TGA) under nitrogen flow to a maximum temperature of 600 °C. Results indicate that increasing the clay content led to higher residual mass after thermal degradation, demonstrating improved thermal stability, although the onset of degradation occurred at lower temperatures for composites with higher clay content. The study indicates that clay improves the structural integrity of the composite through intercalation and lamellar interactions with HBL, while untreated fibres and the polymer matrix affect the degradation process. For future work, alkaline treatment of both HBL and clay is recommended to achieve improved bonding and thermal properties.

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1. INTRODUCTION

Usage of composite materials has grown tremendously, with applications spanning construction, electronics, furniture, and transportation [1]. Composites are produced by merging multiple distinct materials, resulting in a material with improved or unique characteristics [2]. Fibre reinforcement has been recognised as among the best ways to improve how composites work, especially when it comes to strength

and heat resistance [3]. Traditionally, researchers have focused on synthetic fibres as reinforcement. However, natural fibres have recently gained greater attention because of their cost-effectiveness, light weight, biodegradability, renewable and environmental compatibility [4]. The transition from synthetic to natural fibres is further driven by growing global recognition of environmental sustainability and the need to minimise ecological impact [4]. The use of natural fibres contributes to lower pollutant emissions and reduced greenhouse gas generation, positioning them as a viable substitute for synthetic materials [5].

Recently, the growing demand for environmentally friendly composites in the automotive industry, along with the ecological advantages of natural fibres, has intensified research interest in this area [6]. Numerous studies have explored the improvement of polymer composite properties by incorporating natural fibres, including abaca, coir, cotton, flax, hemp, jute, silk, sisal, and wool [7]. A study by Hamidon et al. [8] looked at how kenaf fibre can make thermoplastic polyurethane (TPU) stronger. They tested pure TPU and TPU mixed with 20%, 30%, 40%, and 50% kenaf fibre. The results showed that adding more fibre made the composite materials less stable under heat, although they were still better at handling heat than the kenaf fibre alone. Another study by Bharadwaj et al. [9] found that pure polyester degraded faster than clay-based nanocomposites at temperatures below 400 °C, but the nanocomposites exhibited slower degradation at higher temperatures. Similarly, research on sisal–nanoclay composites by Venkatram et al. [10] found that unreinforced composites degraded at approximately 500 °C, whereas clay-reinforced composites showed delayed degradation at about 510 °C.

Based on previous research, this study aims to make polyester composites stronger against heat by using *Hevea brasiliensis* leaves (HBL) through adjustments in clay content. Additionally, the study looks at how different amounts of clay affect how much natural fibre composites lose weight when exposed to high temperatures. The samples were analysed using Thermogravimetric Analysis (TGA), and the results are discussed in detail.

2. METHODOLOGY

2.1 Materials

HBL, which served as the natural fibre in the composite, was collected from Muar, Johor, Malaysia. Polyester resin, bought from a private company, acted as the main material. Methyl Ethyl Ketone Peroxide (MEKP), a substance used to help harden materials and start the process of making them solid, was also purchased from the same private company. Clay, used as the filler material, was obtained from a claypot shop in Perak, Malaysia.

The mould used for specimen preparation was fabricated using plasticine, using a 600 mL mineral water bottle cap as the shaping template. TGA was used to assess the composites' thermal degradation by monitoring mass loss resulting from decomposition, dehydration, or oxidation [10–11]. The analysis was performed using a PerkinElmer 8000 model instrument.

2.2 Preparation of HBL

The collected leaves were prepared for incorporation into the polymer–clay composite. The leaves were first oven-dried at 100 °C for 1 hour using a UN110 oven to reduce moisture content. The dried leaves were then ground using a GM200 grinder at 4 rpm for 2 minutes to produce fine powder. Grinding was repeated until complete pulverisation was achieved. The resulting powder was sieved using an AS200 sieve for 10 minutes at an amplitude of 50 mm to obtain particles with an average size of 63 μm.

2.3 Preparation of Natural Fibre Composite Samples

Each composite formulation consisted of 50 g of material. An empty beaker was weighed before preparation. The baseline mixture included 40 g of polyester resin and 10 g of HBL powder. Clay and MEKP were measured at 0.5 g and 0.75 g, corresponding to 1 wt% clay and 1.5 wt% MEKP relative to the total composite mass.

After weighing, all ingredients were combined in a beaker. The HBL and clay were added to the polyester resin and stirred until equally dispersed. Subsequently, MEKP (1.5 wt%) was gently incorporated into the mixture and stirred to initiate curing and allow the composite to solidify. Once homogenised, the mixture was poured into the mould and allowed to dry for 24 hours at room temperature.

All steps were repeated for formulations containing 3 wt% and 5 wt% clay.

2.4 Characterisation Using TGA

The analysis was conducted using TGA, which measures changes in the sample's weight to gauge its thermal stability as the temperature increases. About 10 mg of each sample was loaded into a ceramic pan and heated under a nitrogen atmosphere to 600 °C [12], at a rate of 10 °C/min, with a gas flow of 20 mL/min.

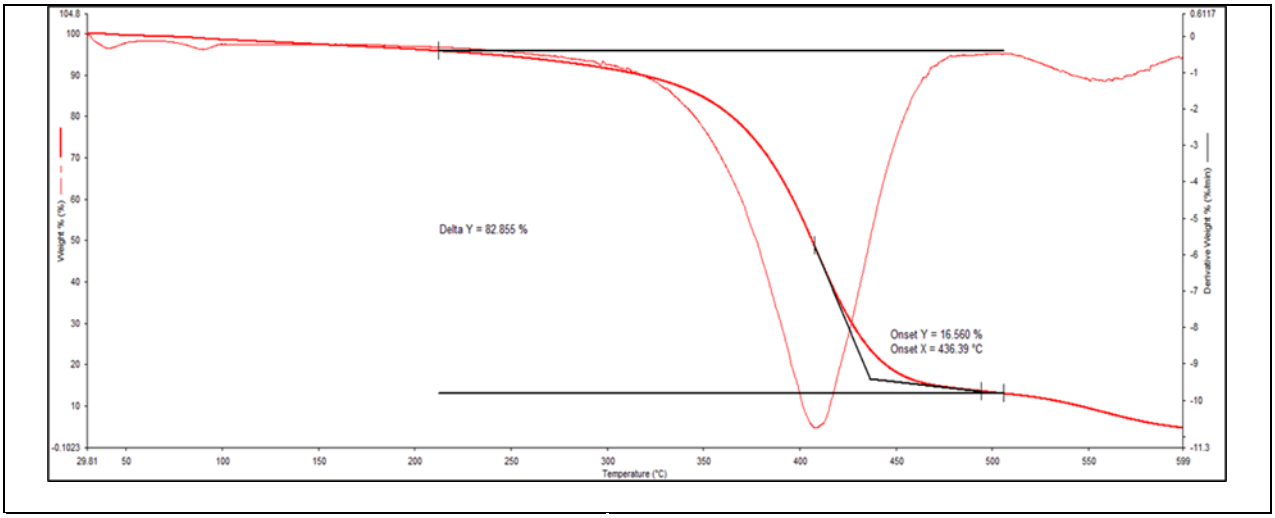

Figure 1. Thermal degradation of natural fibre composite with 1% clay.

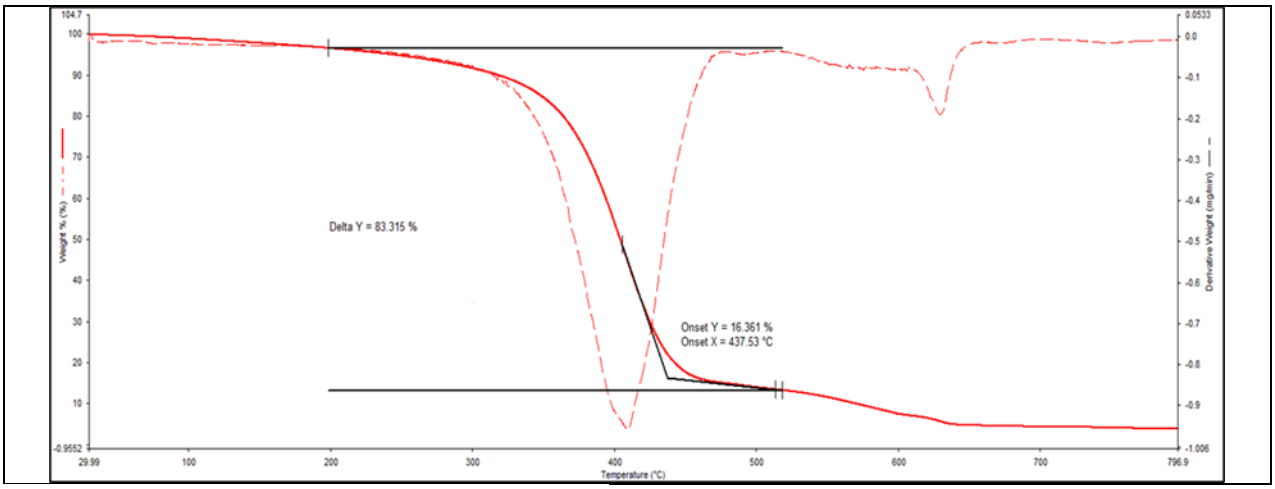

Figure 2. Thermal degradation of natural fibre composite with 3% clay.

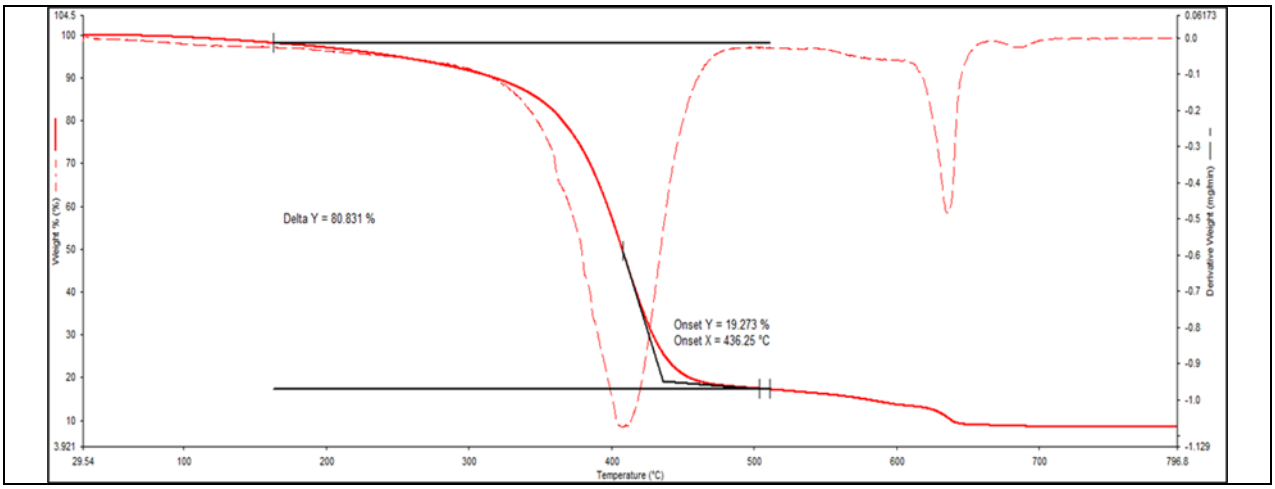

Figure 3. Thermal degradation of natural fibre composite with 5% clay.

3. RESULTS AND DISCUSSION

The thermal behaviour of the natural fibre composite containing 1% clay heated under a nitrogen atmosphere up to 600 °C is shown in Figure 1. Degradation of the composite commenced between 200 °C and 250 °C. At 598.98 °C, the sample retained 4.67 wt% of its original mass. The delta-Y value indicates that the composite experienced an overall weight loss of 82.855%, with the most rapid degradation occurring between 200–250 °C and continuing until approximately 400–450 °C. The recorded residue mass was 0.467 mg out of the original 10 mg sample.

The thermal degradation profile for the composite with 3% clay is shown in Figure 2. The slope of the degradation curve is similar to that observed for the 1% clay composite. The delta-Y value shows an 83.315% weight loss, with degradation mostly occurring in the temperature range of 200–450 °C. At 600 °C, the remaining mass was 7.54 wt%, corresponding to 0.754 mg of the original 10 mg sample.

Figure 3 illustrates the TGA curve for the composite containing 5% clay. The sample began to degrade earlier, at approximately 150–200 °C, which is lower than the degradation onset temperatures for the 1% and 3% clay composites. The sharp degradation phase occurred between 350 °C and 400 °C. The delta-Y value was 80.831%, and the final residue at 600 °C was 13.73 wt% (1.37 mg).

Comparing the results from Figures 1 to 3, it is evident that increasing the clay content results in a higher residual weight at 600 °C. These results indicate that clay improves the composite system's thermal stability. Among the tested formulations, the composite containing 5% clay exhibited the highest residual mass, followed by the 3% and 1% clay composites, suggesting that 5% clay provided the greatest resistance to thermal degradation. However, the overall trend in the degradation onset temperature did not follow the initial expectation of the study.

Composites with higher clay content exhibited earlier degradation onset temperatures compared to those with lower clay content (Table 1). This behaviour is attributed to the poor compatibility between the untreated clay and HBL with the hydrophobic polymer matrix. Both clay and HBL are hydrophilic materials, which reduces their interfacial bonding efficiency with polyester resin [1, 13–14]. Despite this weak interaction, the clay and HBL were sufficiently dispersed within the polymer matrix to form workable composites.

Table 1 - Summary of TGA results

Clay Content (%)	Weight Loss (wt%)	Degradation Onset (°C)
1	95.33	200 – 250
3	92.46	200
5	86.27	150 – 200

Due to the limited availability of published studies involving untreated clay and HBL, interpretations were made based on observed behaviour. For the composite with 1% clay, the delayed degradation onset suggests that the fibre–clay ratio was still sufficiently low to allow effective adsorption onto the polymer layer, resulting in relatively stronger bonding. Consequently, higher temperatures were required to initiate degradation. For the composite containing 3% clay, partial adsorption still occurred, but some components were not strongly bonded to the polymer, resulting in an earlier onset of deterioration. In the composite with 5% clay, excess clay and HBL content, combined with poor interfacial adhesion, allowed the unbound components to degrade at the lowest temperature range.

In all samples, the initial shallow slope of the TGA curves corresponds to the breakdown of loosely bound clay and HBL components, while the steeper region reflects the degradation of the primary composite structure comprising polymer, clay, and HBL.

Although composites with higher clay content degraded earlier, they exhibited lower overall weight loss, resulting in higher residual mass at 600 °C. This is because clay consists of a lamellar structure containing interlayer spaces that allow natural fibres to be accommodated or intercalated within these spaces [15]. The increased intercalation enhances structural integrity and requires higher energy for complete decomposition, thereby reducing total weight loss. Thus, higher clay content increases the number of interlayer sheets available, improving the composite's thermal stability.

Malaysian clay typically consists of kaolinite, illite, and smectite, each of which contributes unique structural characteristics that reinforce composite stability [16–17]. Kaolinite consists of alternating tetrahedral (Si–O) and octahedral (Al–OH) sheet layers arranged in a 1:1 lamellar structure held together by hydrogen bonding [16, 18–19]. Illite and smectite minerals exhibit a 2:1 structure, with octahedral sheets sandwiched between two tetrahedral sheets [20–21]. The stacked hydroxyl-rich interlayers of these minerals allow interaction with organic materials such as HBL, contributing to their suitability as fillers [15].

These minerals also possess inherent chemical inertness, allowing clay-based composites to withstand high temperatures during thermal testing [16]. This explains why composites containing higher clay

percentages, particularly the 5% clay formulation, demonstrated superior overall thermal stability.

4. CONCLUSION

This study shows that clay helps improve the heat stability of polyester materials strengthened with *Hevea brasiliensis* leaves (HBL). Composites with higher clay content exhibited greater residual mass at elevated temperatures, indicating enhanced thermal resistance, although the onset of degradation occurred at lower temperatures for formulations with higher clay loading. The presence of HBL also contributed positively to the composite's structural integrity and overall stability.

To optimise the performance of the composite, it is recommended to treat both HBL and clay with a basic substance, like sodium hydroxide (NaOH), to enhance their structural properties. In addition, analysing the volatile fractions of HBL and polyester separately would provide a clearer understanding of their individual contributions to overall weight loss and thermal behaviour. Implementing these modifications would help refine the composite's properties and support its potential for broader application across various industrial sectors.

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