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Morphology and Thermal Behavior of Natural Fiber-Reinforced Recycled HDPE Composites Using Bentonite, Fly Ash, and Rice Husk Ash Fillers

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ABSTRACT

Plastic waste accumulation has become a critical environmental concern, requiring sustainable recycling strategies. Recycled high-density polyethylene (rHDPE) has attracted attention as a potential polymer matrix due to its ability to reduce plastic waste while maintaining desirable material properties. However, the performance of rHDPE composites is strongly influenced by filler type. This study investigated the effects of bentonite, fly ash, and rice husk ash fillers, each incorporated at 5% by weight, on the morphology and fire performance of short cocofiber-reinforced rHDPE composites. Composite specimens were fabricated through thermal molding in an oven without external pressure. Morphological characteristics were analyzed using Scanning Electron Microscopy (SEM), while fire performance was evaluated using the ASTM D635 horizontal burning test based on Time to Ignition (TTI) and Burning Rate. A control specimen (rHDPE reinforced with cocofiber without filler) was also tested for comparison. The results showed that filler type significantly affected the morphology and fire performance of composites. Compared with the control specimen, which exhibited an average TTI of 6.3 s and the lowest Burning Rate of 16.88 mm/min, rice husk ash exhibited the highest average TTI of 8.13 s and the lowest Burning Rate of 14.97 mm/min,

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indicating a marked improvement in fire resistance. SEM analysis revealed that rice husk ash improved filler distribution, reduced void formation and particle agglomeration, and enhanced filler–matrix interaction, resulting in a denser microstructure than the other filler types and the control. These characteristics may have contributed to improved fire performance by reducing heat transfer and delaying ignition during combustion. Overall, rice husk ash demonstrated promising potential as a sustainable filler for enhancing the microstructure and fire performance of short cocofiber-reinforced rHDPE composites.

Introduction

Plastic consumption based on synthetic polymers has increased substantially over recent decades, resulting in the accumulation of large quantities of plastic waste and posing one of the major global environmental challenges. Due to its resistance to natural degradation, plastic waste can persist in the environment, causing soil and aquatic pollution while threatening ecosystem sustainability [1]. The proportion of plastic waste continues to increase annually and has become one of the major contributors to municipal solid waste, highlighting the urgent need for sustainable waste management strategies. According to the Indonesian Ministry of Environment and Forestry (KLHK), plastic waste accounted for 19.92% of the total national municipal solid waste generated in 2025 [2]. This situation underscores the importance of implementing effective plastic waste management approaches, particularly recycling, to reduce waste accumulation and mitigate its associated environmental impacts [3].

High-density polyethylene (HDPE) is one of the most widely used petroleum-based thermoplastic polymers owing to its excellent mechanical performance, high chemical resistance, and relatively low production cost, making it suitable for a broad range of industrial and household applications [4]. However, HDPE is derived from non-renewable fossil resources and exhibits poor biodegradability, resulting in persistent environmental pollution after disposal. The continuously increasing volume of post-consumer HDPE waste has therefore become a major challenge for waste management systems. Consequently, recycling post-consumer HDPE into recycled high-density polyethylene (rHDPE) has emerged as a promising strategy for valorizing plastic waste, reducing the demand for virgin fossil-based polymers, and supporting the implementation of circular economy principles [5].

Mechanical recycling has become one of the most widely adopted approaches for converting post-consumer HDPE into recycled high-density polyethylene (rHDPE) and mitigating plastic waste accumulation [6]. The utilization of rHDPE as a polymer matrix not only provides environmental benefits by reducing the consumption of virgin fossil-based polymers and lowering emissions associated with new polymer production, but also enhances the value of post-consumer plastic waste. Nevertheless, repeated service life and recycling processes may induce material degradation, resulting in the deterioration of mechanical properties and thermal stability [7]. Such degradation remains a major challenge in the development of rHDPE-based composites, necessitating effective reinforcement strategies to restore or enhance material performance. Accordingly, the incorporation of reinforcing materials has attracted considerable attention as an effective strategy for enhancing the performance of rHDPE composites and supporting the development of sustainable engineering materials.

In this study, short coconut fiber (cocofiber), with an average fiber length of ±3 mm, was selected as the natural fiber reinforcement owing to its wide availability, low cost, low density, and favorable mechanical properties making it a promising candidate for sustainable polymer composite development. The incorporation of bentonite, fly ash, and rice husk ash as fillers in polymer composites has attracted increasing research interest due to their potential to modify the composite morphology and enhance fire performance. Bentonite is a naturally occurring layered aluminosilicate mineral that is environmentally friendly, abundant, and relatively inexpensive [8]. These characteristics make bentonite an attractive filler for polymer composite applications. In addition, fly ash and rice husk ash, which are industrial and agricultural by-products, respectively, represent promising value-added fillers because their utilization enhances the value of materials that are otherwise underutilized [9], [10]. However, differences in the physical characteristics and chemical composition of these fillers are expected to produce distinct effects on the morphology and fire performance of the resulting composites. Therefore, a systematic comparison of these fillers is essential to better understand their effectiveness in tailoring the morphology and fire performance of natural fiber-reinforced rHDPE composites.

Recent studies have demonstrated that the incorporation of different inorganic fillers significantly influences the mechanical and physical properties of recycled HDPE composites. The type and composition of fillers have been reported to affect tensile strength, fracture behavior, impact resistance, density, and other physical characteristics of natural fiber-reinforced HDPE composites. For example, bentonite clay was found to improve the impact strength and density of recycled HDPE composites reinforced with jute fibers [11], while hydroxyapatite enhanced the physical and impact behavior of woven ramie-reinforced HDPE composites [12]. Similarly, the incorporation of eggshell waste filler has been shown to improve the impact strength and physical properties of recycled HDPE composites [13]. In addition, the selection of filler type and volume fraction significantly influences the tensile and fracture behavior of HDPE–ramie composites [14]. These findings highlight the important role of filler selection in tailoring the overall performance of recycled HDPE composites and provide a foundation for further investigations focusing on their morphology and fire performance.

Beyond mechanical and physical properties, several studies have also investigated the influence of inorganic fillers on the morphology and thermal behavior of recycled HDPE composites. A previous study reported that natural fiber-reinforced recycled HDPE composites incorporating *Pennisetum setaceum* fibers can be mechanically recycled for up to five cycles with only limited deterioration in their material properties [15]. In addition, bentonite has been reported as a promising filler for enhancing the thermal stability, improving the composite morphology, and strengthening the interfacial interaction between the filler and the polymer matrix [16]. Likewise, the incorporation of fly ash into HDPE composites has been shown to influence both the morphology and thermal stability through improved particle dispersion and changes in the degree of crystallinity [17]. Similarly, rice husk ash has been reported to modify the morphological characteristics of HDPE composites by filling microvoids within the polymer matrix, resulting in a denser composite structure [18]. Although bentonite, fly ash, and rice husk ash have been extensively investigated as fillers for polymer composites, previous studies have primarily focused on their mechanical, physical, or thermal properties individually. However, studies directly comparing the effectiveness of these three fillers in natural fiber-reinforced recycled HDPE composites under identical processing conditions remain scarce, particularly in terms of morphology and fire performance.

Therefore, a systematic investigation under identical material compositions and testing conditions is required to evaluate the effectiveness of each filler in modifying the composite microstructure and improving fire performance [19]. Furthermore, understanding the interaction between natural fibers and different fillers is essential for optimizing the morphological characteristics and fire performance of rHDPE composites intended for engineering applications. Based on these considerations, this study aims to evaluate the effects of bentonite, fly ash, and rice husk ash fillers on the morphological characteristics and fire performance of natural fiber-reinforced rHDPE composites.

This study is expected to provide scientific insight into the role of natural fibers and filler type in determining the morphology and fire performance of rHDPE composites, while also serving as a practical and cost-effective reference for developing sustainable recycled polymer composites from plastic waste, industrial by-products, and agricultural residues, and forming a basis for future research in this field.

Material and Methods

This study employed an experimental laboratory approach to investigate the effects of different filler types on the morphology and fire performance of natural fiber-reinforced recycled high-density polyethylene (rHDPE) composites. Bentonite, fly ash, and rice husk ash were used as filler materials, while rHDPE served as the polymer matrix and natural fibers served as reinforcement. Composite specimens were fabricated using predetermined material compositions and subsequently characterized using Scanning Electron Microscopy (SEM) and horizontal burning tests in accordance with ASTM D635.

Materials. Post-consumer high-density polyethylene (HDPE) waste was used as the polymer matrix in this study. The collected HDPE waste was cleaned and subsequently processed into pellets prior to composite fabrication.

Natural fibers used as reinforcement in this study were short coconut fibers (cocofiber) with an average length of ±3 mm, selected as reinforcement due to their wide availability, low cost, low density, and favorable mechanical properties for polymer composite applications. Prior to composite fabrication, the cocofiber was subjected to alkali (NaOH) treatment to improve interfacial adhesion between the fiber and polymer matrix. The fibers were immersed in a 5% NaOH solution for 5 hours at room temperature, followed by thorought rinsing with distilled water until a neutral pH was achieved, and subsequently oven-dried at 200ºC for 2 hours prior to use. This alkali treatment is expected to remove surface roughness and enhancing mechanical interlocking with the rHDPE matrix.

Bentonite, fly ash, and rice husk ash were used as filler materials to evaluate their effects on the morphology and fire performance of rHDPE composites. To ensure compatibiltity with the short cocofiber reinforcement and to promote homogeneous dispersion within the rHDPE matrix, the filler particles were sieved to a particle size significantly smaller than the fiber length, i.e., in the range of approximately 100-250 μm (equivalent to 60-140 mesh). This particle size range was selected so that the filler could occupy the interfibrillar spaces around the ±3 mm cocofiber without disrupting fiber wetting or matrix continuity during fabrication, thereby facilitating more uniform filler distribution within the composite.

Composite Fabrication. Natural fiber-reinforced recycled high-density polyethylene (rHDPE) composites containing bentonite, fly ash, and rice husk ash as fillers were fabricated by heating the constituent materials in molds according to the predetermined compositions. Each composite specimen was formulated with a fixed matrix-to-reinforcement ratio consisting of 80 wt% rHDPE and 15 wt% cocofiber, with each filler (bentonite, fly ash, or rice husk ash) incorporated at 5 wt% relative to the total composite weight, replacing an equivalent fraction of the rHDPE matrix. A control specimen, consisting of 80 wt% rHDPE and 15 wt% cocofiber without any filler, was also prepared for comparison. The composite fabrication procedure consisted of the following steps:

1. Preparation of the constituent materials.
2. Weighing the materials according to the predetermined compositions.
3. Arranging the rHDPE, natural fibers, and fillers in the molds according to the predetermined compositions.
4. Heating the molds in an oven at 200 ºC for 2 hours to melt the rHDPE and allow homogeneous mixing of the constituents.
5. Cooling the molds to room temperature.
6. Removing the composite specimens from the molds.

Analysis of Scanning Electron Microscopy (SEM). The morphological characteristics of the composites were characterized using Scanning Electron Microscopy (SEM). The analysis was conducted to observe the surface morphology and fracture characteristics of natural fiber-reinforced recycled high-density polyethylene (rHDPE) composites. Particular attention was given to evaluating the interfacial adhesion between the polymer matrix, natural fibers, and fillers, as well as identifying filler dispersion, void formation, particle agglomeration, and fracture mechanisms.

Prior to SEM observation, the specimens were cut into appropriate dimensions and dried to eliminate residual moisture. When necessary, the specimen surfaces were coated with a thin conductive layer using a sputter coater to improve electrical conductivity and obtain high-quality micrographs. SEM observations were carried out at various magnifications depending on the analysis requirements. The acquired SEM micrographs were qualitatively analyzed to assess filler dispersion, fiber–matrix interfacial adhesion, void formation, particle agglomeration, and fracture surface characteristics.

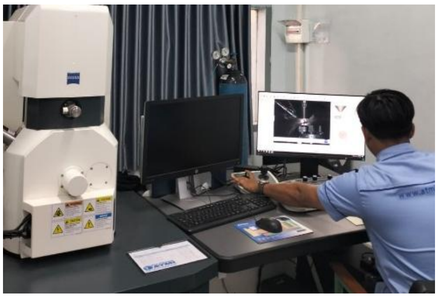

Figure 1. Scanning Electron Microscope instrument

Horizontal Burning Test. The horizontal burning test was conducted to evaluate the fire performance of recycled high-density polyethylene (rHDPE)-based composites. The test was performed in accordance with ASTM D635, which specifies the standard test method for determining the burning rate, extent, and duration of burning of plastics in a horizontal position.

Composite specimens were prepared according to the dimensions specified in ASTM D635 and placed in a horizontal position during testing. One end of each specimen was exposed to a flame for the ignition duration specified in the standard. After removal of the ignition source, the flame propagation time across the specified distance was recorded to determine the linear burning rate of the composites.

Figure 2. Horizontal burning test setup

The linear burning rate was calculated using the following equation:

$$V = \frac{60L}{t} \qquad (1)$$

Where:
V = linear burning rate (mm/min),
L = burning length (mm),
t = burning time (s).
The obtained results were used to evaluate the effects of bentonite, fly ash, and rice husk ash fillers on the fire performance of recycled HDPE-based composites.

Result and Discussion

Morphological Characteristics of rHDPE Composites. The fracture surface morphology of natural fiber-reinforced recycled high-density polyethylene (rHDPE) composites containing bentonite, fly ash, and rice husk ash fillers is presented in Figure 3,4, and 5. All SEM micrographs were obtained at a magnification of 1,000× to enable a consistent comparison of the morphological characteristics among the composites. The micrographs reveal differences in filler dispersion, void formation, particle agglomeration, and filler-matrix interfacial adhesion depending on the filler type. These morphological variations indicate that each filler exhibits different interactions with the rHDPE matrix, which may influence the overall composite performance.

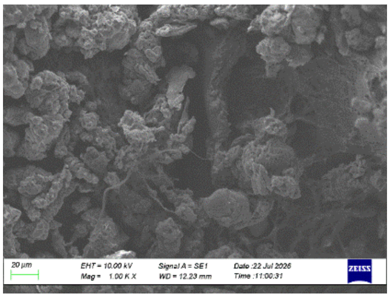

Figure 3. SEM image of the bentonite composite.

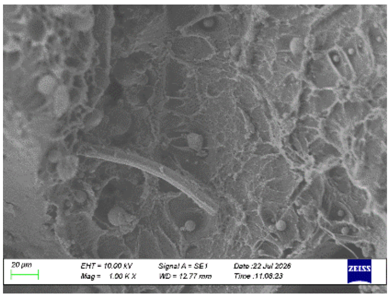

Figure 4. SEM image of the fly ash composite.

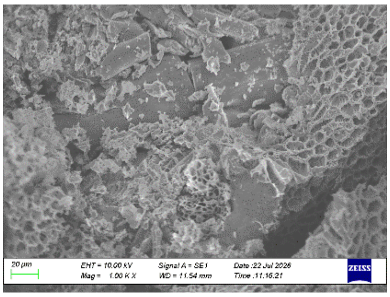

Figure 5. SEM image of the rice husk ash composite.

The bentonite-filled composite exhibited a relatively rough fracture surface characterized by pronounced particle agglomeration, numerous voids, and interfacial gaps between the filler particles and the polymer matrix. These features indicate that the dispersion of bentonite within the rHDPE matrix was relatively non–uniform. The presence of interfacial defects suggests weaker filler-matrix interaction, resulting in microstructural discontinuities that may facilitate crack propagation and increase the accessibility of heat and oxygen during combustion.

Compared with the bentonite-filled composite, the fly ash-filled composite exhibited a more homogeneous particle distribution with fewer agglomerates and voids. The fracture surface appeared denser, indicating improved contact between filler particles and the polymer matrix. These morphological characteristics suggest stronger filler-matrix interaction, which may reduce microstructural defects and contribute to improved fire performance.

Among the investigated fillers, the rice husk ash-filled composite exhibited the most homogeneous morphology. The filler particles were more uniformly dispersed throughout the matrix, accompanied by fewer voids and reduced particle agglomeration. In addition, the fracture surface appeared more compact, indicating stronger interactions among the filler, natural fibers, and polymer matrix. Such a compact microstructure may reduce pathways for heat transfer and oxygen diffusion, thereby contributing to improved resistance against ignition and flame propagation.

Overall, the SEM analysis demonstrates that the filler type plays an important role in determining the morphological characteristics of natural fiber-reinforced recycled HDPE composites. Compared with bentonite and fly ash, the incorporation of rice husk ash produced a more homogeneous microstructure with improved filler–matrix interaction and fewer microstructural defects. These characteristics may contribute to differences in fire performance, which are discussed in the following section.

Horizontal Burning Test.

Figure 6. Horizontal burning test spesiment.

The horizontal burning test was conducted to evaluate the fire resistance of natural fiber-reinforced recycled high-density polyethylene (rHDPE) composites containing bentonite, fly ash, and rice husk ash fillers. A control specimen (rHDPE reinforced with cocofiber without filler) was also tested under identical conditions to serve as a baseline for evaluating the effectiveness of each filler. The evaluated parameters included Time to Ignition (TTI) and Burning Rate, which were used to assess the ignition resistance and flame propagation behavior of the composites.

Figure 7. Horizontal burning test procedure.

The results of the Time to Ignition (TTI) and Burning Rate tests are presented in Tables 1 and 2.

Table 1. Time to Ignition of the composites.

Filler	Sample	TTI (s)
Bentonite	A	6.8
	B	6.6
	C	6.5
Fly Ash	A	7.6
	B	7.4
	C	7.3
Rice Husk Ash	A	8.3
	B	8.1
	C	8

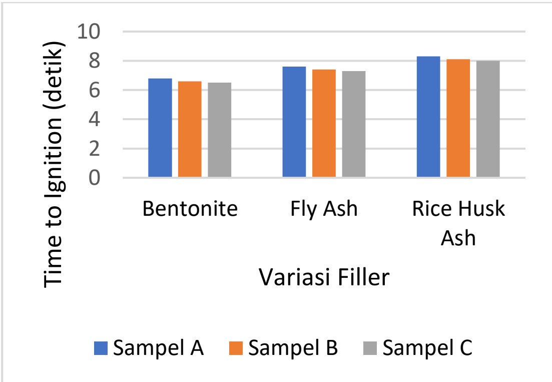

Figure 8. Time to Ignition values of the composites.

The composites filled with bentonite, fly ash, and rice husk ash exhibited average TTI values of 6.63 s, 7.43 s, and 8.13 s, respectively, compared with 6.3 s for the unfilled control specimen. Among the three fillers, the rice husk ash-filled composite exhibited the longest ignition time, indicating superior resistance to ignition. A higher TTI value implies that the material requires greater thermal energy to initiate combustion, thereby enhancing its resistance to ignition. These findings demonstrate that incorporating rice husk ash is more effective in improving the fire resistance of the composite than incorporating bentonite or fly ash, and that all three fillers improved ignition resistance relative to the unfilled control.

Table 2. Burning Rate of the composites.

Filler	Sample	Burning Rate mm/min
Bentonite	A	19.35
	B	17.91
	C	17.14
Fly Ash	A	17.65
	B	16.22
	C	15.38
Rice Husk Ash	A	16.26
	B	14.63
	C	14.02

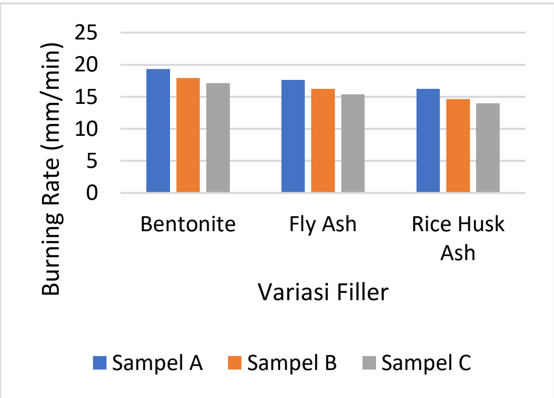

Figure 9. Burning Rate values of the composites.

The average Burning Rate values for composites containing bentonite, fly ash, and rice husk ash were 18.13 mm/min, 16.42 mm/min, and 14.97 mm/min, respectively, compared with 16.88 mm/min for the unfilled control specimen. The progressive reduction in Burning Rate indicates a slower flame propagation along the composite surface, reflecting improved resistance to flame spread. Consequently, the composite containing rice husk ash exhibited the highest fire resistance by suppressing flame propagation more effectively than the fly ash- and bentonite-filled composites. Furthermore, the results reveal an inverse relationship between TTI and Burning Rate, where a longer ignition time corresponds to a lower flame propagation rate.

The enhanced fire resistance of the rice husk ash-filled composite can be attributed to its high silica (SiO_2) content. During combustion, the silica-rich layer acts as a protective thermal barrier that limits heat transfer and suppresses the release of volatile degradation products from the polymer matrix. As a result, thermal degradation proceeds more slowly, leading to delayed ignition and reduced flame propagation. Fly ash also contributes to improved fire resistance due to the presence of thermally stable inorganic oxides, including SiO_2, Al_2O_3, and Fe_2O_3. However, its fire-retardant effect remained lower than that of rice husk ash, which generally contains a higher silica content capable of forming a more effective protective barrier during combustion. In contrast, the bentonite-filled composite exhibited the highest Burning Rate and the lowest TTI, indicating that bentonite was less effective than the other fillers in enhancing the fire resistance of the rHDPE composite under the present experimental conditions.

Overall, the horizontal burning test demonstrates that the filler type significantly influences the fire resistance of natural fiber-reinforced recycled HDPE composites. The overall fire resistance was ranked as follows: rice husk ash > fly ash > bentonite, as evidenced by higher TTI values and lower Burning Rate values. These findings indicate that rice husk ash is the most effective filler for enhancing the fire resistance of rHDPE composites by delaying ignition and reducing flame propagation.

Conclusion

This study demonstrates that the incorporation of different inorganic fillers significantly influences the morphology and fire performance of natural fiber-reinforced recycled high-density polyethylene (rHDPE) composites. Among the fillers investigated, rice husk ash exhibited the best overall performance, showing the most significant improvement in fire resistance compared with bentonite and fly ash, as reflected by the longest ignition time and the slowest flame propagation rate among the three fillers. Morphological characterization using Scanning Electron Microscopy (SEM) revealed that the rice husk ash-filled composite exhibited a more homogeneous filler distribution, fewer voids and particle agglomerates, and improved filler-matrix interfacial adhesion. This compact microstructure may have contributed to improved fire resistance by restricting heat transfer and delaying ignition during combustion. Overall, rice husk ash proved to be the most effective filler for improving the morphology and fire resistance of cocofiber-reinforced rHDPE composites, highlighting its potential as an environmentally sustainable filler for the development of polymer composites with enhanced fire resistance. Future research is recommended to further evaluate the mechanical properties (e.g., tensile, flexural, and impact strenght) of rice husk ash-filled rHDPE composites, in order to provide a more comprehensive assessment of their safety and suitability for practical engineering applications.

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