Jurnal Kejuruteraan 38(7) 2026: 3427-3435
https://doi.org/10.17576/jkukm-2026-38(7)-21

Effect of Oil Palm Frond-Based Biochar on Natural Rubber Latex Foam Composites Physical Properties, Flammability and Thermal Stability

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Received 2 January 2026, Received in revised form 25 June 2026
Accepted 25 July 2026, Available online 30 October 2026

ABSTRACT

Natural rubber (NR) latex foam is a favorable rubber foam due to the unique properties and characteristics of NR. NR latex foam, however, has poor thermal stability and flammability. The favorable properties are attributed to the cis-1,4 isoprene repeating units, while the allylic carbon of 1,4 isoprene leads to poor thermal stability and flammability. A flame retardant such as oil palm frond-based biochar (OPFB) is a potential, sustainable solution. This study focuses on the effect of OPFB on low-ammonia NR latex foam composites. Within the OPFB loadings studied (0-30 phr), the density increased while the volume shrinkage decreased with OPFB loading, due to a higher solid-to-air-phase ratio. A high cross-link density of the foam, even without OPFB, is hypothesized to have led to no significant effect on hardness. The flammability, tested according to the UL 94 horizontal burning foamed material test, showed that OPFB reduced flammability. However, even at 30 phr, the foam composites were still not able to meet the UL 94 requirements of < 40 mm/min. Thermogravimetric analysis showed better thermal stability with OPFB loadings. Thus, it can be concluded that OPFB is a potential sustainable filler in the applications of NR latex foams which require thermal stability.

Keywords: Oil palm frond biochar; natural rubber latex foam; density; flammability; TGA

INTRODUCTION

Natural rubber (NR) latex is obtained from the sap of the *Hevea brasiliensis* tree, which remains one of the most economically important plantation crops in tropical countries, particularly in Southeast Asia. Commercial NR latex undergoes a concentration process to increase its dry rubber content before being used in the manufacture of various latex-based products. The latex concentrates are preserved with ammonia only or with a combination of ammonia and secondary preservative(s) to prevent microbial degradation and maintain storage stability (Blackley 1997). Ammonia is the most widely used preservative due to its strong alkaline and biocidal properties, cost effectiveness, and ease of availability. Depending on the intended application, different preservation systems are employed to ensure the stability and quality of the concentrated latex during transportation and processing. Table 1 summarizes the types of preservatives used in concentrated NR latex (Gazely et al. 2005). Among the numerous products manufactured from concentrated NR latex, NR latex foam represents one of the most commercially significant applications because of its unique combination of elasticity, comfort and durability.

Foams are porous, lightweight and relatively soft materials consisting of interconnected or closed cellular structures. Their highly porous architecture contributes to excellent cushioning characteristics, low density, and superior energy absorption capabilities. The soft and porous structure enables their use in applications that require noise absorption, vibration damping and impact cushioning. Some commercially available foams in the market include NR foam, polyurethane foam and styrene butadiene foam (Choi et al. 2004; Mostafa et al. 2015; Vahidifar et al. 2016; Shao et al. 2016; Rostami-Tapeh-Esmaeil et al. 2021). Among these materials, NR foam possesses a higher commercial value because it is produced from a renewable

natural resource and exhibits excellent elasticity, resilience, flexibility, tear resistance and damping properties (Ariff et al. 2020). In addition, the open-cell structure of NR latex foam provides excellent breathability and moisture permeability, enhancing user comfort in long-term applications. Compared to petroleum-based polymeric foams, NR latex foam also offers a lower environmental footprint because it originates from renewable biomass and can potentially contribute to more sustainable material development.

TABLE 1. Types of preservatives in concentrated NR latex (Gazely *et al.* 2005)

Name (Abbreviation)	Preservative system (% by weight)
High or full ammonia (HA)	0.7 % ammonia
Low ammonia TZ (LA-TZ)	0.2 % ammonia; 0.025 % Zinc Oxide; 0.025 % Tetramethylthiuram Disulphide
Low ammonia pentachlorophenate (LA-SPP)	0.2 % ammonia; 0.2 % sodium pentachlorophenate
Low ammonia Boric Acid (LA-BA)	0.2 % ammonia; 0.24 % boric acid

NR latex foam is particularly suitable for applications in bedding, mattresses, pillows, upholstery, automotive seating, packaging and acoustic insulation, where vibration damping, comfort and noise absorption are critical (Ariff et al. 2020; Rostami-Tapeh-Esmaeil et al. 2021). Its high resilience allows it to retain its original shape even after prolonged compression, making it an attractive material for products requiring long service life. Despite these attractive characteristics, the wider application of NR latex foam is limited by its relatively poor thermal stability and high flammability, which pose considerable safety concerns, especially in fire-sensitive environments (Guo et al. 2017; Huang et al. 2018). As a hydrocarbon-based polymer, natural rubber readily undergoes thermal degradation and combustion when exposed to elevated temperatures or direct flame. During burning, the material can generate significant heat release and smoke, accelerating fire propagation and increasing potential hazards. Consequently, improving the fire resistance of NR latex foam while preserving its desirable physical and mechanical properties has become an important research objective for both academia and industry.

To overcome these limitations, numerous flame-retardant systems have been investigated to improve the thermal stability and fire performance of NR latex foam (Guo et al. 2017; Huang et al. 2018; Wang et al. 2019; Ramli et al. 2023). Generally, flame retardants can be classified into halogenated, inorganic and intumescent systems, each operating through different fire suppression mechanisms. Halogenated flame retardants are highly effective because they interrupt free-radical chain reactions occurring during combustion. However, their application has gradually declined owing to increasing environmental regulations and concerns regarding the release of toxic and corrosive gases during burning (Vinay & Anshuman, 2017). Inorganic flame retardants, such as aluminium hydroxide, magnesium hydroxide and various mineral fillers, are considered safer alternatives due to their lower toxicity and smoke suppression capabilities. Nevertheless, they often require relatively high filler loadings to achieve satisfactory flame retardancy, which may adversely affect the density, flexibility, mechanical performance and processability of the foam (Wang et al. 2019). Intumescent flame retardants function by promoting the formation of an expanded carbonaceous char layer during combustion, thereby acting as a thermal barrier that restricts heat transfer, oxygen diffusion and the release of volatile combustible products. In recent years, increasing environmental awareness and the demand for sustainable materials have stimulated the search for renewable, environmentally benign and cost-effective flame-retardant fillers capable of replacing conventional additives without compromising material performance.

One promising sustainable alternative that has attracted increasing research interest is biochar (Ramli et al. 2023; De Silva et al. 2023). Biochar is a carbon-rich material produced through the pyrolysis of biomass under oxygen-limited conditions. Depending on the biomass source and pyrolysis conditions, biochar possesses a highly aromatic carbon structure, porous morphology, high thermal stability and abundant surface functional groups that make it suitable for a wide range of engineering applications (Ketabchi et al. 2017; Vinay & Anshuman, 2017; Varma et al. 2018; Ramli et al. 2022). Besides functioning as an environmentally friendly filler, biochar may also serve as a low-cost reinforcing agent and partial replacement for conventional mineral fillers, thereby reducing overall material cost. During combustion, the carbonaceous nature of biochar facilitates the formation of a stable protective char layer that limits heat transfer and oxygen penetration, consequently delaying thermal decomposition and flame propagation. Furthermore, the incorporation of biochar into polymer matrices has been reported to improve thermal stability, dimensional stability and, in some cases, mechanical properties due to enhanced filler-matrix interactions (Ramli et al. 2023; De Silva et al. 2023). These multifunctional characteristics have made biochar an attractive candidate for the development of sustainable polymer composites with improved fire safety and reduced environmental impact. The focus of this study

is therefore to evaluate the applicability of biochar OPFB as a potential and sustainable filler on LA-TZ latex foam based on the physical properties, flammability as well as thermal stability.

METHODOLOGY

MATERIALS

LATZ of grade G-TEX LATZ with total solid content of 60 % was supplied by Getahindus (M) Sdn. Bhd. OPFB was pyrolyzed and characterized by using the Kon-Tiki kiln reported by Karananidi *et al*. (2020). OPFB was ground using a Universal Mill (Model: SY-10) post-pyrolysis. The ground OPFB was sieved to < 106 μm with a sieve shaker (Model: EFL200).

Table 2 shows the LA-TZ latex compounding formulation, while Table 3 summarizes the LA-TZ latex foam composite formulations. The ingredients employed are potassium oleate, sulfur dispersion, zinc oxide dispersion, zinc diethyl dithiocarbamate, zinc dibutyl dithiocarbamate, zinc 2-mercapto-benzothiazole dispersion, antioxidant dispersion (Wing stay-L), diphenyl guanidine dispersion and sodium silicofluoride dispersion. These ingredients were purchased from Alpha Nanotech (M) Sdn. Bhd., Selangor, Malaysia. The purchased ingredients were in solution or dispersion form. They were used as received.

FOAM FABRICATION

The high-density LA-TZ foam composites were prepared according to the fabrication steps reported by Ramli *et al.* (2023). The mixture was molded and vulcanized. After vulcanization, the foam composites were removed from the mold for washing and drying. The foam composites were dried in a convection oven (Memmert UN55) at 55°C until constant weight.

TABLE 2. LA-TZ latex foam compounding formulation

Ingredients	Total Solid Content (%)	Dry weight (phr)
LA-TZ latex	60	100
Potassium oleate	20	1.50
Sulphur dispersion	60	2.50
Zinc oxide dispersion	60	0.15
Zinc diethyl dithiocarbamate dispersion	50	0.75
Zinc dibutyl dithiocarbamate dispersion	50	0.25
Zinc 2-mercaptobenzothiazole dispersion	50	1.0
Antioxidant dispersion (Wing stay-L)	50	1.0

phr parts per hundred rubber

TABLE 3. LA-TZ latex foam composite formulations

Ingredients	Total Solid Content (%)	Dry weight (phr)
LA-TZ	60	100
Diphenyl guanidine dispersion	40	0.6
Zinc oxide dispersion	60	5.0
Sodium silicofluoride dispersion	50	1.2
OPFB	100	0, 10, 20, 30

phr parts per hundred rubber

TESTING AND CHARACTERIZATION

The density for the dried LA-TZ foam composites was determined by measuring the dried mass and the volume of the dried foam composites. The volume was determined by measuring the length, width and thickness of the foam composites. The density was calculated by using equation (1). An average of triplicate measurements was reported.

The volume shrinkage of the dried foam composites was determined by using equation (2). The average of triplicate measurements was reported.

$$Density = \frac{Mass\ of\ dried\ LA{-}TZ\ foam\ composites}{\text{Length x Width x Thickness of dried LA-TZ foam composites}} \quad (1)$$

$$Volume\ shrinkage = \frac{Length\ x\ Width\ x\ Thickness\ of\ dried\ LA{-}TZ\ foam\ composites}{\text{Length x Width x Thickness of the mold used}} \quad (2)$$

The Shore F hardness was performed according to the ASTM D 2240 using a Shore F durometer. A total of five test specimens with a size of 200 x 200 x 40 (length × width × thickness) ± 5 mm each were used. For each test specimen, measurements were taken at five different locations. Each location was at least 6.0 mm apart from each other. The highest and lowest readings were then discarded, and the hardness value was calculated from the average of the remaining three readings. Removing the highest and lowest values from a small dataset before averaging is known as calculating a robust trimmed mean. It prevents localized material anomalies or minor operator inconsistencies from skewing the true baseline of the compound (Mukuno et al. 2025).

The flammability of foam composites was evaluated via the UL 94 horizontal burning foamed material test (UL 94 HBF) according to ISO 9772 (Ramli *et al.* 2023). For each foam composite, five test specimens of size 150 × 50 × 13 mm (length × width × thickness) ± 5 mm were employed. For each test specimen, the length at 25 mm, 60 mm and 125 mm was marked and placed horizontally on a metallic grid. Test specimens were burned for 60 s from the corner which is closest to the 25 mm mark. A methane burner with a flame height of 38 mm was used. The time taken for the flame to move from the 25 mm gauge mark to the 125 mm gauge mark was recorded. The burning velocity was calculated by using equation (3).

$$"Burning\ velocity" = \frac{L_d}{t_p} \quad (3)$$

where L_d is the distance between 25 mm and 125 mm gauge marks, and t_p is the time elapsed when the flame passes the 25 mm gauge mark to the time the flame stops or the flame reaches the 125 mm gauge mark. A material is rated to pass UL 94-HBF if the burning velocity does not exceed 40 mm/min over a 100 mm span or ceases from burning before it reaches 125 mm gauge mark

Thermogravimetric analysis (TGA) was performed to determine the thermal decomposition of the foam composites. It was performed by using Mettler-Toledo TGA/DSC 1. Test specimen of 3-4 mg were heated from 30 to 900 °C with a heating rate of 10 °C/min under a nitrogen flow rate of 20 ml/min. Upon reaching 900 °C, they were held for 10 min. The residual weight (TG) and its derivative (DTG) were analyzed. The onset of decomposition temperature, decomposition inflection point and its weight loss as a function of temperature, as well as the final residue, were calculated.

RESULTS AND DISCUSSION

Figure 1 shows the dried density of the LA-TZ foam composites at different OPFB loadings, while Table 4 presents the corresponding one-way ANOVA analysis. Based on the results, it can be concluded that the density increased with increasing OPFB loading. The average density increased from 0.2386 g/cm³ for the control sample (0 phr) to 0.2637 g/cm³ at 30 phr OPFB, indicating a gradual increase in the compactness of the foam structure. According to Blanco-Canqui (2017), the density of OPFB ranges from 1.5 to 2.0 g/cm³, which is considerably higher than that of LA-TZ foam (approximately 0.2345 g/cm³). Therefore, the incorporation of OPFB increased the overall density of the foam composites as the biochar particles replaced part of the low-density foam matrix. Similar findings have been reported by Dananjaya et al. (2024), who observed that increasing biochar loading resulted in higher composite density due to the greater proportion of solid filler within the material. The ANOVA results further confirmed that the differences in density among the various OPFB loadings were statistically significant, demonstrating that OPFB loading is an important factor influencing the density of LA-TZ foam composites.

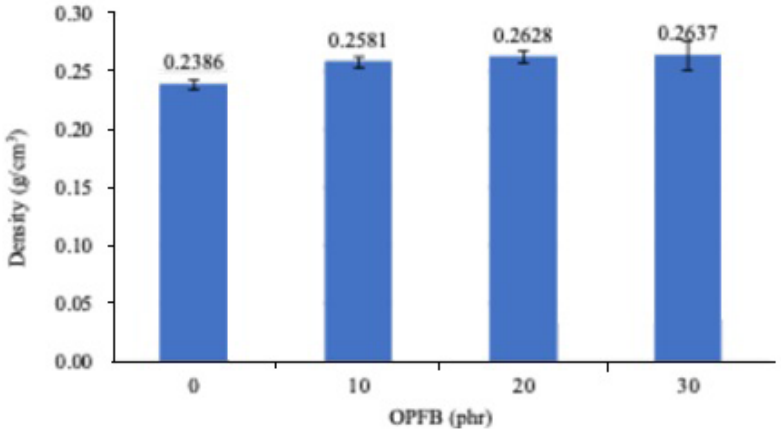


FIGURE 1. Density of dried LA-TZ foam composites at different OPFB loadings

TABLE 4. One-way ANOVA on the LA-TZ foam composites' density

Source of Variation	SS	df	MS	F	P-value	F crit
Between Groups	0.0017	3	0.0006	10.72	0.0035	4.07
Within Groups	0.0004	8	5.26136E-05			

Figure 2 shows the volume shrinkage of the dried LA-TZ foam composites at different OPFB loadings, while the corresponding one-way ANOVA analysis is presented in Table 5. As the OPFB loading increased, the volume shrinkage of the foam composites decreased noticeably, from 22.37% for the control sample to 12.91% at 30 phr OPFB. This reduction indicates that the incorporation of OPFB improved the dimensional stability of the foam during the drying process. For the LA-TZ foam composites, the actual volume contraction decreased with increasing OPFB loading because OPFB is a rigid solid filler that occupies space within the foam matrix and restricts structural collapse during drying. Consequently, higher OPFB loadings produced foam composites with lower shrinkage. The observed volume shrinkage trend is consistent with the density results presented in Figure 1. Higher density reflects a greater solid-to-air phase ratio within the foam structure (Ramli et al. 2022). Therefore, as the OPFB content increased, the increased solid fraction contributed to higher density while simultaneously reducing volume shrinkage, demonstrating an inverse relationship between these two physical properties.

The indentation of an indenter measures hardness. Harder materials are more difficult to indent. Figure 3 shows the hardness of the foam composites, and the one-way ANOVA results are tabulated in Table 6. Typically, higher density leads to harder foam (Ramli et al. 2022). However, the results obtained do not show significant differences in the hardness of the foam composites within the OPFB loading range studied. This may be due to the higher crosslink density achieved in the foam even without the presence of OPFB (Shojaie et al. 2021). Further research is required to visualize pore structure and filler dispersion across the foam cross section to better understand the lack of hardness trend observed. Additionally, a compression set and more sensitive tensile tests should be carried out to elucidate how OPFB filler particles interact with the rubber chains during large-scale deformation.

The burning velocity of the LA-TZ foam composites in Figure 4 shows a decreasing trend with increasing OPFB loading. Burning velocity decreased with OPFB loading, and the flame did not exceed 125 mm gauge. This indicates that OPFB has flame retardancy capability, even though it does not meet the UL 94 HBF requirement of 40 mm/min. Similarly, Ramli et al. (2022) also showed the flame retardancy capability of oil palm frond-based biochar. According to the trendline equation, an OPFB loading of 103 phr will meet the requirement. While our study suggests that OPFB has the potential to be used as an eco-friendly flame retardant filler, additional tests need to be carried out to study the impact of such a high loading on the mechanical integrity and foaming ability of LA-TZ foam.

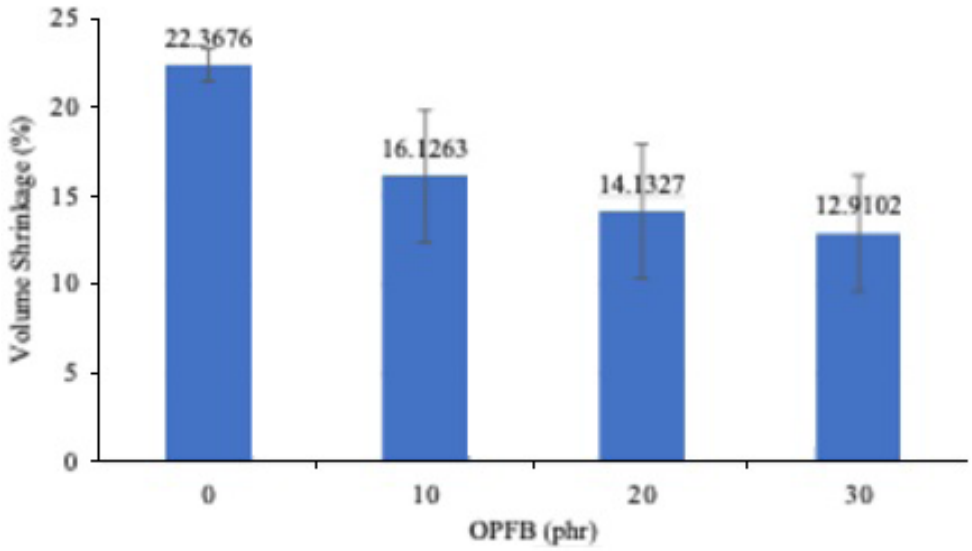


FIGURE 2. Volume shrinkage of dried LA-TZ foam composites at different OPFB loading

TABLE 5. One-way ANOVA on the LA-TZ foam composites' volume shrinkage

Source of Variation	SS	df	MS	F	P-value	F crit
Between Groups	159.0145	3	53.0048	5.32	0.026	4.0662
Within Groups	79.7161	8	9.9645			

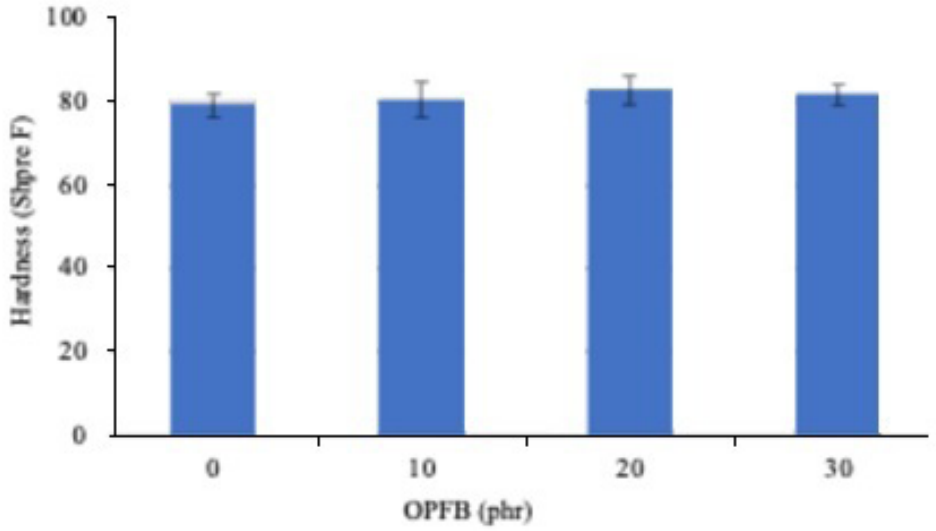


FIGURE 3. Hardness of dried LA-TZ foam composites at different OPFB loading

TABLE 6. One-way ANOVA on the LA-TZ foam composites' hardness

Source of Variation	SS	df	MS	F	P-value	F crit
Between Groups	42.8095	3	14.269	1.224	0.333	3.239
Within Groups	186.516	16	11.657			

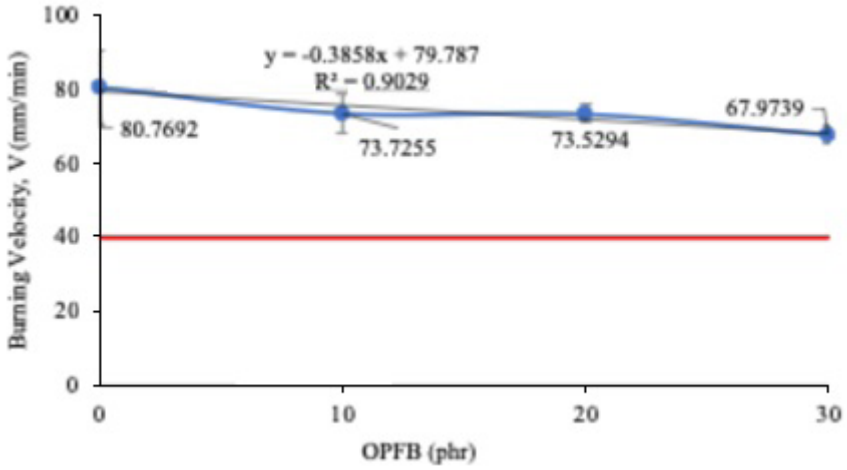


FIGURE 4. Burning velocity of dried LA-TZ foam composites at different OPFB loading, showing the UL 94 HLB requirement of 40 mm/min

The thermograms of TGA are summarized in Figure 5, while Figure 6 shows the derivative curves. The onset of decomposition temperature directly indicates a material's thermal stability. The decomposition of LA-TZ foam is at 333 °C with a weight loss of 83.9 %. Its inflection point is 368 °C. With the presence of OPFB, a higher onset temperature as well as inflection point was observed (Ramli et al. 2023).

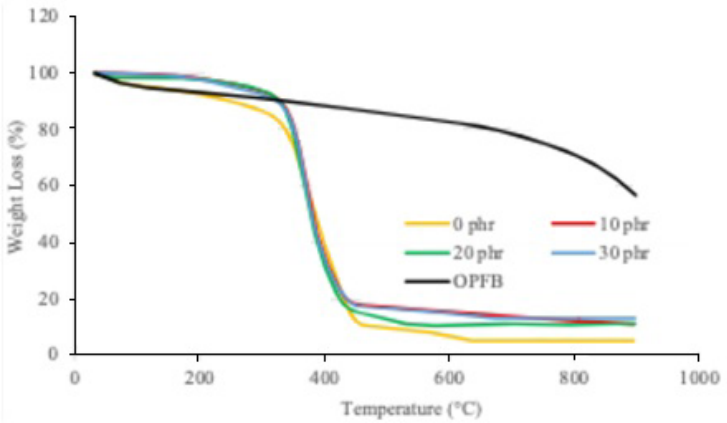


FIGURE 5. TGA curves of LA-TZ foam composites and OPFB only

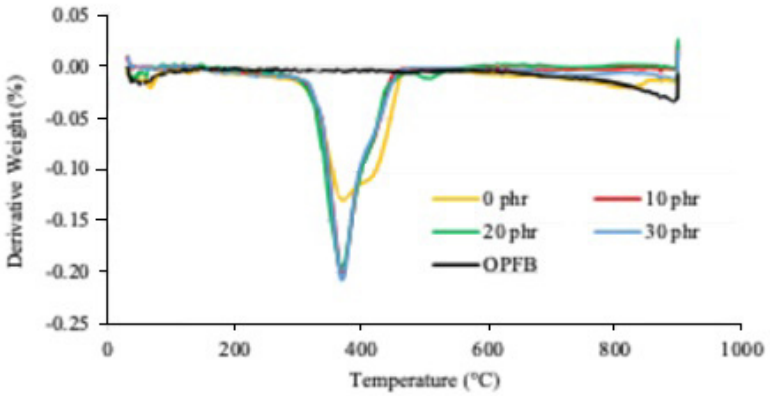


FIGURE 6. Derivative weight curves of LA-TZ foam composites and OPFB only

CONCLUSION

The incorporation of oil palm frond biochar (OPFB) into LA-TZ foam composites significantly influenced several important physical, flammability, and thermal properties of the resulting materials. The presence of OPFB in LA-TZ foam composites resulted in higher density and lower volume shrinkage. As the loading of OPFB increased, the density increased, while the volume shrinkage progressively decreased. This behaviour is attributed to the ability of OPFB to increase the ratio of the solid phase relative to the air phase within the foam structure, producing a more compact and dimensionally stable composite. However, no significant differences in hardness were observed at OPFB loadings between 0 and 30 phr. This phenomenon is hypothesized to be associated with the relatively high crosslink density of the latex foam, which dominates the mechanical response and minimizes the influence of the biochar filler on hardness. Furthermore, OPFB demonstrated considerable potential as a sustainable bio-based filler by reducing the flammability and enhancing the thermal stability of the LA-TZ foam composites. The improved thermal resistance indicates that OPFB can contribute to delaying thermal degradation and enhancing the overall heat resistance of the composite material. Nevertheless, despite the observed reduction in flammability, the foam composites did not satisfy the requirements of the UL 94 HBF flammability standard, indicating that additional flame-retardant modifications may be necessary for applications requiring higher fire safety performance. Overall, the findings of this study demonstrate that OPFB is a promising and environmentally sustainable filler for LA-TZ foam composites, offering improvements in dimensional stability and thermal performance while supporting the utilization of oil palm biomass as a value-added material in polymer composite applications.

ACKNOWLEDGEMENT

The authors are grateful for the facilities provided by the Malaysian Rubber Board and the financial support of this research from the Ministry of Higher Education (MoHE) via the Fundamental Research Grant Scheme (FRGS) – FRGS/1/2020/STG04/ UNIKL/02/2. The authors declare that there is no conflict of interest with respect to the publication of this paper.

DECLARATION OF COMPETING INTEREST

None.

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