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Flammability and Soil Burial Performance of Sugar Palm (*Arenga pinnata (wurmb) merr*) Fiber Reinforced Epoxy Composites

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ABSTRACT

This study investigates the effects of soil burial and flammability on sugar palm fibre (SPF) (*Arenga pinnata (wurmb) merr*)-reinforced epoxy composites. In order to determine the flammability and biodegradability properties, experiments are conducted in accordance with ASTM standards. The hand lay-up method was used to fabricate composite samples with two different weight ratios between epoxy and SPF, which were 70:30 and 50:50. Biodegradability and flammability properties were investigated using horizontal burning tests, limiting oxygen index (LOI), cone calorimetry, and soil burial. It was found that the Epoxy/SPF-50 was the composite that exhibited the fastest degradability at 0.81%/week. The result of the horizontal burning test showed that the addition of SPF reduced the burning rate but slightly increased it at 50 wt% because the ratio between epoxy and SPF exceeds the optimum fibre loading. The Epoxy/SPF-50 exhibited a better LOI value at 23.3 than pure epoxy (control), which was 19.8. From the cone calorimetry test, it was observed that the time to ignition (TTI) and total heat release (THR) values were decreased when the amount of SPF increased. Char production increases the flame-retardant protection of SPF-reinforced epoxy composites. To the best of the authors' knowledge, no published study has been conducted on the flammability and biodegradability characteristics of SPF-reinforced epoxy composites.

Keywords: Biocomposites, cone calorimetry, flammability, soil burial, sugar palm fibre

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INTRODUCTION

Over the last decade, the increasing use of natural fibres in polymer composites has significantly reduced environmental impacts. Owing to their biodegradability, availability, simplicity of processing, low cost, considerable features, and lightweight, lignocellulose fibre-reinforced polymer composites are recommended for use in construction, automotive and furniture industries (Song et al., 2023; Alaseel et al., 2022; Ibrahim et al., 2012; Jawaid & Abdul Khalil, 2011; Sanjay et al., 2018; Tarique et al., 2021). Sugar palm trees could produce various products, including palm sugar, fruits, and fibres (Aworinde et al., 2021; Ilyas et al., 2018; Khan et al., 2021). The SP tree is a forest plant that was previously classified as a Palmae family member but now belongs to the subfamily *Arecoideae* as well as the tribe *Caryoteae* (Alaaeddin et al., 2019; Atiqah et al., 2018; Ilyas et al., 2020; Tarique et al., 2021). The SP is likewise a fast-growing palm that can reach maturity in as little as ten years (Mogea et al., 1991). Indo-Malay region, South Asia, and Southeast Asia are covered by the geographical distribution of SPs (Atiqah et al., 2019; Tarique et al. et al., 2021). Furthermore, by utilizing SPF, plant waste can be recycled. Furthermore, SPF is widely available at a low cost and is easily accessible. Bachtiar et al. (2009) evaluated the mechanical behaviour of SPF and found it to have Young's modulus of 3.69 GPa, a tensile strength of 190.29 MPa, a strain to failure of 19.6%, and a density of 1.26 kg/m^3.

Owing to their outstanding mechanical, thermal, and electrical characteristics, modified epoxy resins are now utilized to fabricate natural fibre-reinforced composite (NFRCs) and create various industrial products. Natural fibre integration is also the most intriguing approach to altering epoxy resin. Natural fibres as reinforcement in polymers have seen a rise in attention and use in recent years in engineering as well as research technology (Mohammed et al., 2023; Alamri & Low, 2012; Farhana Mat Nasir et al., 2020; Low et al., 2007; Shih, 2007; Tarique et al., 2022). Das and Biswas (2016) investigated the impact of fibre length on the mechanical behaviour of coir fibre-reinforced epoxy composites, finding that tensile strength attained its maximum value at 12 mm fibre length. Venkateshwaran et al. (2011) examined the estimate of optimal fibre length for banana epoxy composite, finding that increasing fibre length and weight ratio enhanced tensile strength and modulus, likely a 15 mm fibre length. Aji et al. (2011) investigated the effect of fibre length on tensile characteristics of epoxy resin composite reinforced with kenaf/PALF fibres, finding that a fibre length of 0.25 mm provided the best maximum tensile strength, although a fibre length of 2 mm reduced tensile modulus property owing to weak interface bonding between the matrix and reinforcement.

In addition, the mechanical properties of epoxy composites based on a stacking sequence of *Cyperus pangorei* and jute fibres were studied by Vijay and Singaravelu (2016) when compared to the other three composites, the mechanical strength of one produced with *C. pangorei* as the core and jute fibres as the skin layer was shown to be better. Edhirej et al. (2017) demonstrate that the observation of sugar palm fibres' usage as rope and traditional construction material inspired them to investigate the possibility of employing them as a composite material. The fibres are twisted to the proper diameter before being woven into rope. The fibres must endure wind loads and give protection from rain and tropical sun to be used as traditional roofing in rural tropical conditions because sugar palm fibres have adequate endurance. Previous research on tensile as well as flexural properties of sugar palm epoxy composites has focused on the use of woven roving, long random, and chopped random fibre composites, with the results showing that the woven roving sugar palm epoxy composites gave better properties than the long random and chopped random fibre composites (Sharba et al., 2016). However, all the samples performed poorly compared to glass fibre epoxy composites, meaning fibre treatment was essential for improving the materials.

Several studies have demonstrated that natural fibre can increase biocomposites' resistance to ignition. By reinforcing twill woven hemp fabric with epoxy composites, Kozłowski and Władyka-Przybylak (2008) found that the flammability of the base matrix composites was lowered, as measured by higher limiting oxygen index (LOI) values and a reduced heat release rate of 25%. Additionally, composites' static and dynamic mechanical properties from the modified fabric were enhanced. Bharath et al. (2014) report that treated composites performed better in fire and flame resistance tests (UL 94 V and UL 94 HB) and had lower rates of flame propagation and mass loss. Treated sisal fibre (SF)-reinforced recycled polypropylene (RPP) composites' flammability was evaluated using a horizontal burning test with UL-94 (Gupta et al., 2012). In light of issues about safety, waste disposal, and the decline of nonrenewable resources, researchers and scientists are also concentrating on using renewable resources (Alaseel et al., 2022; Hisham et al., 2011; Liu et al., 2006). Numerous research activities have been done on reinforcing natural fibres that could replace synthetic fibres (glass and carbon fibres) in composite applications, such as coir, date palm, bamboo, oil palm empty fruit bunch (OPEFB), hemp, sisal, flax, jute, and others (de Vasconcellos et al., 2014; Deo & Acharya, 2010; Mahjoub et al., 2014; Mishra & Biswas, 2013; Scida et al., 2013; Yousif et al., 2012).

Because the current scenario concerns using naturally abundant material to substitute synthetic material, this research deals with NFRCs and using innovative plant fibres to reinforce polymer composite. However, to our knowledge, studies dealing with SPF/Epoxy composites have not been performed. As a result, the primary purpose of this research was to investigate the biodegradability and flammability characteristics of epoxy-based composites reinforced with SPF.

MATERIALS AND METHODS

Materials

The SPF was collected from sugar palm trees at Kampung Kuala Jempol, Negeri Sembilan, Malaysia, as shown in Figure 1. The epoxy resin and hardener, namely Zeepoxy HL002 TA/B, are supplied by ZKK Sdn. Bhd, Cheras, Kuala Lumpur, Malaysia, were used in the fabrication process of the composites.

Figure 1. Sugar palm tree and sugar palm fibre

Fabrication of SPF/Epoxy Composites Testing Sample

The SPF-reinforced epoxy composites were produced at room temperature using a hand lay-up technique. SPF was mixed into the matrix with a stirrer with 500 RPM to create a matrix and filler mixture; as a result, a homogeneous mixture of matrix and filler was created. The resin is a colourless, viscous liquid with a viscosity of 5500–1000Cps at 30°C, while the hardener has a viscosity of 30–20Cps at 30°C. The ratio of epoxy resin to hardener was 2:1. Table 1 lists the compositions of the SPF-reinforced epoxy composites. A thin plastic sheet is put to the bottom and top of the mould to achieve a smooth surface for the composites. The epoxy was combined with the SPF before being placed into the 300 mm × 300 mm × 3 mm mould. The mould was cured at room temperature for 24 hrs, after which composite samples were removed.

Table 1
The formulations of the SPF-reinforced epoxy composites

Name of composites	Epoxy Resin (wt. %)	SPF (wt. %)
Pure Epoxy	100	0
Epoxy/SPF-30	70	30
Epoxy/SPF-50	50	50

Characterization of Composite Sample

Soil Burial Test. A soil burial test was accomplished to evaluate composites' biodegradability. Specimens were prepared with four replicates of each sample and cut to the following dimensions: 10 cm (L) × 4 cm (W) × 0.3 cm (H). 4, 8, 12, and 16 weeks were used for the biodegradability test. Specimens were buried 10 beneath the surface in moist soil in a polybag. Throughout the test period, the polybags were outdoors. After a specified time the specimens were taken out of the soil and cleaned with distilled water. Using Equation 1, the weight loss, W_{loss} (%), was calculated.

$$W_{loss} = \frac{W_{initial} - W_{final}}{W_{initial}} \times 100\% \qquad (1)$$

Horizontal Burning Test. The flammability test was carried out in a horizontal position. The specimens had dimensions of 125 mm × 13 mm × 3 mm when ready, according to ASTM D635-18 (2018). Two reference lines were drawn as starting and finishing points at 25 mm and 100 mm intervals. The sample was clamped horizontally at the end, making both reference lines visible using a retort stand. The specimen was lit from the other end, and the timer was started as soon as the flame was 25 mm away from the sample. The test was run in triplicate, and it was noted how long the flame took to advance 100 mm. Equation 2 was used to determine the sample's burning rate.

$$V = L\ t \qquad (2)$$

Where V, L, and t represent linear burning rate (mm/min), burnt length (mm), and time (min), respectively.

Limiting Oxygen Index (LOI)

The ASTM D2863-09 (2010) is used to evaluate the LOI. This experiment aimed to identify the lowest oxygen content of the sample combustion. The specimens had the following measurements: 100 mm × 6.5 mm × 3 mm. The specimen was placed vertically in the middle of a glass chamber and lit for 10 seconds until ignition. Ten replicate specimens were used in the test until the last five specimens, which were tested, had an oxygen concentration deviation of 0.2 vol%. Equation 3 calculated LOI based on the last five test specimens that burned.

$$LOI\ (vol\%) = C_f + kd \qquad (3)$$

Where C_f is the final oxygen value concentrations, in volume % to one decimal place for the previous five measurements, d is the interval difference between oxygen concentration levels in per cent volume, and k is a factor derived from experimental values.

Cone Calorimetry

The cone calorimetry testing was conducted following ISO 5660-1 (2002). The specimens had dimensions of 100 mm × 10 mm × 3 mm. The specimen was wrapped in aluminium foil on the sides and bottom before being placed horizontally on the specimen holder. The specimens' surfaces were spark-ignited and irradiated with a 35 kW/m^2 heat flux.

RESULTS AND DISCUSSION

Soil Burial

Soil burial testing for SPF-reinforced epoxy composite was done for 16 weeks. It was observed that the weight loss of an epoxy resin was raised in the natural soil environment after the reinforcement of SPF. As expected, loss of weight loss of composite was more significant with an increase in burial time in soil. The weight of pure epoxy had lost 3.55% of its original weight by the end of week 16, while the weights of Epoxy/SPF-30 and Epoxy/SPF-50 had lost 9.74% and 12.88%, respectively. The average degradation rates for pure epoxy and Epoxy/SPF-30 are 0.22%/week and 0.61%/week, respectively. Epoxy/SPF-50 had the highest average degradation rate of 0.81%/week. Figure 2 illustrates the trend of weight loss (%) of SPF-reinforced epoxy composites as a function of biodegradation time after soil burial analysis.

Pure epoxy recorded a minimal weight loss because the polymer matrix is not easy to degrade. It was noticed that increasing the amount of SPF increased the degradability rate. Epoxy/SPF-50 recorded the highest rate of degradability. The existence of cellulose in SPF allows water molecules to easily absorb. Due to its hygroscopic nature, cellulose in SPFs can absorb water from the environment and swell. The increasing hygroscopic properties of composites enhanced microbial activity, resulting in weight loss (Ilyas et al., 2020; Minh et al., 2019). The mechanism of biological degradation comprises water

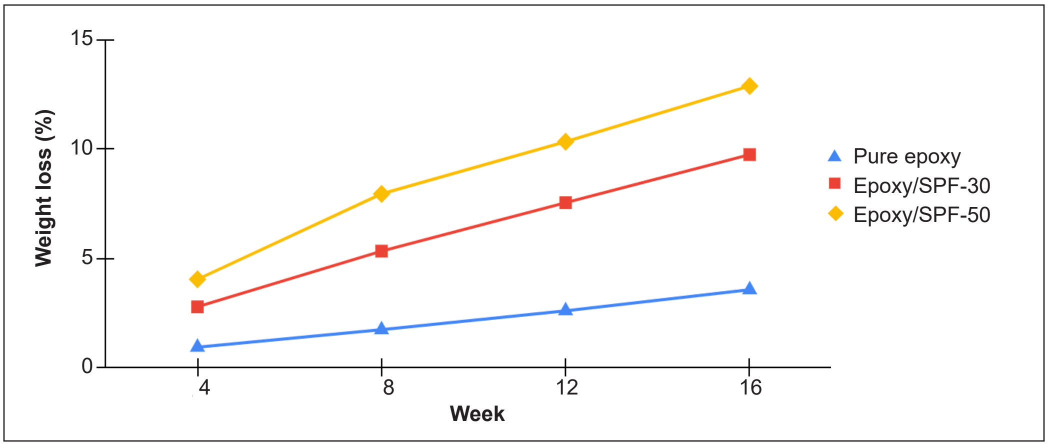

Figure 2. The trend of the sample's weight loss (%)

molecules penetrating the material, strong covalent bonds breaking, and microorganisms degrading the hemicellulose and cellulose.

Horizontal Burning Test

The SPF-reinforced epoxy composites' horizontal burning test was tested following ASTM D635 (2018). The fire was stopped when it reached the 100 mm mark of the specimens. Figure 3 shows the average burning rate of the samples.

According to the findings, incorporating SPF reduces the rate of burning. Epoxy/SPF-30 burned at a lower rate of 15.4 mm min^{-1} than pure epoxy, which burned at 26 mm min^{-1}. The flame took time to propagate alongside the Epoxy/SPF-30 sample and produced char simultaneously. The SPF frequently flows into char during burning, providing additional flame-retardant protection (Suriani et al., 2021). This finding indicates that reinforcing the SPF reduced the epoxy composite's burning rate. However, the burning rate increased slightly in Epoxy/SPF-50 due to the SPF/EP ratio being more than optimum fibre loadings. As a result, the composite's ability to produce char is reduced during the burning process (Xiao et al., 2018; Tarique et al., 2022). Compatibility of fibre and matrix is critical for providing the best flame-retardant characteristics to composite. The matrix polymer determines a composite's flammability, the variety of fibre used, and the interfacial bonding between the two. The compatibility of fibre and matrix is affected by their interfacial connection.

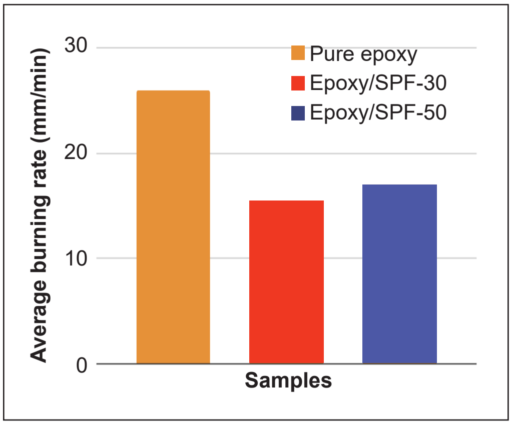

Figure 3. The average rate of burning of composite samples

Limiting Oxygen Index (LOI)

The LOI is a widely used fire index for characterizing the flammability of polymer material. It describes the lowest oxygen concentrations required to help a material's flammable combustion. The LOI values of specimens are shown in Table 2.

The limit Oxygen value of pure epoxy is 19.8% and is classified as combustible as its LOI value is lower compared to the oxygen contents of air, which is 21%. Material with less than 21% LOI value is classified as flammable under the standard LOI criteria. Conversely, materials are considered to be

Table 2
The limiting oxygen index (LOI) of the samples

Samples	LOI
Pure Epoxy	19.8
Epoxy/SPF-30	22.5
Epoxy/SPF-50	23.3

self-extinguishing if their LOI value is greater compared to 21% because, according to the standard air composition, oxygen content in the air is 21%, as materials with a value higher compared to this cannot support burning at room temperature without an external fire source (Karunakaran et al., 2016). Pure epoxy possessed a low LOI value due to the poor flammability properties of the polymer matrix. According to Prabhakar et al. (2015), polymer matrices are weak against flame propagation and thermal load. Polymer matrices themselves depend upon reinforcement and filler. The Epoxy/SPF-50 recorded a better LOI value of 23.3 compared to the Epoxy/SPF-50, which is 22.5. Composites' flammability is influenced by interactions between and among its constituent parts. However, the burning rate of the epoxy/SPF-50 was lower because higher LOI values indicate lower flammability, a lower burning rate, and better flame retardance. Combining them can make the composite less flammable (Gurunathan et al., 2015). However, the LOI value was not significantly increased between the 30 wt.% and 50 wt.% of SPF, which may be related to the non-polar behaviour of SPF that affects low fibre dispersion.

Cone Calorimetry

Cone calorimetry gathers data like TTI, HRR, and THR. Table 3 shows selected data obtained from the cone calorimetry test.

TTI describes the time needed for ignition when materials are exposed to a constant heat flux (35 kW/m^2) and in an oxygen-controlled environment. Thus, the higher TTI is preferable as well as considered to be less flammable. From Table 4, Epoxy/SPF-50 has the lower TTI at 62 seconds, while Epoxy/SPF-30 has the higher TTI at 97 seconds. The fast ignition of Epoxy/SPF-50 could be due to the high lignin content of SPF. Lignin breakdown contributes more to char generation than cellulose and hemicellulose. However, lignin decomposition begins at a lower temperature, between 160 and 400°C (Ali et al., 2021; Fu et al., 2017). Therefore, the increase in lignin content will increase flammability.

The heat release rate is a crucial and additional factor in determining a material's flammability (HRR). When a material is exposed to fire, HRR is the heat released per unit area. In addition to being a crucial parameter for describing fire behaviour, it also plays a role in defining concepts like a fire hazard. HRR curve for specimens is shown in Figure 4.

According to observation, the HRR curve for SPF-reinforced epoxy composites has two peaks. By forming carbonic char structures, the first peak reflects the charring process. The

Table 3
The selected data obtained from the cone calorimetry test

Samples	TTI (S)	pHRR (kW/m^2)	THR (MJ/m^2)
Pure Epoxy	82	706.3	99
Epoxy/SPF-30	97	468.5	79
Epoxy/SPF-50	62	355.1	71

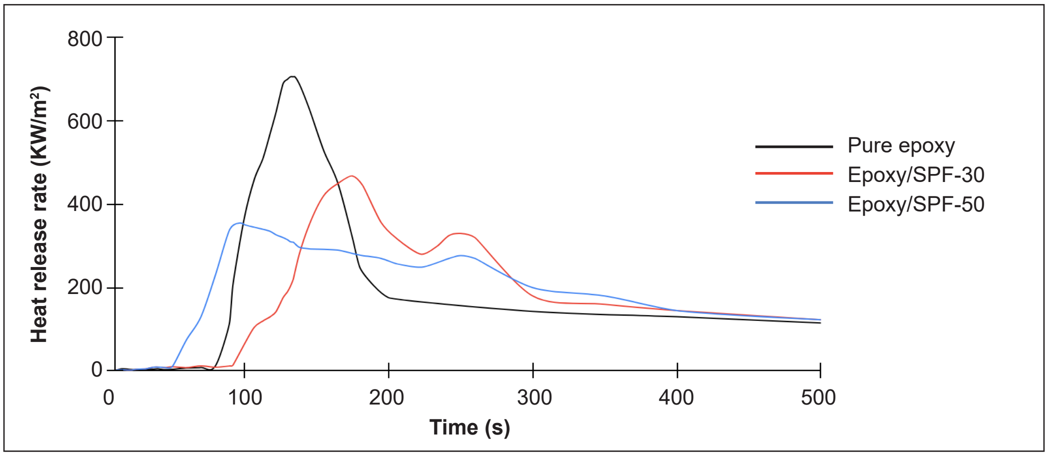

Figure 4. The HRR curves for the samples

char layer protects and prevents the movement of mass and volatiles from the condensed to the gas phase. Therefore, after the first peak, the combustion rate decreased, and a drop in the HRR curve was seen. As the burning process goes on, a surplus of trapped volatiles causes high internal pressures during the escape, which accelerates the formation of voids and causes the char residue to crack and degrade, which further encourages combustion and results in another peak HRR (Chee et al., 2020).

The maximum amount of heat released during combustion is referred to as the peak HRR (pHRR), and the total amount of heat released is shown by the area under the HRR curve (THR). From Figure 4, the pure epoxy exhibited the highest pHRR at 706.3 kW/m^2. When the amount of SPF increased, the TTI and THR values from the cone calorimetry test decreased. The Epoxy/SPF-30 showed better thermal stability than Epoxy/SPF-50. Additionally, char production supports the flame-retardant resistance of SPF-reinforced epoxy composites. A significant reduction of pHRR between 33% and 50% was seen on the SPF-reinforced epoxy composites than pure epoxy. The greatest reduction was observed on Epoxy/SPF-50, with a pHRR of 355.1 kW/m^2 and a THR of 75 MJ/m^2. The reduction of pHRR and THR of the sugar palm composite could represent the increase in char production (Hatanaka et al., 2016).

CONCLUSION

This research aims to develop an epoxy composite reinforced with SPF. The Epoxy/SPF-30 composite recorded the lowest burning rate for horizontal burning at 15.4 mm/min due to char production from SPF, which increased flame-retardant protection. The LOI value increases when the amount of SPF is increased. Epoxy/SPF-30 is better than Epoxy/SPF-50 due to the low dispersion of fibre when the amount of SPF is increased. From the cone calorimetry test, TTI and THR values decreased when SPF increased. In addition, the char production helps to increase flame-retardant protection of SPF-reinforced epoxy composites. The degradability rate of sugar palm-reinforced epoxy composites was increased as the amount of SPF increased. After 16 weeks of soil burial testing, pure epoxy recorded the lowest weight loss, 3.55%, and Epoxy/SPF-50 recorded the highest weight loss, 12.88%. Nonetheless, the progress in this field has enabled the use of natural-fibre-based composites in a wide range of industries, including construction, automotive, and aerospace.

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