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Mechanical performance and morphological behaviour of hybrid kenaf and empty fruit bunch fibres reinforced poly(lactic acid) biocomposites

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Abstract
The development of high-performance biodegradable biocomposites is essential to address plastic waste and resource sustainability challenges. However, biocomposites often exhibit limited mechanical performance and moisture sensitivity due to the hydrophilic nature of lignocellulosic fibres. This study investigates hybrid biocomposites based on poly(lactic acid) (PLA) reinforced with kenaf and empty fruit bunch fibres (EFB) to enhance structural and degradation performance. Hybrid biocomposites were fabricated via internal mixing followed by compression moulding. Hybridisation significantly improved mechanical properties of biocomposites. The incorporation of empty fruit bunch fibres into kenaf reinforced PLA increased tensile strength by approximately 47% and tensile modulus by 32% compared with non-hybrid biocomposites. The hybrid biocomposites also exhibited enhanced density and the highest biodegradation rate, with about 0.33% weight loss after ten days of soil burial. Fourier transform infrared analysis confirmed reduced hydroxyl and ester groups after fibre treatment, indicating improved interfacial compatibility. Scanning electron microscopy revealed stronger fibre–matrix adhesion, contributing to increased stiffness and strength. These findings demonstrate that kenaf and empty fruit bunch fibre hybridisation effectively enhances mechanical performance while maintaining biodegradability, supporting sustainable biocomposites development.

Highlights

- Hybrid biocomposites were fabricated using PLA reinforced with kenaf and EFB fibres.
- Hybridisation improved tensile strength (~47%) and modulus (~32%).
- Alkali treatment enhanced fibre–matrix adhesion, confirmed by Fourier transform infrared spectroscopy and SEM.
- Fibre incorporation increased density and accelerated biodegradation.
- The developed hybrid biocomposites support SDG 9, SDG 12, and SDG 13.

Keywords Hybrid natural fibre biocomposite, Kenaf, Empty fruit bunch, Tensile, Soil burial, Density, Morphology

1 Introduction

The globe is at the present dealing with a worldwide environmental challenge that may impact people's quality of life in the future. The widespread use of non-biodegradable plastic products is driven by their low cost, ease of processing, and durability, which has resulted in a rapid rise in global plastic waste [1–3]. Since these materials do not naturally decompose, they pose immediate threats to the environment and negatively affect the quality of life for both humans and animals [4, 5]. Prolonged exposure of plastics to the environment further leads to their breakdown into microplastics and nanoplastics, which are highly hazardous to ecosystems and human health [6, 7]. The issue of waste management resulting from inappropriate disposal of waste is one of the problems. In order to promote these changes, the United Nations (UN) developed the Sustainable Development Goal (SDG) in recognition of the severity of the issues. Moreover, the bibliometric analysis revealed a marked increase in scientific attention to plastic waste management following the declaration of the United Nations Sustainable Development Goals (UN-SDGs). Among contributing nations, India ranked first in influence, followed by China, the United Kingdom, and France [8].

The rapid accumulation of non-biodegradable plastic waste has emerged as a critical global environmental challenge, contributing to long-term ecosystem contamination, microplastic pollution, and increasing greenhouse gas emissions associated with fossil-based polymer production. Addressing these challenges requires the development of alternative materials that reduce reliance on petroleum resources while improving end-of-life degradability. Within this context, biodegradable polymer composites reinforced with renewable natural fibres have gained increasing attention as promising materials capable of supporting more sustainable material cycles [9]. Such materials align with broader sustainability objectives, including reducing plastic waste generation, promoting renewable resource utilisation, and improving material circularity.

As a sustainable alternative, biodegradable materials have gained attention [9], as they can break down into simpler compounds readily metabolised by microorganisms such as bacteria, yeasts, and fungi [10]. Recent years have witnessed a study and analysis of numerous natural fibres in biocomposite applications as part of the drive for ecologically friendly materials. One of them is a natural fibre that may be used to produce various types of industrial applications and is derived from plants, animals, or minerals [11–14]. They have gained attention as potential alternatives to synthetic fibre composites due to their renewable and biodegradable nature. This work addresses the increasing demand for sustainable reinforcement strategies by systematically investigating the incorporation of natural fibres into hybrid biocomposites systems. Systematic mechanical characterisation is undertaken to critically assess the reinforcement effectiveness of hybrid biocomposites, providing insight into their structural performance within the context of sustainable material design and inherent material limitations [15, 16].

The use of natural fibres in biocomposites offers several advantages, including their abundance, low cost, and reduced environmental impact compared to traditional synthetic fibres. Additionally, the incorporation of natural fibres in biocomposites can enhance their mechanical properties, such as tensile strength and stiffness, while maintaining a lightweight characteristic. Furthermore, natural fibres have inherent damping abilities, making them ideal for applications that require vibration and noise reduction [17–20].

Hybrid biocomposites are materials that combine two different types of natural fibres, such as cotton and jute, to create a stronger and more versatile material. Several studies have been conducted to explore the potential of hybrid biocomposites. One such study focused on the processing, mechanical, physical, electrical, thermal, and dynamic mechanical properties of hybrid biocomposites. The researchers found that the combination of different natural fibres in hybrid biocomposites led to improved mechanical properties, including increased tensile strength and stiffness [16, 21–23].

Natural fibres commonly used in industry, such as jute, sisal, oil palm, kenaf, and flax, have established global markets and production lines. The fact that natural fibres have a similar specific modulus and a higher specific strength than glass fibre is one of the factors contributing to this increased interest [24, 25]. Kenaf, scientifically designated as Hibiscus cannabinus and belonging to the Malvacea family, serves as a significant natural plant fibre reinforcement in Polymer Matrix Composites (PMCs), finding applications across diverse industrial sectors. Renowned for its cellulosic composition, kenaf offers notable economic and ecological advantages [26, 27]. Within a mere three months post-seeding, kenaf exhibits rapid growth potential, flourishing under varied weather conditions, reaching heights exceeding 3 m and base diameters ranging from 3 to 5 centimeters. This observation is corroborated by prior research, indicating growth rates of up to 10 centimeters per day under optimal environmental conditions [28, 29].

Kenaf has gained significant attention in recent years due to its dual capacity to absorb nitrogen and phosphorus from soil, with absorption rates notably surpassing those of various stress conditions, at 0.81 g/m^2 per day for nitrogen and 0.11 g/m^2 per day for phosphorus [30, 31]. Additionally, kenaf demonstrates remarkable carbon dioxide accumulation rates, outpacing conventional trees in photosynthesis. To leverage its potential as an alternative crop, kenaf must be integrated into value-added products, a practice observed since ancient times. Traditionally used for ropes and canvas, kenaf has evolved into a versatile raw material, replacing wood in pulp and paper industries to mitigate deforestation. Its applications extend to non-woven mats in automotive industries and textiles. With approximately 35% bast fibre and 65% core, kenaf offers diverse applications, including industrial socks for oil spill absorption and woven/non-woven textiles [32, 33]. Notably, kenaf bast fibre stands out for its superior toughness and high aspect ratio, making it an ideal reinforcing fibre in thermoplastic composites, with individual fibres boasting tensile strength and modulus values reaching 11.9 GPa and 60 GPa, respectively [34–36].

Utilising Empty Fruit Bunch Fibres (EFB) as a biocomposite material presents numerous advantages, characterised by its abundant and cost-effective nature, coupled with strong fibre properties and low density [37, 38]. The versatility of EFB-based biocomposites extends to various product applications, bolstering the economic value of Oil Palm Empty Fruit Bunch Fibres (OPEFB) and contributing to sustainable waste management practices [39]. Furthermore, these biocomposites serve as intermediate products for the production of eco-friendly materials, aligning with the growing societal interest and demand for environmentally conscious solutions [40, 41].

A comparison of properties between PLA and typical polymers highlights PLA's superior tensile modulus compared to Poly(vinyl chloride) (PVC), Poly(propylene) (PP), and nylon, as well as its higher flexural strength than PP [42, 43]. These characteristics underscore PLA's potential across various engineering domains. Previous literature from the past two decades has documented significant opportunities for enhancing PLA's physical and mechanical properties. Moreover, PLA offers the advantage of being processed through simple conventional methods, requiring minimal energy and time inputs, thus rendering it a cost-effective and easily accessible polymer [44–47]. Its versatility extends to molding and reshaping into diverse forms using processes like extrusion and injection molding, contributing to its growing adoption in food industries as a substitute for leading polymers.

The use of sustainable natural fibres as reinforcing polymers in biocomposite materials has substantial environmental benefits. The integration of natural fibres not only improves the mechanical strength and thermal resistance of the biocomposite but also enhances its biodegradability and reduces reliance on finite, non-renewable resources, in line with sustainability imperatives and circular economy frameworks [48]. However, these natural fibres frequently have inherent defects that reduce their effectiveness and commercial feasibility. Poor fibre-matrix adhesion, limited compatibility, and susceptibility to degradation can all have an impact on the mechanical strength, durability, and overall performance of reinforced polymer composites. To overcome these issues, it is critical to create treatments that improve fibre characteristics and integration within the polymer matrix. These treatments aim to improve the fibre-matrix interface, enhance adhesion, and reduce degradation while retaining the usability and market potential of the resulting biocomposite goods [49–51].

According to studies, the surface of kenaf and empty fruit bunch fibres can be treated with alkaline solutions to remove lignin and hemicellulose, wax, and oils that create roughness [52–54]. The treatment of kenaf and empty fruit bunch fibres will improve their thermal stability. By efficiently resolving these problems, academics and industry professionals can realise the full potential of sustainable natural fibres as reinforcements in biocomposites, resulting in environmentally acceptable and financially viable solutions for a variety of applications.

Moreover, in recent times, heightened attention is directed towards natural fibres as promising alternatives to plastic in manufacturing and packaging, particularly in Malaysia where these materials are readily available [55–58]. With abundant supplies of natural fibres like kenaf and empty fruit bunch fibres, there exists significant potential for their widespread commercial utilisation, potentially substituting unsustainable materials commonly used to date. The cost-effectiveness of utilising kenaf and empty fruit bunches stems from their abundant plant-derived sources, offering advantages to manufacturers by reducing raw material costs [59–61]. This cost efficiency, in turn, benefits Malaysia's economy by bolstering opportunities in agriculture-based and forestry sectors, reducing dependence on imports and providing cheaper products to consumers. The mechanical properties of composites derived from kenaf and empty fruit bunch fibres depend on the morphologies of the fibres and the interfacial bonding strength with the matrix [62–64]. Treatment of kenaf and empty fruit bunch fibres through alkaline treatments aids in removing lignin, hemicellulose, and surface impurities, thereby enhancing their qualities and thermal stability, as evidenced by previous studies [65–68].

The recent study found that biocomposites have lesser environmental effect than standard polymeric materials. They enable the substitution of conventional composites with environmentally sustainable and potentially cost-effective alternatives through the incorporation of polymers that can be engineered to substantially reduce ecological impact [48]. However, the long-term manufacture and commercial application of biocomposites is heavily reliant on their cost [69, 70]. Biocomposites fabrication with natural fibres has been proposed as a potential method for lowering production costs. Kolozis et al. [71] and Rodriguez et al. [72] used a life cycle cost performance technique to evaluate the economic consequences of biocomposites made from banana fibre. Their approach divided production expenses into four categories: material, energy, labor, and machine. Although the banana fibre was inexpensive, the pre-treatments used for property improvement raised the material cost. However, the scientists discovered that the banana fibre biocomposite had a 17% lower manufacturing cost than the polyester resin. Furthermore, replacing fibreglass with natural fibres lowered production energy costs by 80% and biocomposite costs by 5% [73, 74]. A market research report on global biocomposite usage estimated $19.6 billion in 2020, with an expected increase to $38.07 billion and a compound annual growth rate (CAGR) of 14.2% by 2025. However, the global synthetic fibre market, which was valued at $147.16 billion in 2019, is only predicted to increase at a 5% CAGR by 2025, indicating a drop in consumption rates [47, 75]. Collectively, these market dynamics indicate a progressive shift towards biocomposites as environmentally responsible materials, reinforcing their strategic importance in achieving long-term sustainability goals within the biocomposites sector.

Furthermore, global plant fibre production has expanded dramatically over the past two decades and is projected to increase from 107 million tonnes in 2018 to 145 million tonnes by 2030. With technological innovations aimed at discovering novel composite materials and new applications, coupled with increased investment in research and the commercialisation of finished products, the biocomposites market is expected to expand significantly. This growth will result in substantial economic benefits for the biocomposites industry [76–78]. This field of study is gaining popularity among researchers for the development of innovative polymers and materials with applications across diverse sectors, including nanocomposites, automotive, construction, paper and pulp, electronic sensors, and packaging industries.

Alongside these technological advancements, researchers are increasingly focusing on integrating sustainability principles, particularly the circular economy (CE), into biocomposites development.

The circular economy (CE) in biocomposites production benefits the environment, economy, and society, as illustrated in Fig. 1. Biocomposites require substantial energy for production and have a relatively high unit cost per kilogram; thus, their disposal in landfills represents a loss of high-value materials and economic resources, while also eliminating any potential for material reuse. By exploring the synergistic effects of natural fibres in enhancing the performance of PLA-based biocomposites, this study aims to provide valuable insights into their potential applications across various sectors, including automotive, construction, packaging, and consumer goods. Additionally, the research seeks to optimise processing parameters to achieve the desired properties of hybrid biocomposites, facilitating the development of sustainable and environmentally friendly materials. Moreover, to enhance the effectiveness of the CE, biocomposites

Fig. 1 Circular Economy in biocomposites manufacturing. Figure drawn based on data from Ref [80, 83].

development should integrate the principles of the blue economy, which advocates for the utilisation of ocean-based resources to create high-value products.

Although hybrid natural fibre reinforced biocomposites with PLA have been extensively utilised across various fields with different fibre combinations, the specific combination of PLA with both kenaf and empty fruit bunch (EFB) fibres, in the context of advancing the circular economy, has not been thoroughly investigated in the existing literature [79–84]. Earlier studies have investigated PLA biocomposites reinforced with natural fibres such as kenaf or oil palm empty fruit bunch (EFB), including hybrid fibre systems [84]. However, many of these studies utilised untreated fibres, which often resulted in weak interfacial bonding between the hydrophilic fibres and the hydrophobic polymer matrix, limiting the mechanical performance of the composites.

Furthermore, while hybrid natural fibre systems have been explored, the combined effects of fibre hybridisation and chemical surface treatment on both structural performance and degradation behaviour remain insufficiently investigated. In particular, studies involving kenaf and oil palm empty fruit bunch (EFB) fibres have often utilised untreated fibres, which restricts stress transfer efficiency and limits the overall performance of the biocomposite system [84]. In addition, hybrid kenaf and EFB fibre composites have been explored primarily with other polymer matrices or untreated fibres, which resulted in limited fibre–matrix adhesion and reduced stress transfer efficiency.

In contrast, the present study introduces alkali-treated kenaf and EFB fibres in a PLA matrix, aiming to improve fibre surface cleanliness, remove hemicellulose and lignin components, and enhance fibre–matrix interfacial bonding. The study further evaluates the combined effects of fibre hybridisation and chemical treatment on the mechanical, morphological, and biodegradation behaviour of PLA biocomposites.

Therefore, this study aims to address this gap by investigating the effects of fibre hybridisation and alkaline surface treatment on the structural performance and degradation behaviour of Hybrid kenaf and empty fruit bunch fibres PLA biocomposites. The research focuses on evaluating the mechanical properties, morphological characteristics, and soil burial degradation behaviour of the developed hybrid biocomposites. Mechanical evaluation in this work primarily focuses on tensile behaviour, which provides fundamental insight into fibre–matrix interfacial efficiency and reinforcement effectiveness in hybrid biocomposites.

By integrating agricultural biomass residues with a biodegradable polymer matrix, this work seeks to contribute to the development of environmentally responsible hybrid biocomposite materials with potential applications in lightweight structural components, sustainable packaging, and other resource-efficient material systems.The study also explores how the utilisation of agricultural residues such as EFB fibres can contribute to resource valorisation and waste reduction within the oil palm industry, supporting broader circular economy strategies.

2 Materials and methods

2.1 Materials and instruments

Materials: Empty fruit bunch (EFB) fibres and kenaf fibres were used as natural reinforcement, while poly(lactic acid) (PLA) pellets, (average molecular weight, Mw ≈ 60,000 g. mol^{-1}, Sigma-Aldrich, USA) served as polymer matrix. Sodium hydroxide (NaOH) solution (6 wt%) was used for fibre surface treatment. Prior to treatment, the EFB and kenaf fibres were reduced to lengths of approximately 2–4 cm using a cutting mill (SM 200, Retsch GmbH, Haan, Germany) to facilitate uniform processing and mixing in the biocomposite preparation stage.

Instruments: The main instruments used in this study included an internal mixer (HAAKE Rheomix OS, Thermo Fisher Scientific, Karlsruhe, Germany) for composite compounding, a hydraulic hot press (HEI-50, Hytech Engineers Co., Ltd., Taipei, Taiwan) for compression moulding, a universal testing machine (Instron 5569, Instron, Norwood, MA, USA) (Fig. 2) for tensile testing, and a scanning electron microscope (TM-1000, Hitachi, Krefeld, Germany) for morphological analysis.

2.2 Treatment of fibres

Empty fruit bunch (EFB) and kenaf fibres underwent alkaline treatment to improve fibre surface characteristics and interfacial compatibility with the polymer matrix. Both fibres were treated with a 6 wt% NaOH solution for 3 h at room temperature. The 6wt.% NaOH concentration was selected based on its efficacy in treating kenaf and EFB fibres, as demonstrated by studies conducted by Kamarudin et al. [85], Nasidi et al. [86], Muhammad et al. [87]. Alkali treatment removes surface impurities such as waxes, lignin, and hemicellulose, thereby increasing surface roughness and improving fibre–matrix adhesion. After treatment, the fibres were thoroughly washed with running tap water followed by

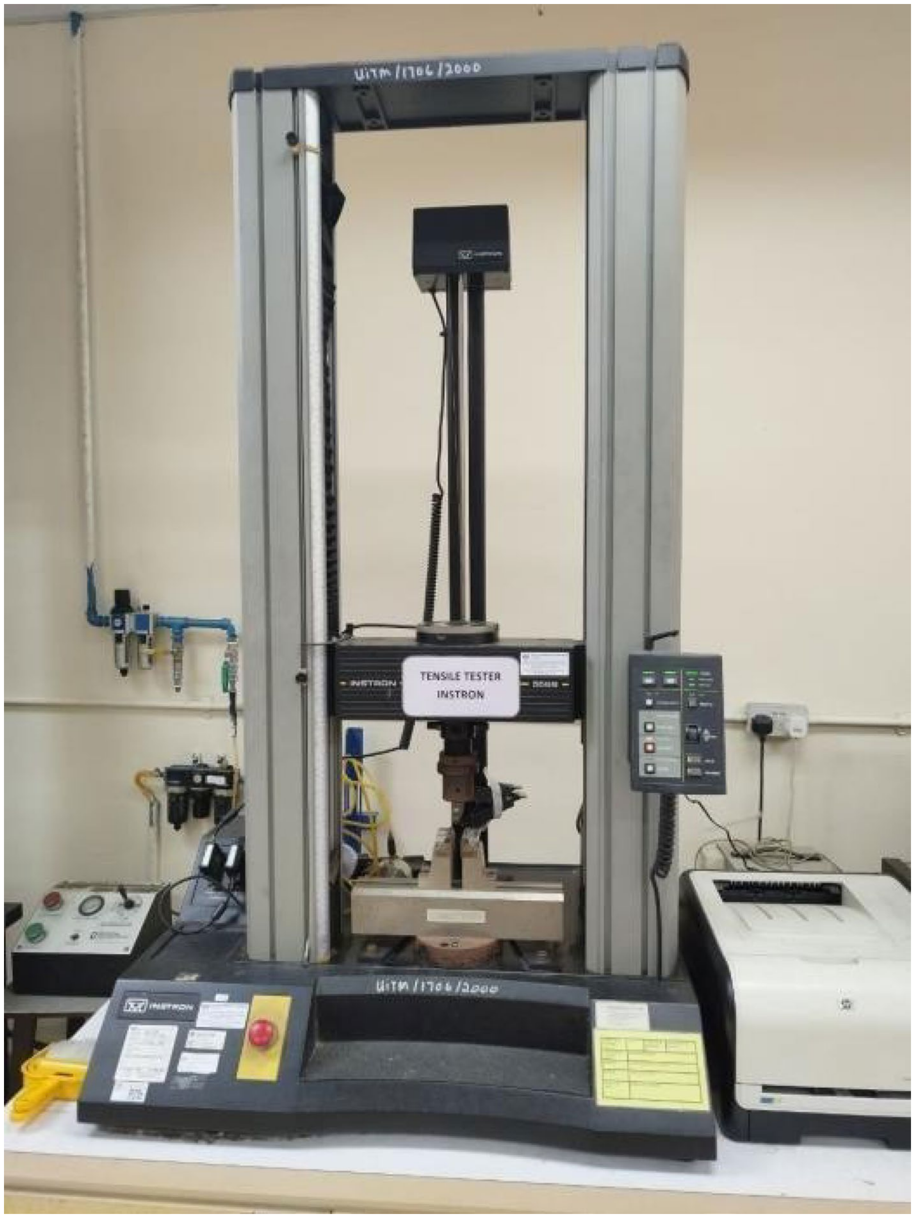


Fig. 2 INSTRON machine for tensile test

distilled water until a neutral pH was obtained. The fibres were subsequently dried in an oven at 80 °C for 3 h to remove residual moisture prior to biocomposite fabrication [88].

2.3 Fabrication of hybrid kenaf-empty fruit bunch fibres PLA biocomposites

Poly(lactic acid) (PLA) pellets were thoroughly blended with kenaf and empty fruit bunch fibres using a Thermofisher internal mixer (HAAKE Rheomix OS, Thermo Fisher Scientific, Karlsruhe, Germany) at $170^{\circ C}$ for 10 min at a rotation speed of 50 rpm. The resulting mixture was then compressed for 10 min at $190^{\circ C}$ using a hydraulic hot press (HEI-50, Hytech, Taipei, Taiwan). The composition of the resulting hybrid natural fibre reinforced biocomposite samples varied, as detailed in Table 1. The PLA and natural fibres were blended using an internal mixer followed by compression moulding, following procedures commonly reported for thermoplastic biocomposite fabrication [89].

Table 1 Formulation ratio composition of hybrid kenaf-empty fruit bunch fibres PLA biocomposite

Types	Ratio composition	PLA (wt%)	EFB (wt%)	Kenaf (wt%)
1	Pure EFB	70	30	0
2	Pure Kenaf	70	0	30
3	Treated Kenaf- EFB/PLA	70	15	15
4	Untreated Kenaf-EFB/PLA	70	15	15
5	PLA	100	0	0

Fibre loading plays a critical role in determining the mechanical performance and processability of biocomposites. In this study, the PLA to fibre ratio of 70:30 wt% was selected based on commonly reported compositions for PLA biocomposites, where fibre contents between 20 and 40 wt% are frequently employed to achieve a balance between mechanical reinforcement, processability, and biodegradability.

Previous studies have reported that fibre contents around 30 wt% often represent an optimal reinforcement level for PLA biocomposites, as this composition provides sufficient fibre content to enhance stiffness and biodegradability while maintaining acceptable melt processability and fibre dispersion within the polymer matrix [89]. At lower fibre loading levels, the reinforcing effect of the fibres may be limited due to insufficient fibre–matrix interaction, whereas excessive fibre loading (> 40 wt%) can lead to fibre agglomeration, increased void formation, and poor wetting of fibres by the polymer matrix. These effects may reduce the overall mechanical performance of the composites.

Therefore, a total fibre loading of 30 wt% was selected in this study to achieve a balanced combination of reinforcement efficiency, structural integrity, and processing stability in the PLA biocomposite system [65].

For the hybrid biocomposite system, an equal distribution of kenaf and EFB fibres (15:15 wt%) was selected to investigate the synergistic interaction between the two natural fibres within the PLA matrix. Hybridisation with equal fibre proportions is commonly adopted as a baseline formulation in hybrid composite studies to ensure balanced reinforcement contributions from each fibre type and to simplify interpretation of the hybrid effect. Investigating additional fibre ratios such as 5:25, 10:20, or 20:10 may provide further insights into the optimisation of hybrid fibre content; however, such parametric optimisation was beyond the scope of the present study and may be considered in future work.

3 Characterisation of hybrid kenaf-empty fruit bunch fibres PLA biocomposites

The biocomposite samples were fabricated into ASTM standard dumbbell shapes from flat sheet samples using an Industrial Co. Ltd. cutter. The specimens' width and thickness were measured at three different points and the average were calculated. Tensile tests were performed utilising the universal testing machine (Instron 5569, Instron, Norwood, MA, USA), following the ASTM D638 standard method, with a cross head speed of 5 mm/min.

To evaluate the biodegradability of the composite samples, an initial measurement of their weight were recorded. The samples were subsequently buried in compost soil at a depth of 7.5 cm and incubated for a period of 10 days. Sampling was conducted at 2-day intervals. During each sampling, the samples were carefully excavated, cleaned to remove any adhering soil, and re-weighed. The biodegradation behaviour of the

PLA-based biocomposites was evaluated using a soil burial degradation test. The procedure was conducted following methods adapted from ISO 17556:2019 and ASTM D5988, where soil serves as a natural microbial inoculum source. Compost soil containing naturally occurring microorganisms was used as the degradation medium to simulate a biologically active environment [90]. The overall weight loss (%) due to the soil burial was then calculated using the following formula in Eq. (1):

$$\%\ \text{Weight loss} = \frac{\mathrm{W_o} - \mathrm{W_f}}{\mathrm{W_o}} \times 100 \quad (1)$$

The $\mathrm{W_o}$ represent the weight of the samples before and $\mathrm{W_f}$ after soil burial. Samples with a higher weight loss % indicate faster degradation.

Although the degradation duration was relatively short, the test was designed to provide preliminary insight into the early-stage biodegradation behaviour of the hybrid PLA/kenaf/EFB biocomposites under microbially active soil conditions.

The density of hybrid biocomposite samples had been measured based on ASTM D 792 standard by using the densimeter (MD-300 S, Alfa Mirage Co. Ltd., Osaka, Japan).

For scanning electron micrographs, the morphology of fractured samples from the tensile test were analysed using a scanning electron microscope (TM-1000, Hitachi, Krefeld, Germany) following standard sample preparation procedures reported in biocomposite morphology studies [91]. Prior to analysis, a thin layer of gold was applied to enhance visual inspection. Following gold spraying, the samples were scanned using an accelerating voltage of 1.0 kV.

4 Results and discussion

All mechanical and physical tests were conducted with at least three replicates ($n = 3$), and the results are presented as mean ± standard deviation. Statistical analysis was performed to evaluate the variability of the measurements and ensure reliability of the reported data. Error bars representing the standard deviation are included in the figures to indicate experimental variation.

4.1 Tensile properties of hybrid kenaf-empty fruit bunch fibres reinforced PLA biocomposites

The tensile strength of the hybrid kenaf-empty fruit bunch fibres PLA biocomposites is presented in Fig. 3. Neat PLA material exhibited the highest tensile strength of 37.47 MPa, which is consistent with previously reported values for PLA-based materials in the range of 30–60 MPa depending on processing conditions [92]. The incorporation of natural fibres reduced the tensile strength, where kenaf/PLA (KF/PLA) showed 9.9 MPa, while EFB/PLA exhibited 13.87 MPa. The hybrid treated kenaf/EFB/PLA biocomposite demonstrated a slightly higher tensile strength (14.55 MPa) compared with the untreated hybrid biocomposite (12.93 MPa), suggesting improved fibre–matrix interaction after alkaline treatment.

Figure 4 illustrates the tensile modulus of the hybrid kenaf-empty fruit bunch fibres PLA biocomposites. The tensile modulus of neat PLA was 22,658 MPa, whereas EFB/PLA and KF/PLA biocomposites showed reduced modulus values of 8,856 MPa and 7,503 MPa, respectively. Similar reductions in mechanical strength after natural fibre incorporation have been reported in PLA-based biocomposites due to fibre

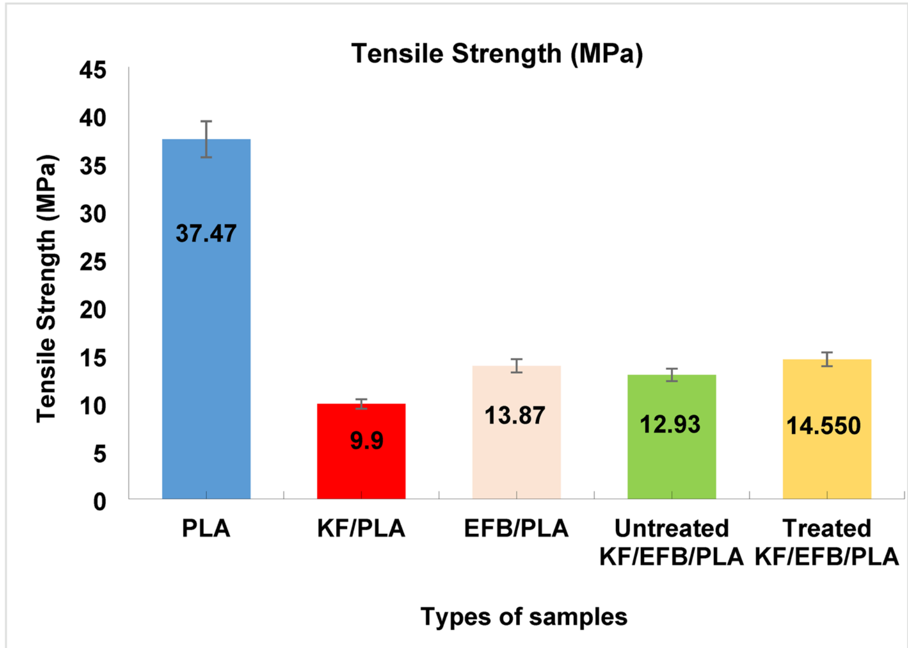


Fig. 3 Tensile strength of PLA, treated and untreated hybrid kenaf-empty fruit bunch fibres reinforced PLA biocomposites

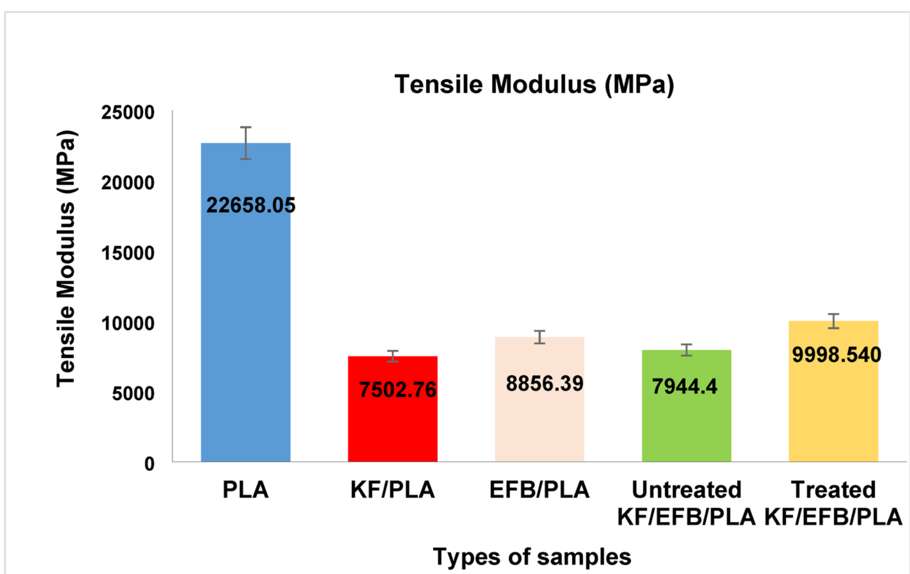


Fig. 4 Tensile modulus of PLA, treated and untreated hybrid kenaf-empty fruit bunch fibres reinforced PLA biocomposites

agglomeration and imperfect interfacial bonding [93–95]. This difference may be attributed to variations in fibre morphology, cellulose content, and fibre–matrix interfacial adhesion, which influence the stress transfer efficiency within the biocomposite structure.

Among all the samples tested, the pure PLA sample recorded the highest tensile strength and tensile modulus, which is expected given the absence of fibre reinforcement, whereas the addition of kenaf and EFB fibres reduced these properties [92, 93].

When kenaf fibres (KF/PLA) and EFB fibres (EFB/PLA) were incorporated, both the tensile strength and tensile modulus were lower compared to pure PLA. This reduction is attributed to the weaker fibre materials acting as stress concentrators, thereby hindering load transfer within the biocomposite structure [94, 95].

Based on the result obtained from this test, it can be shown that EFB/PLA has a higher tensile strength and tensile modulus compared to kenaf/PLA biocomposite. This can be due to the superior mechanical properties of EFB compared to kenaf as EFB may possess higher strength and stiffness making them better able to withstand applied forces [63, 96]. However, the hybridisation of untreated kenaf/EFB/PLA has lower tensile strength and modulus than EFB/PLA, possibly due to the weaker fibre addition from kenaf. Another factor that can influence these differences is the interfacial adhesion between the fibres and PLA matrix [63, 97]. The stronger bonding between fibre and matrix allows for efficient stress transfer from matrix to the fibre which in this case the EFB, resulting in improved mechanical properties [64, 98, 99].

The comparison between untreated and treated kenaf/EFB/PLA biocomposites demonstrates a slight improvement in both tensile strength and tensile modulus following the treatment process. This enhancement can be attributed to the removal of impurities and improved fibre-matrix adhesion achieved through the treatment [100–103]. However, the improvement is relatively modest. The treated kenaf/EFB/PLA biocomposites exhibit higher tensile strength compared to the untreated ones. The treatment process with 6% NaOH modified the fibre surfaces, enhancing their compatibility with the PLA matrix and promoting a stronger interfacial bond. This, in turn, leads to improved stress transfer and increased tensile strength. Additionally, the surface roughness of the samples can differ; the untreated samples tend to have rougher surfaces.

The observed decrease in tensile strength and tensile modulus in the biocomposite materials represents a trade-off for the enhanced biodegradability and other desirable properties imparted by the natural fibres. These results suggest that the primary focus of these biocomposites is on their sustainability and environmental friendliness, rather than just focusing on achieving high mechanical performance.

When compared with previously reported PLA-based biocomposites, the tensile strength obtained in this study is within the range reported for untreated fibre reinforced PLA systems. For instance, kenaf/PLA composites reported by Faruk et al. [25] showed tensile strengths between 10 and 25 MPa depending on fibre loading and surface treatment, while hybrid PLA biocomposites typically exhibit values between 12 and 30 MPa depending on fibre dispersion and processing methods. These comparisons indicate that the mechanical behaviour observed in this study is consistent with trends reported in recent literature.

In this study, tensile testing was selected as the primary mechanical evaluation method because tensile properties are highly sensitive to fibre–matrix interfacial bonding and stress transfer efficiency in fibre-reinforced biocomposites. Although impact strength and flexural properties are also important for assessing the structural performance of biocomposite materials, the focus of the present work was to evaluate the influence of fibre hybridisation and alkaline treatment on tensile behaviour and interfacial interactions within PLA-based biocomposites. Future studies will include additional mechanical tests such as flexural and impact testing to provide a more comprehensive assessment of the mechanical performance of the hybrid kenaf/EFB reinforced PLA biocomposites.

4.2 Soil burial test properties of hybrid kenaf-empty fruit bunch fibres reinforced PLA biocomposites

The degradation behaviour of the biocomposites was evaluated using a soil burial test, and the results are presented as mean weight loss percentages, as shown in Fig. 5. The inclusion of natural fibres significantly influenced the degradation behaviour of the PLA matrix due to the hydrophilic nature of lignocellulosic fibres, which facilitates moisture uptake and microbial colonisation.

The weight loss percentages of various biocomposites materials were measured after a 10-day soil burial test, with the results presented in Fig. 5. The pure PLA material exhibited no weight loss throughout the test duration, maintaining a constant measurement of 0%. For the kenaf/PLA (KF/PLA) biocomposite, the weight loss percentages were as follows: day 2 (0%), day 4 (0%), day 6 (0.05%), day 8 (0.09%), and day 10 (0.13%). This trend indicates a gradual increase in weight loss over time, suggesting some level of degradation.

Similarly, the weight loss percentages for the EFB/PLA biocomposite were: day 2 (0%), day 4 (0%), day 6 (0.04%), day 8 (0.06%), and day 10 (0.09%). These results also indicate a slight increase in weight loss over the test period, suggesting degradation of the biocomposite.

For the untreated kenaf/EFB/PLA biocomposite, the weight loss percentages were: day 2 (0%), day 4 (0.09%), day 6 (0.16%), day 8 (0.29%), and day 10 (0.32%). These results demonstrate a progressive increase in weight loss, indicating material degradation over time.

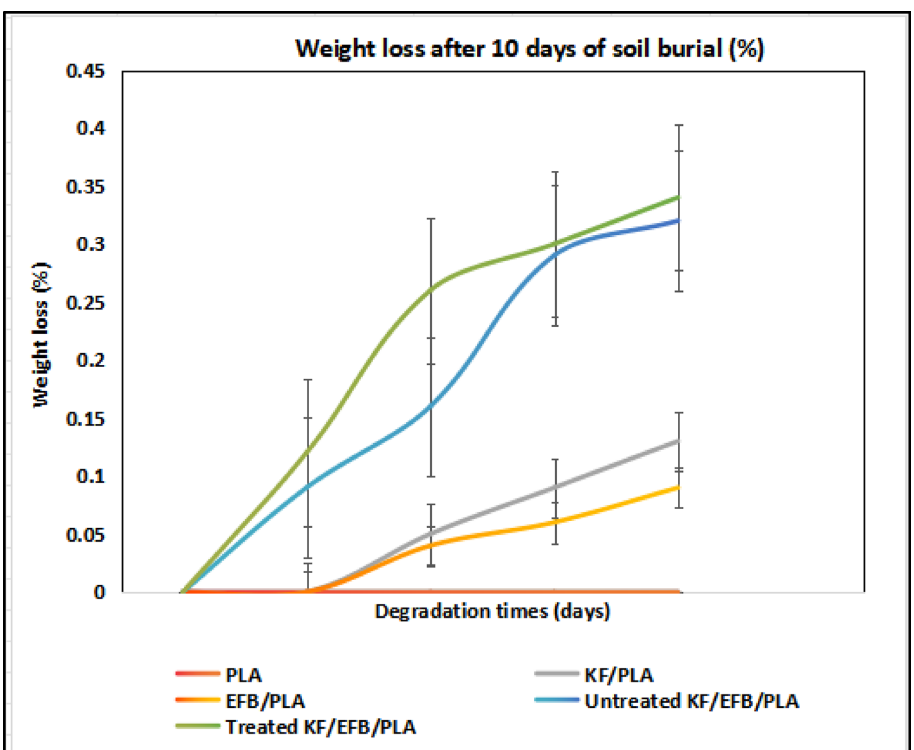


Fig. 5 Degradation rate of PLA, treated and untreated hybrid kenaf-empty fruit bunch PLA biocomposites in 10 days

The treated kenaf/EFB/PLA biocomposite showed weight loss percentages as follows: day 2 (0%), day 4 (0.12%), day 6 (0.26%), day 8 (0.30%), and day 10 (0.34%). Similar to the untreated composite, the treated biocomposite exhibited an increasing trend in weight loss, suggesting material degradation.

The weight loss percentages observed in the soil burial test provide insights into the degradation behavior of the different biocomposites materials. Pure PLA is known for its biodegradable properties, indicating its capacity to undergo natural decomposition over time [104]. However, the timeframe for biodegradation can vary depending on several factors, such as environmental conditions, microbial activity, and material composition.

In the specific context of our experiments, a 10-day soil burial test was conducted. While this period provides some insights into the initial degradation behaviour and fibre-induced degradation mechanisms, longer exposure periods are required to observe substantial structural breakdown of PLA-based composites. The 10-day test period offers a glimpse into the early biodegradation tendencies of PLA, but longer durations are required to fully evaluate the extent of its biodegradability. Therefore, further investigations over extended periods are necessary to comprehensively understand the complete biodegradability of PLA.

The initial lag phase observed during the first four days may be attributed to the microorganisms' efforts to acclimate and adapt to their new environment [105]. It can be seen that the longer the burial time, the higher the weight loss of the biocomposite, indicating increased degradation over time. The incorporation of natural fibres in the biocomposite has accelerated the degradation rate of the samples, resulting in more pronounced weight loss after 10 days of soil burial [106].

The kenaf/PLA (KF/PLA) and EFB/PLA biocomposites exhibited some level of degradation, as indicated by the gradual increase in weight loss percentages over time. This suggests that the inclusion of kenaf fibres or EFB fibres in the PLA matrix renders the biocomposites more susceptible to degradation under soil burial conditions.

Among the hybrid compositions, treated Kenaf/EFB/PLA exhibits the highest biodegradability. The alkaline treatment process removes lignin and other cellulosic components, making the fibres more accessible to microorganisms for degradation [107]. Comparing the single-fibre composites, Kenaf/PLA has a higher degradation rate than EFB/PLA. This is attributed to the higher cellulose content in kenaf, which makes it more susceptible to microbial attack and enzymatic degradation in soil. In contrast, the high lignin content in EFB acts as a barrier, reducing the accessibility of microorganisms and enzymes [108].

Hemicelluloses have been reported to be the fibre component most responsible for moisture absorption and biodegradation [109]. This factor contributes to the higher weight loss observed in the hybrid biocomposite, as the addition of kenaf facilitates microbial activity in the soil. However, there were no observable color changes in the specimens, likely due to the short burial time.

The use of compost soil in this method accelerates the degradation of PLA and natural fibres more effectively than other types of soil environments. PLA is known to be biodegradable under specific conditions, and compost soil provides an environment rich in microorganisms and organic matter, which enhances the degradation process. Composting involves the breakdown of organic materials through the activity of microorganisms such as bacteria, fungi, and other decomposers. These microorganisms secrete enzymes that break down organic compounds, including biodegradable plastics like PLA, resulting in the weight loss observed in the biocomposites. The presence of moisture, oxygen, and optimal temperature in compost soil creates favorable conditions for microbial activity and enzymatic degradation. However, the degradation rate of PLA in compost soil can vary depending on factors such as the specific composition of the compost, temperature, moisture content, and the size or thickness of the PLA item.

The natural fibres used in this research, namely kenaf and empty fruit bunches, also degrade faster in compost soil. Composed of cellulose, hemicellulose, lignin, and other organic materials, these fibres are susceptible to degradation by microorganisms present in the soil, particularly bacteria and fungi [110]. Bacteria and fungi produce enzymes capable of breaking down the organic components of natural fibres.

It should be noted that the soil burial experiment in this study was conducted for 10 days to evaluate the early-stage degradation behaviour of the PLA-based biocomposites. PLA is known to exhibit relatively slow biodegradation under natural environmental conditions, and complete degradation may require extended periods ranging from several weeks to months depending on temperature, microbial activity, and soil conditions. Therefore, the short experimental duration in this work primarily provides insight into the initial degradation trend and the influence of natural fibre incorporation on the degradation behaviour of the biocomposites. The weight loss observed during soil burial is attributed to the combined effects of microbial activity and hydrolytic degradation of the PLA matrix and lignocellulosic fibres in the compost soil environment. Longer-term degradation studies would be necessary to fully evaluate the biodegradation performance of the hybrid PLA biocomposites under environmental conditions.

The degradation behaviour of the biocomposites was assessed through soil burial testing by monitoring the percentage weight loss of the samples over the exposure period. Although microbial activity in the soil was not quantitatively determined in this study, mass loss measurements obtained from soil burial tests are widely employed as a practical indicator for evaluating the degradation behaviour of biodegradable biocomposites under natural environmental conditions. The observed weight reduction is therefore reasonably attributed to a combination of microbial activity and hydrolytic degradation processes occurring in the soil environment. Nevertheless, further investigations employing molecular-level characterisation techniques, such as gel permeation chromatography (GPC), would provide deeper insight into polymer chain scission and the detailed degradation mechanisms of the biocomposites.

4.3 Density of hybrid kenaf-empty fruit bunch fibres reinforced PLA biocomposites

The effect of alkaline treatment on the densities of untreated and treated hybrid PLA/kenaf/EFB biocomposites is shown in Fig. 6.

Incorporating EFB and kenaf fibre into the PLA matrix changed the density of PLA. Neat PLA was (1.226 g/cm^3) compared with the untreated and treated PLA kenaf biocomposites which are 1.220 g/cm^3 and 1.228 g/cm^3. The density of PLA with the treated kenaf and EFB biocomposite is 1.237 g/cm^3.

Generally, the introduction of filler affects the packing density and free volume of the polymer chain. The incorporation of natural fibres influences the packing structure of the polymer matrix and may introduce microvoids or interfacial gaps, which can slightly alter the overall density of the biocomposite system. Therefore, increased filler

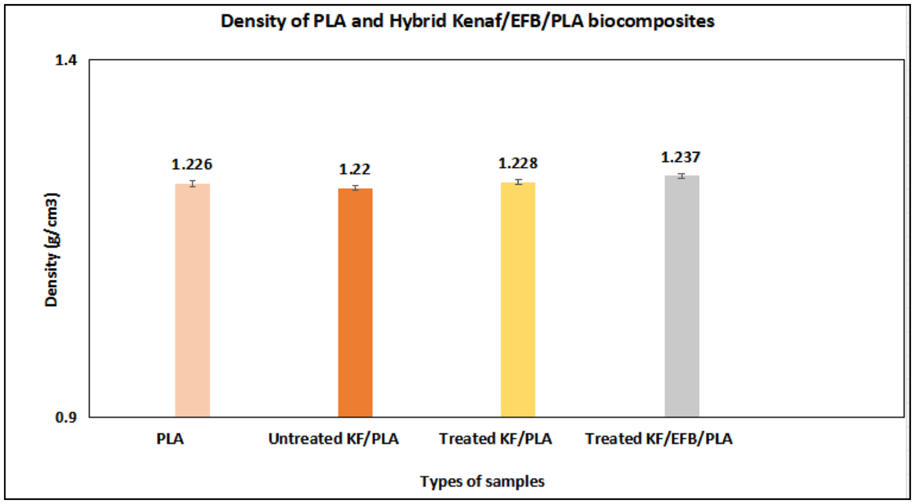


Fig. 6 Density of Pure PLA and Hybrid Kenaf/EFB/PLA biocomposites

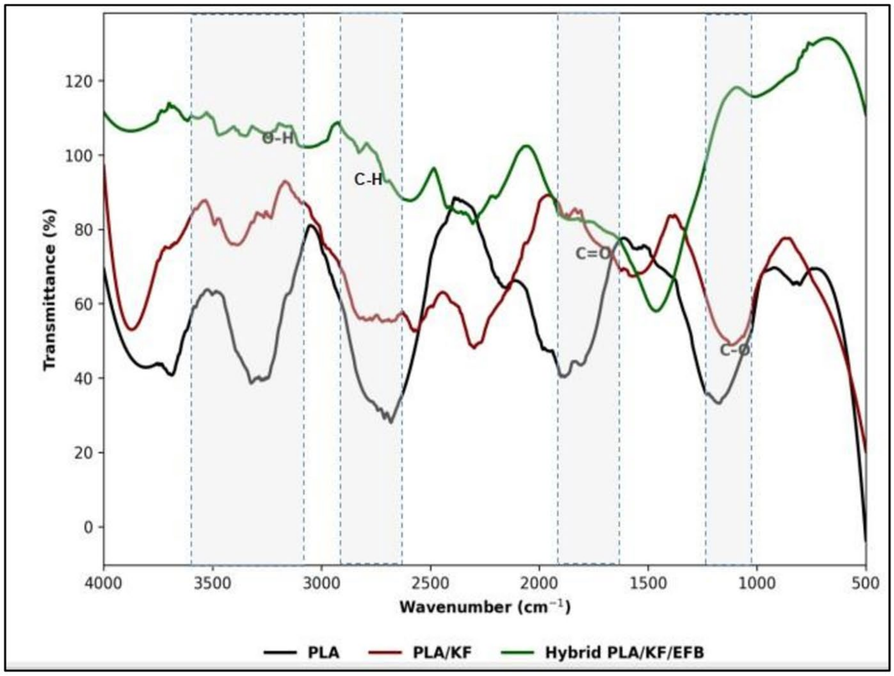
concentration increases free volume, hence leading to the density reduction visible in Fig. 6. Low density leads to lighter weight biocomposites. Both film sheets were prepared using a dry processing technique (hot pressing). In this study, when the treated kenaf and EFB was incorporated into the PLA, it caused softening by reducing the intermolecular attractive forces among the chains in the PLA [67, 111, 112].

Treated hybrid biocomposites had higher densities compared with untreated hybrid biocomposites. As anticipated, the percentage of void content was much lower for the treated hybrid biocomposites compared with untreated hybrid biocomposites [113–115]. This result was due to the efficiency of the alkaline treatment in improving fibre-matrix adhesion and dispersion in the hybrid biocomposites. Thus, it was observed that the treatment of the fibre helped reduce the voids of the PLA matrix interface, as supported by the SEM images [116–118].

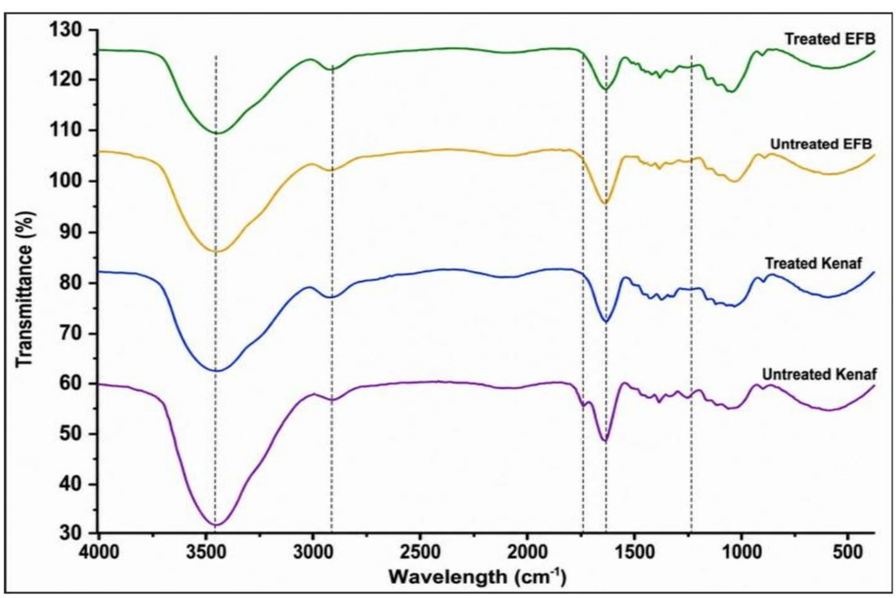
4.4 Fourier transform infrared of hybrid kenaf-empty fruit bunch fibres reinforced PLA biocomposites

Fourier-transform infrared spectroscopy (FTIR) was employed to identify the functional groups and chemical interactions present in neat poly(lactic acid) (PLA), untreated and alkali treated kenaf and empty fruit bunch fibres, as well as the resulting hybrid PLA based biocomposites. The FTIR spectra of neat PLA, PLA/kenaf biocomposites and PLA/kenaf/EFB hybrid biocomposites are presented in Fig. 7, allowing comparison of the characteristic absorption bands and evaluation of potential interfacial interactions between the polymer matrix and natural fibres.

In the wavenumber region of 3200–3600 cm^{-1}, a broad absorption band corresponding to O–H stretching vibrations was observed. This peak is associated with hydroxyl groups originating from cellulose, hemicellulose, and lignin components present in kenaf and EFB fibres.The broadening and slight shift of this peak toward lower wavenumbers in the biocomposite spectra indicate the formation of intermolecular hydrogen bonding between the hydroxyl groups of the fibres and the ester groups of the PLA matrix, suggesting partial interfacial interaction between the reinforcement and the polymer matrix. The gradual increase in the intensity and broadness of the O–H band with increasing kenaf fibre content suggests enhanced hydrogen bonding interactions



(a)



(b)

Fig. 7 FTIR of (**a**) Pure PLA, PLA/Kenaf, Hybrid PLA/Kenaf/EFB (**b**) Treated EFB, Untreated EFB, Treated Kenaf, Untreated Kenaf between the fibres and the PLA matrix. However, due to the relatively inert backbone structure of PLA, which mainly contains ester linkages without reactive side chains, the chemical bonding between PLA and natural fibres is primarily governed by physical interactions and hydrogen bonding rather than strong covalent bonding.

In addition, characteristic absorption bands were detected in the region of 2820–2940 cm^{-1}, corresponding to C–H stretching vibrations of $-CH_2$ and $-CH_3$ groups which are associated with lignin structures and aliphatic chains within both the fibres and PLA matrix [119, 120]. The presence of these peaks confirms the coexistence of lignocellulosic fibre components and the PLA polymer phase within the biocomposite systems.

For PLA/Kenaf/EFB hybrid biocomposites, the FTIR spectrum exhibited characteristic peaks corresponding to both PLA and lignocellulosic fibres, confirming the successful incorporation of the natural reinforcements within the polymer matrix. The prominent C=O stretching peak at approximately 1759 cm^{-1}, characteristic of the ester groups in PLA, remained visible in the hybrid composite spectrum. Meanwhile, the presence of O–H stretching bands (3503–3362 cm^{-1}) and C–O stretching bands (1213–1091 cm^{-1}) indicates the contribution of cellulose-rich fibres within the composite structure. The coexistence of these functional groups suggests the formation of a hybrid biocomposite system in which the PLA matrix and natural fibres interact mainly through hydrogen bonding and interfacial physical interactions.

The FTIR analysis further revealed differences between untreated and alkali-treated kenaf and EFB fibres, which provide insight into the structural modifications induced by chemical treatment (Fig. 7b). The FTIR spectra of untreated kenaf and EFB fibres exhibited broad transmittance bands within the 1041.03–3463.35 cm^{-1} range, reflecting the presence of lignocellulosic components including cellulose, hemicellulose, and lignin. Strong absorption peaks at 1636.63 cm^{-1} (C=O stretching) and 3458.36 cm^{-1} (O–H stretching) further confirmed the presence of hydroxyl and carbonyl functional groups in the untreated fibres [121].

The FTIR spectra of untreated kenaf and EFB fibres exhibited several characteristic bands within the 1041–3463 cm^{-1} region, representing typical lignocellulosic constituents including cellulose, hemicellulose, and lignin. Strong absorption peaks observed at 3458 cm^{-1} (O–H stretching) and 1636 cm^{-1} (C=O stretching) further confirmed the presence of hydroxyl and carbonyl functional groups in the untreated fibres [121]. Additional peaks at 1736 cm^{-1} (C=O stretching of ester groups), 1245 cm^{-1} (C–O stretching of alcohol groups), and 1380 cm^{-1} (C–C stretching of alkanes) were also detected, which are commonly associated with hemicellulose and lignin structures in natural fibres.

Upon alkali treatment, significant spectral changes were observed. The disappearance of peaks corresponding to C=O (1736.26 cm^{-1}) and C–O (1245.35 cm^{-1}) indicated the effective removal of hemicellulose and partial delignification of the fibre structures. Alkali treatment promotes saponification and hydrolysis reactions, which break ester linkages within hemicellulose and facilitate the dissolution of lignin components. Furthermore, the O–H hydroxyl peaks (3458.36 and 1636.63 cm^{-1}) and the ester C–O peak (2893.19 cm^{-1}) became broader and less intense compared to untreated fibres. This reduction in intensity suggests partial elimination of hydroxyl and ester functional groups, reflecting the degradation and removal of amorphous hemicellulose and lignin fractions. Such structural modifications are consistent with the action of alkali treatment in disrupting hydrogen bonding and dissolving non-cellulosic components, thereby enhancing the exposure of cellulose fibrils [122]. These structural modifications improve the fibre surface roughness and enhance the potential for interfacial adhesion with the PLA matrix.

Overall, these FTIR findings confirm that alkali treatment effectively induces degradation and removes non-cellulosic components from kenaf and EFB fibres, including hemicellulose and lignin, thereby enhancing cellulose exposure and improving fibre–matrix interfacial adhesion. This structural modification enhances the potential for interfacial adhesion and compatibility with the PLA matrix, which is crucial for optimising the mechanical performance and interfacial bonding of the hybrid PLA biocomposites.

4.5 Scanning electron micrographs of hybrid kenaf-empty fruit bunch fibres reinforced PLA biocomposites

SEM micrographs of the pure EFB and kenaf fibres, tensile fracture surfaces of neat PLA, and hybrid untreated and treated PLA/Kenaf/EFB biocomposites are presented in Fig. 8. SEM micrographs of the fracture surfaces provide insight into the interfacial interactions between the fibres and the PLA matrix. The fracture surface of neat PLA exhibited an irregular and relatively rough morphology, which is characteristic of brittle fracture behaviour [123]. The failure mechanism is attributed to micro-crack initiation followed by rapid crack propagation, allowing cracks to propagate freely throughout the polymer matrix due to the absence of reinforcing phases capable of dissipating applied stress.

For the pure EFB, the SEM images revealed the presence of irregular particle-like features on the fibre surface, which can be attributed to the silica bodies naturally present in empty fruit bunch (EFB) fibres [124]. These silica deposits are commonly found in oil palm biomass and may contribute to surface heterogeneity of the fibres. Meanwhile, for the pure kenaf, noticeable voids and interfacial gaps could be seen within the surface. These voids are likely associated with the hollow lumen structure of kenaf fibres and inadequate wetting of the fibre surface. The presence of such voids can act as stress concentration sites, facilitating crack initiation and propagation under tensile loading [125]. Consequently, poor fibre–matrix interfacial bonding and internal void formation contribute to reduced mechanical performance.

In the hybrid untreated PLA/Kenaf/EFB biocomposites, the SEM micrographs showed a more complex fracture morphology due to the presence of two different fibre types within the matrix. While the hybridisation of fibres can potentially improve stress distribution within the composite system, the untreated hybrid samples still exhibited fibre pull-out, interfacial debonding, and microvoids. These features indicate incomplete interfacial compatibility between the hydrophilic fibres and the relatively hydrophobic PLA matrix, limiting the effectiveness of load transfer.

In contrast, in the hybrid treated PLA/Kenaf/EFB biocomposites, there is a noticeable increase in surface interaction and adhesion between the fibres and polymer matrix. The SEM images revealed that the fibres were more firmly embedded within the PLA matrix, with fewer observable gaps and reduced fibre pull-out. Alkali treatment removes surface impurities such as hemicellulose, lignin, waxes, and oils, thereby increasing fibre surface roughness and enhancing mechanical interlocking with the polymer matrix. The micrographs of the hybrid fibre treated biocomposite show a smooth fractured surface, indicating strong fibre adhesion to the polymer matrix. Effective adhesion between fibres

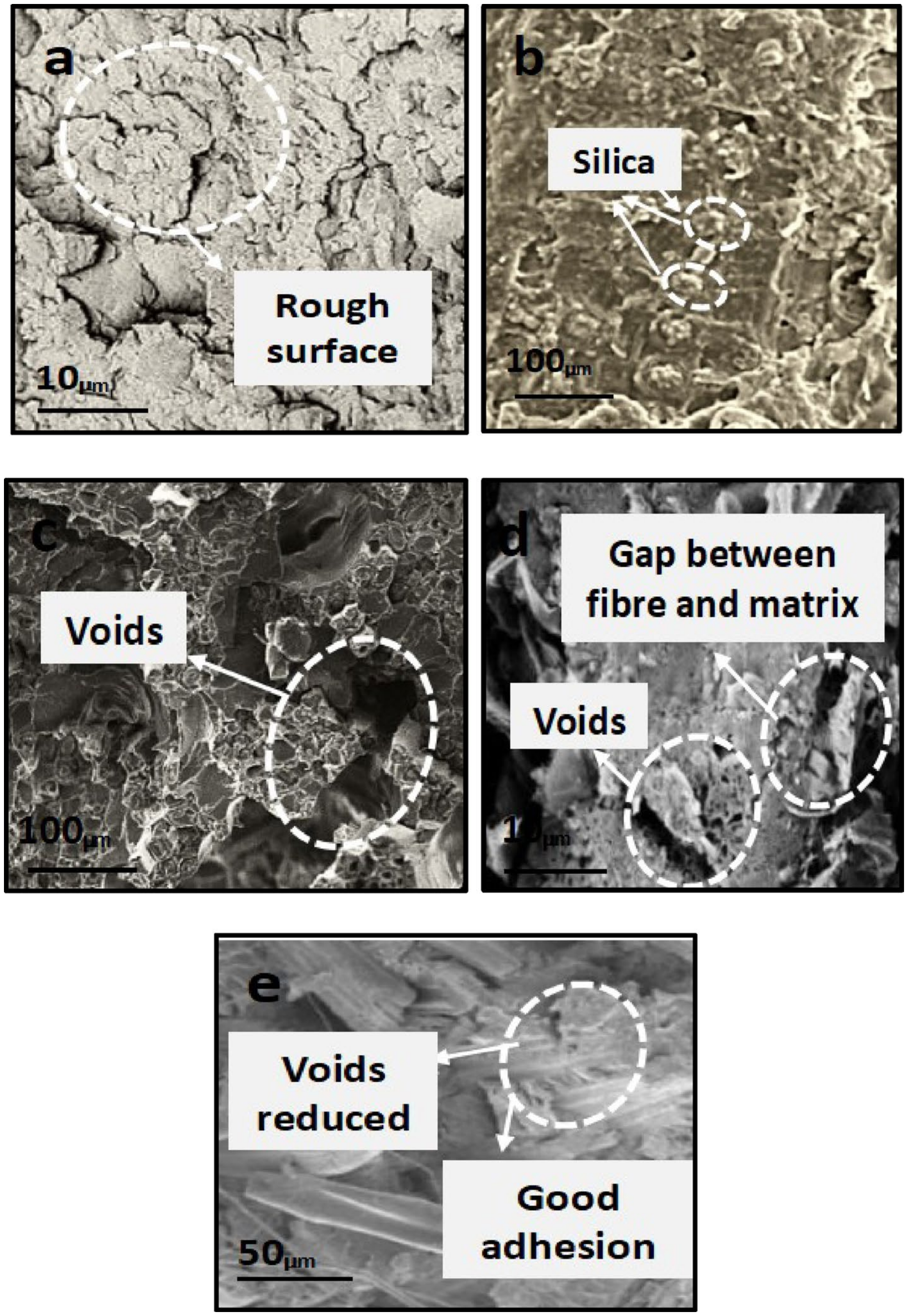


Fig. 8 SEM micrographs of tensile fracture surfaces showing morphological characteristics of the biocomposites (**a**) Neat PLA; (**b**) Pure EFB; (**c**) Pure Kenaf; (**d**) Hybrid Untreated PLA/Kenaf/EFB biocomposite (**e**) Hybrid Treated PLA/Kenaf/EFB biocomposite

and the matrix is crucial for enhancing the mechanical properties of biocomposites [126].

The SEM micrographs reveal improved fibre dispersion and reduced interfacial gaps in the hybrid biocomposite, suggesting enhanced fibre–matrix interaction, which may contribute to improved mechanical behaviour. The presence of well-embedded fibres and reduced fibre pull-out suggests improved interfacial interaction between the fibres and PLA matrix, which may contribute to the mechanical behaviour observed in the tensile tests. Therefore, incorporating both fibres promotes favorable wettability properties, reduces the formation of voids at the fibre-polymer interface, and yields composites with high stiffness and strength, ultimately contributing to a higher storage modulus [127, 128]. Comparative morphological observations provide qualitative evidence supporting the mechanical performance and degradation behaviour of the hybrid PLA biocomposites.

Overall, the SEM observations demonstrate that fibre hybridisation combined with alkali treatment significantly improves the interfacial compatibility between the natural fibres and the PLA matrix, resulting in enhanced structural integrity and mechanical behaviour of the hybrid biocomposites.

5 Conclusion

The current study demonstrates that incorporating kenaf and EFB fibres as fillers in the PLA matrix enhances the hybrid biocomposites with favorable qualities. Hybridising EFB and kenaf fibres improved tensile strength and modulus, indicated that the high mechanical properties of one fibre were able to compensate for the lower mechanical properties of another fibre. Akali treatment with 6% NaOH enhanced interfacial bonding and increased strength. The presence of two different fibres promoted good wettability properties, reducing the formation of voids at the fibre-polymer interface and resulting in hybrid biocomposites with high stiffness and strength. Longer burial times resulted in higher degradation rates, with natural fibres accelerating the process. Among the biocomposites tested, the hybrid treated PLA/kenaf/EFB biocomposite exhibited the highest biodegradability, highlighting the potential of natural fibre-based biocomposites as environmentally friendly materials with enhanced properties. The study further illustrated that hybridising these fibres leads to improved the mechanical properties. Overall, the FTIR analysis confirmed that alkali treatment effectively removed hemicellulose and lignin, thereby reducing the intensity of hydroxyl and ester groups and exposing more cellulose fibrils. This structural modification not only enhanced the fibre surface cleanliness but also increased the availability of functional groups that can interact with the PLA matrix. Consequently, the improved fibre–matrix interfacial compatibility is expected to contribute to better stress transfer and enhanced mechanical performance in the resulting biocomposites. Continued research and development in this area could lead to the development of biocomposites with even greater strength and performance, providing sustainable alternatives to traditional materials. For instance, exploring alternatives to kenaf could be beneficial. The biodegradation tests underscored the influence of burial time and natural fibres on degradation rates. The hybrid treated PLA/kenaf/EFB biocomposites developed in this study show potential for several sustainable material applications. Owing to their improved mechanical properties and biodegradable nature, these materials could be utilised in automotive interior components, packaging materials, household product casings, and non-load-bearing construction panels. The use of oil palm empty fruit bunch fibres, an abundant agricultural residue, promotes biomass valorisation and reduces reliance on petroleum-derived reinforcements. In the context of the circular economy, the integration of renewable fibres with biodegradable polymers offers a promising pathway for developing environmentally responsible materials that can reduce long-term plastic accumulation, carbon footprint and support sustainable resource management. Future research could expand to investigate the long-term biodegradation behavior of these biocomposites under various environmental conditions, such as extending burial times to 3–6 months, to further enhance understanding of their sustainability and potential applications. Furthermore, future studies incorporating microbial population analysis or soil microbiological characterisation would provide deeper insight into the biodegradation mechanisms of the hybrid biocomposites. Moreover, incorporating GPC analysis would allow a more detailed understanding of the molecular degradation mechanisms of the biocomposites.

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Author contributions
Writing—original draft preparation, Investigation, Data curation, Methodology, Writing- reviewing and editing: S.H.Kamarudin; Editing, Investigation: N. Suhaimi, S.N.S.Mohamad, H.B.Hafiz, S.Ikawati, U.H.Abdullah, M.S.M.Basri.

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Data availability
The datasets generated and/or analysed during the current study are available from the corresponding author on reasonable request.

Declarations

Ethics approval and consent to participate
Not applicable.

Consent for publication
Not applicable.

Competing interests
The authors declare no competing interests.

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