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Characterization of Bio-Retted Kenaf (*Hibiscus Cannabinus L.*) Bast Fibre

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ABSTRACT

The bio-retted kenaf (Hibiscus cannabinus L.) bast fibres offer significant advantages over synthetic fibre regarding their being lightweight, environmentally friendly, and cheap. During the characterization procedure, the chemical composition as well as the physical, thermal, mechanical, crystallinity, and morphological properties of bio-retted kenaf (Hibiscus cannabinus L.) bast fibre were assessed. The chemical composition analysis of the bio-retted bast fibre of kenaf (H. cannabinus L.) revealed that it contained a significantly high proportion of cellulose (58.72%). While, in the tensile test, the bast fibre of bio-retted kenaf (Hibiscus cannabinus L.) exhibited an average tensile strength of 3876 ± 1122 MPa and modulus of elasticity of 577 ± 177 GPa. X-ray diffraction (XRD) analysis of the bio-retted kenaf (Hibiscus cannabinus L.) bast fibre revealed its crystalline size to be 19.65 nm and crystallinity index to be 68.62%. The thermogravimetric analysis (TGA) performed on the bast fibre of kenaf (Hibiscus cannabinus L.) revealed that it maintains a thermal stability of 240°C. The scanning electron microscope (SEM) was employed to examine the fibre's morphology. The fibre exhibited flaws and a smoothed surface, as determined by the morphological evaluation. The utilisation of bio-retted bast fibre derived from kenaf (Hibiscus cannabinus L.) as a reinforcing agent in applications involving elevated temperatures exhibited encouraging outcomes.

Keywords: Bio-retted kenaf (hibiscus cannabinus l.); mechanical properties; crystallinity index; thermogravimetric analysis; morphological

INTRODUCTION

For numerous reasons, including biodegradability, affordability, abundant availability, non-toxicity, low density, recyclable nature, and environmental goodness, natural fibres are a widely adopted technology in composite materials that holds significant potential. The increasing market demand for composites derived from natural fibres has resulted in a notable surge in the prominence of kenaf fibres. This, in turn, has recently propelled the global cultivation of kenaf as an industrial crop. (Abbas et al. 2022). Commonly referred to as kenaf, the *Hibiscus cannabinus L.* plant produces an annual warm-season fibre crop. It belongs to the Malvaceae botanical family. As a perennial plant, kenaf (*Hibiscus cannabinus L.)* is inexpensive, lightweight, abundant, environmentally friendly, and has low density and good thermal properties. It also has little impact on climate change (Chung et al. 2018). Regardless of the conditions, kenaf (*Hibiscus cannabinus L.*) plant can attain a height of nearly three metres in just three months and develop heart-shaped leaves along a strong, straight stem. Under optimal environmental conditions, kenaf (*Hibiscus cannabinus L.*) plant has the potential to develop at a rate of 10 cm per day (Akil et al. 2011). Kenaf (*Hibiscus cannabinus L.*) was introduced 4000 years ago originated with roots in ancient Africa and is now successfully produced throughout the world. As a dicotyledonous plant, kenaf (*Hibiscus cannabinus L.)* has three major layers in its stem or stalk. Phloem constitutes the outer cortical layer, at times referred to as the bast tissue layer. The inner woody layer, xylem, is also referred to as the central pith layer and the core tissue layer. It consists primarily of nonferrous cells and a substance looking like a sponge (Ashori et al. 2006). More than 20 countries in the commercial cultivation of kenaf (*Hibiscus cannabinus L.)* and the top producers of kenaf (*Hibiscus cannabinus L.)* for fiber production included countries like China, India, Bangladesh, and Thailand. India, Pakistan, Indonesia, Japan, Thailand, Vietnam, and Malaysia are among the countries where kenaf (*Hibiscus cannabinus L.)* is the native plant (Ashori et al. 2006).

Retting or degumming is usually carried out to extract bast fibres. This is the process by which the fibres are isolated from the stem so that the non-cellulosic substances that are adhered to them can be eliminated. To generate individual filaments, pectin and other cementitious compounds are eliminated during the process (Rozyanty et al. 2021). Chemical, mechanical, enzymatic, bio-retting, water, and dew retting are just a few of the numerous retting processes. Enzymatic retting is the retting process where enzymes such as pectins and xylanases are used to detach the gum and pectin material in the bast (Tholibon et al. 2019). In the bio-retting process, microorganisms, including bacteria and fungi, play a crucial role in breaking down the non-cellulosic components, that bind the fibers to the plant stalks. Bio-retting process is the process involving the function of enzymatic activities, whether direct microbial application or using their extracted enzymatic formulations (Hossain et al. 2021). This bio-retting process will produce good quality and high-strength fibre. By avoiding the application of detrimental chemicals, this process ensures environmental sustainability and minimizes the risk it poses to the ecosystem.

Numerous scientific studies in the area of creating composite structures based on high-strength polymers have made it abundantly evident how important natural fibres are, despite their relative weakness in comparison to synthetic fibres. Manimaran *et al*. (2020) characterized natural cellulosic fibres from the nendran banana peduncle plant. The nendran banana peduncle plant can withstand temperatures as high as 365°C, according to the researchers. This suggests that it could be a suitable material for reinforcing light-load vehicle components and construction machinery (Manimaran et al. 2020). Moshi *et al.* (2020) characterized natural cellulosic fibre extracted from Grewia damine flowering plant's stem. Based on their studies, the stem of the Grewia damine flowering plant was determined to be appropriate for lightweight applications due to its reduced density (1.378±0.036 g/cm3) (Moshi et al. 2020). Kamaruddin *et al.* (2021) characterized natural cellulosic fibre isolated from Malaysian cymbopogan citratus leaves. According to their report, the tensile test results for Cymbopogan citratus fibre indicated an average tensile strength of 43.81 ± 15.27 MPa and a modulus of elasticity of 1.046 ± 0.33 GPa. This implies that the fibre has the potential to serve as a reinforcing element in thermoplastic green composites (Kamaruddin et al. 2021). To date, several studies have reported on the characterization of fibres as reinforcement in composite materials but, none has been found on the characterization of bio-retted kenaf (*Hibiscus cannabinus L.*) bast fibres that obtained from National Kenaf and Tobacco Board (NKTB-LKTN) in Kelantan. The purpose of this study is to gain further understanding of the chemical, physical, mechanical, and thermal properties of the bio-retted kenaf (*Hibiscus cannabinus L.*) bast fibres. From this study, we will have a better understanding of how this natural fibre might be incorporated into additional types of biodegradable products. Number of studies have been conducted to investigate the usage of bio-retted kenaf (*Hibiscus cannabinus L.*) bast fibres and their composites. Thus, this study set out to investigate the characterization of bio-retted kenaf (*Hibiscus cannabinus L.*) bast fibres for their properties that include density, chemical composition, mechanical properties, thermal stability, morphology, and functional groups. Comparable results were observed and analyzed in relation to different varieties of natural fibres and other types of retting process for bio-retted kenaf (*Hibiscus cannabinus L.*) bast.

EXPERIMENTAL

MATERIAL

The grade A kenaf (*Hibiscus cannabinus L.)* bast fibres were obtained as curled long fibres from kenaf bark with 700-1200mm length from the bio-retting process as in Figure 1. They were sourced locally from National Kenaf and Tobacco Board (NKTB-LKTN) in Kelantan, Malaysia. As for bio-retting process, it was done at National Kenaf and Tobacco Board (NKTB-LKTN). The kenaf ribbons were soaked in normal fresh water for the microbial degradation process (Kamaruddin et al. 2021) for 12 hours. Next, the mixture of water and acid was prepared in a tank and left for 12 hours. Kenaf ribbons were transferred into the tank and a mixture of enzymes and water was added into the tank. The kenaf ribbons in the tank were periodically monitored for three days. Next, the retted kenaf ribbons were washed using water jet and later, the fibres were sun-dried to further eliminate the residual moisture until less than 12%. The fibres were graded according to colour, cleanliness and moisture level before they were delivered.

FIGURE 1. Grade A bio-retted kenaf (*Hibiscus cannabinus L.*) bast fibres

METHODS

CHEMICAL COMPOSITION

The chemical composition of bio-retted kenaf (*Hibiscus cannabinus L.)* bast fibre was examined using Van Soest technique via neutral detergent fiber (NDF), acid detergent fiber (ADF), and acid detergent lignin (ADL) as established by Van Soest and Wine (1968). This investigation was carried out by the Malaysian Agricultural Research and Development Institute (MARDI) using the FOSS Fibertec 2010 Auto Fibre Analysis System.

PHYSICAL PROPERTIES

By using an optical microscope, Olympus (SZ61, Tokyo, Japan) size of ten individual bio-retted kenaf (*Hibiscus cannabinus L.)* bast fibre were measured. The average measurement was determined using ten different locations, and the mean value and standard deviation were subsequently computed. The density was measured using the densimeter Model Precissa XT 220A. This densimeter was employed to measure density by ASTM D792-08 at room temperature. The data analysis for this sample involved calculating the mean value and standard deviation after ten replications of the measurement.

TENSILE PROPERTIES

ASTM D 3379-75 standard was followed in conducting single-fibre tensile test of the fibres. Thus, ten individual fibres of bio-retted kenaf (*Hibiscus cannabinus L.)* bast fibres were randomly identified, and mounted using a mounting tab as illustrated in Figure 2. These tests were carried out by employing an Instron universal testing machine model 5567 (Norwood, Massachusetts, USA). A crosshead speed of 1 mm/min was implemented with 1 kN load cell capacity and 50 mm gauge length.

FIGURE 2. Sample arrangement for tensile test

THERMAL CHARACTERIZATION

The thermal stability of the bio-retted kenaf (*Hibiscus cannabinus L.)* bast fibres were studied using Perkin Elmer Simultaneous Thermal Analyzer (STA) 6000. In ceramic pan, samples containing 5 - 7 mg of the fibre were heated at a constant heating rate of 10°C /min from 25°C to 600°C in a nitrogen atmosphere.

SCANNING ELECTRON MICROSCOPY

A scanning electron microscope instrument model Hitachi Tabletop Microscope TM 1000 (Tokyo, Japan) was used to observe the morphology of the bio-retted kenaf (*Hibiscus cannabinus L.)* bast fibre. A 15 kV acceleration voltage was applied. The micrographs were obtained by enlarging at various magnifications on a 30-mm long sample.

ATTENUATED TOTAL REFLECTANCE-FOURIER TRANSFORM INFRARED SPECTROSCOPY (ATR-FTIR)

FTIR spectrometer (Spectrum 100, Perkin Elmer, USA) using MIRacle ATR (PIKE Technologies, Madison, USA) was applied to identify the characteristic peaks of infrared transmission spectra of bio-retted kenaf (*Hibiscus cannabinus L.)* bast fibre. Using a pressure clamp, the sample was spread onto the zinc selenide crystal's surface to maximize sampling sensitivity and maintain close contact. It was characterized at a resolution of 4 cm^{-1} with a scanning time of 1.5 min over a wavenumber range of 4000 to 660 cm^{-1}.

X-RAY DIFFRACTION (XRD)

Crystallization and amorphous state of bio-retted kenaf (*Hibiscus cannabinus L.)* bast fibre were analysed using a Rigaku Ultima IV X-ray Diffractometer (Rigaku, Tokyo, Japan) with Cu radiation run at 40 kV and 40 mA. A diffraction angle (2θ) ranging from 10° to 50° and scanning rate of 2 ° min^{-1} (Hassan et al. 2018) were employed to scan the samples. According to Eq. 1, the bio-retted kenaf (*Hibiscus cannabinus L.)* bast fibre's crystallinity index (CI) was calculated using following Segal expression (Kamaruddin et al. 2021).

$$CI = \frac{I_{002} - I_{am}}{I_{002}} x\ 100\% \qquad (1)$$

I_{002} represents the peak intensity of the crystalline fraction and I_{am} represents amorphous fractions, respectively. As illustrated in Eq. 2, the crystalline size (CS) was calculated by utilising Scherrer's formula (Kamaruddin et al. 2021).

$$CS = \left(\frac{k\lambda}{\beta cos\theta}\right) \qquad (2)$$

The wavelength of radiation, denoted as $\lambda = 0.1541$ nm, β is the peak's full width at half-maximum in radians, θ is the corresponding Bragg angle and $k = 0.89$ is Scherrer's constant.

RESULTS AND DISCUSSION

CHEMICAL COMPOSITION

The major components in bio-retted kenaf (*Hibiscus cannabinus L.)* bast fibre are cellulose, hemicellulose, and lignin. The crystalline and amorphous regions of cellulose collaboratively, work together to provide the fibre its strength, rigidity, and stability, and they also aid in the stability of the stem and plant wall (Jawaid et al. 2022). Hemicellulose is highly hydrophilic, and amorphous and has low molecular weight compared to cellulose (Kamaruddin et al. 2022). Biodegradation, absorption of moisture, and the thermal degradation of the fibre are all related to hemicellulose. Therefore, the UV deterioration of the fibre is caused by lignin, which is thermally stable (Arjmandi et al. 2021). The comparison results from the chemical composition analysis of the various retted kenaf (*Hibiscus cannabinus L.)* bast fibre are presented in Table 1. It can be seen that 58.72% of cellulose content is present on the surface of bio-retted kenaf (*Hibiscus cannabinus L.)* bast fibre, which is comparable to water-retted and higher than mechanical-retted kenaf. Thus, this bio-retting process produced high cellulose content even though the retting process was faster than water-retting. The cellulose content is also higher than *Corchorus olitorius* (45–71.5%) (Selver et al. 2018), *Cajanus cajan* (55.03%) (Kulandaivel et al. 2020), bamboo pulp (26 to 43%) (Kamaruddin et al. 2021), *Leucas Aspera* (50.7%) (Vijay et al. 2021), *Carica papaya bark* (58.71%) (Moshi et al. 2020) and lesser than *Nendran banana peduncle* (73.20%) (Manimaran et al. 2020), *Coccinia grandis. L* (62.35%) (Senthamaraikannan & Kathiresan, 2018), and *Ceiba pentandra bark* (60.9%) (Moshi et al. 2020). Many scholars kept in view that a higher concentration of cellulose improves mechanical properties such as tensile strength and elastic modulus. However, the strength of the fibre is negatively impacted by the increased hemicellulose content in natural fibre (Perumal & Sarala, 2020). The investigation has shown that the surface of bio-retted kenaf (*Hibiscus cannabinus L.)* bast fibre contained 15.66% hemicellulose, which is lower than water-retted and mechanical-retted kenaf. This indicated that the gummy material, hemicellulose could be removed by the bio-retting process. The lignin content of bio-retted kenaf (*Hibiscus cannabinus L.)* bast fibre was noted at 16.07%, which serves as a barrier to keep out bacterial attacks. The comparison results from the chemical composition analysis of the bio-retted kenaf (*Hibiscus cannabinus L.)* bast fibre from different bio-retting techniques are presented in Table 2. It can be seen that time factor and different water sources on the retting process affect the chemical composition of kenaf. It is assumed that the sufficient removal of non-cellulosic gums (NCGs) occurred for longer retting process and proportionally increase in the cellulose content. The removal of hemicellulose and lignin proportionally increases the cellulosic fibre (Hossain et al. 2022).

TABLE 1. Chemical composition of bio-retted kenaf (*Hibiscus cannabinus L.)* bast fibre and other retted kenaf bast fibres

Fibre	Cellulose (%)	Hemicellulose (%)	Lignin (%)	Reference
Bio-retted kenaf *(Hibiscus cannabinus L.)*	58.72	15.66	16.07	Current study
Water-retted kenaf *(Hibiscus cannabinus L.)*	58.1	16.1	14.1	(Hassan et al. 2018)
Mechanical-retted kenaf *(Hibiscus cannabinus L.)*	54.4	46.5	18.27	(Ismail et al. 2021)

TABLE 2. Chemical composition of bio-retted kenaf (*Hibiscus cannabinus L.)* bast fibre and other bio-retted kenaf bast fibres

Bio-retting technique	Cellulose (%)	Hemicellulose (%)	Lignin (%)	Reference
Freshwater and enzymes (3d)	58.72	15.66	16.07	Current study
Seawater (11d)	79.70 ± 1.48	9.7 ± 0.58	7.78 ± 0.76	(Hossain et al. 2022)
Freshwater (11d)	77.58 ± 0.86	9.18 ± 0.55	8.60 ± 60	
Municipal wastewater (11d)	73.15 ± 1.48	11.70 ± 0.62	10.46 ± 0.64	
Fresh pond water (6d)	56.34±2.41	16.60±1.05	13.35±0.28	(Hossain et al. 2021)
Fresh pond water and Bacillus sp. KRB56 (6d)	69.21±1.04	9.75±0.41	8.24±0.22	
Fresh pond water and Bacillus sp. KRB22 (6d)	67.42±1.07	10.73±0.63	13.35±0.28	

*d denotes days

PHYSICAL PROPERTIES

The mechanical properties of the fibre, especially its tensile strength, is heavily dependent on the size measurement. An optical microscope was used to measure the size of bio-retted kenaf (*Hibiscus cannabinus L.)* bast fibre. The fibre's average size was calculated as 18 ± 10 µm, which was comparably lower than other natural fibres. The main elements influencing the physical properties are the fibre's source, plant condition, plant maturity, and the extraction procedure (Reddy & Yang, 2005).

As it impacts the overall weight of composite materials fabricated from natural fibres, fibre density measurement is an essential element of any characterization study. The density of bio-retted kenaf (*Hibiscus cannabinus L.)* bast fibre was 1.269 ± 0.26 g/cm^3, which was relatively lower than other natural fibres, for example, *Coccinia Grandis Stem* (1.5175 g/cm^3) (Jebadurai et al. 2019), *Grewia damine stem* (1.378±0.036 g/cm^3) (Moshi et al. 2020), and *Thespesia populnea bark* (1.412 g/cm^3) (Kathirselvam et al. 2019). The environment and the plant's rate of growth are two factors that might lead the density value to vary (Moshi et al. 2020). The comparison of the physical properties of bio-retted kenaf (*Hibiscus cannabinus L.)* bast fibre is presented in Table 3. The current study found that the density of bio-retted kenaf (*Hibiscus cannabinus L.)* is lower than mechanical-retted kenaf.

TABLE 3. Physical properties of bio-retted kenaf (*Hibiscus cannabinus L.)* bast fibre with other retted kenaf bast fibres

Fibre	Diameter (µm)	Density (g/cm^3)	Reference
Bio-retted kenaf *(Hibiscus cannabinus L.)*	18 ± 10	1.269 ± 0.26	Current study
Water-retted kenaf *(Hibiscus cannabinus L.)*	110	-	(Hassan et al. 2018)
Mechanical-retted kenaf *(Hibiscus cannabinus L.)*	-	1.33	(Ismail et al. 2021)

TENSILE PROPERTIES

On average, a single fibre had a tensile strength of 3876 ± 1122 MPa, an elasticity modulus of 577 ± 177 GPa, and elongation at break of 1.23 ± 0.3%. The bio-retted kenaf (*Hibiscus cannabinus L.)* bast fibre's tensile strength is influenced by the plant's age, its source, extraction method, moderate value, and the microstructure of the fibre, wherein cracks originating from larger flaws will cause fibre defeat (Bezazi et al. 2014). Therefore, to accurately determine the tensile properties with accurate, at least three duplicates of each fibre sample should be subjected to testing. The enhanced tensile strength of natural fibre is mostly due to their high crystallinity index and abundant cellulose content (Kumaar et al. 2019). In contrast to different natural fibre varieties, the strength of bio-retted kenaf (*Hibiscus cannabinus L.)* bast fibre was higher than *Sansevieria cylindrical* (673.12±51 MPa) (Manimaran et al. 2020), *Sansevieria ehrenbergii* (50-585 MPa) (Kamaruddin et al. 2021) and *Thespesia populnea* (557.82 ± 56.29 MPa) (Kathirselvam et al. 2019), the bio-retted kenaf (*Hibiscus cannabinus L.)* bast fibre's tensile strength showed a moderate value with *Cissus quadrangulari* (1857– 5330 MPa) (Manimaran et al. 2020). Despite having a common structure, natural fibres vary in numerous ways, including lumen internal area, lumen number, fibre cell size and number, secondary cell wall thickness, and cell cross-section. Each fibre's tensile strengths are integrated by its tensile properties. Nevertheless, the tensile properties results show considerable diversity in their shapes, which are not exactly spherical. A comparison of the tensile properties of bio-retted kenaf (*Hibiscus cannabinus L.*) bast fibre is reported in Table 4. The tensile strength of bio-retted kenaf (*Hibiscus cannabinus L.)* bast fibre is remarkably higher than water-retted and mechanical-retted kenaf, which may be due to the extraction process of bio-retting that used an enzyme. It is assumed that during the bio-retting process with the aid of enzyme, pectinase and xylanase activities occurred. These activities enhance the bio-retted kenaf *(Hibiscus cannabinus L.)* bast fibre's tensile strength. This finding was confirmed by Hanana *et al.* that enzymatic treatment of alfa fibres exhibited high tensile properties compared to untreated fibres (Hanana et al. 2015). Fibre strength can also be attributed to the size and structure of the fibre. Due to some defects, it is well recognized that the fibre strength reduces with increasing diameter (Duval et al. 2011). The diameter of bio-retted kenaf *(Hibiscus cannabinus L.)* bast fibre was lower than water-retted kenaf fibre. The bio-retting process with the aid of enzyme had reduced the fibre diameter. Due to the reduction in fibre's diameter, the tensile properties of the bio-retted kenaf *(Hibiscus cannabinus L.)* bast fibre exhibited high tensile properties.

TABLE 4. Tensile properties of bio-retted kenaf (*Hibiscus cannabinus L.)* bast fibre with other retted kenaf bast fibres

Fibre	Tensile Strength (MPa)	Tensile Modulus (GPa)	Elongation at Break (%)	Reference
Bio-retted kenaf *(Hibiscus cannabinus L.)*	3876 ± 1122	577 ± 177	1.23 ± 0.3	Current study
Water-retted kenaf *(Hibiscus cannabinus L.)*	190	24	-	(Hassan et al. 2018)
Mechanical-retted kenaf *(Hibiscus cannabinus L.)*	159.98	8.2	-	(Ismail et al. 2021)

THERMOGRAVIMETRIC ANALYSIS

One major drawback of the application as reinforcing agent is their limited thermal stability at high temperatures. The TGA studied the bio-retted kenaf (*Hibiscus cannabinus L.)* bast fibre's thermal behaviour and determined the suitability of bio-retted kenaf (*Hibiscus cannabinus L.)* bast fibre for high-temperature engineering applications. Figure 3 shows the TGA curve, which reveals three phase of degradation. According to Hassan et al. (2018), the first degradation happened between 50 and 100°C as a result of water or moisture evaporation. The bio-retted kenaf (*Hibiscus cannabinus L.)* bast fibre lost 13.12% of its weight due to moisture evaporation, which happened between 80 and 95 °C. During the second degradation, which happened at temperatures ranging from 280 to 340°C, around 37.66% of the fibre's weight was lost due to hemicellulose was eliminated from the fibre's surface. In the temperature range of 220 to 315°C, hemicellulose decomposes frequently (Yang et al. 2007). Figure 3 illustrates that the hemicellulose degradation process began around 280 °C and was finished at about 315 °C. This result agrees with the findings of other studies, in which hemicellulose degradation temperature range from 220 to 315°C. Since it comprises heterogeneous polysaccharides including galactose, xylose, glucose, and mannose, which are readily devolatilized at low temperatures due to their amorphous nature, hemicellulose is the first component to degrade during thermal analysis (Yang et al. 2007). Derivative thermogravimetry (DTG) graph in Figure 2 shows that cellulose decomposed from 280 to 340 °C as demonstrated by the clear U-shaped peak found at 338 °C. Char or tar from the decomposition of the primary components and lignin decomposed between 340 to 600°C (Yao et al. 2008). Pyrolysis of lignin and oxidative degradation charred residue both produce small peaks at 450°C and 535°C, respectively in the DTG curve (Hassan et al. 2018). Due to several aromatic rings with different branches and functional groups, lignin degrades at a wide range of temperatures. (Yang et al. 2007). The excess material after the completion of lignin degradation is known as residual mass. The residual mass of bio-retted kenaf (*Hibiscus cannabinus L.)* bast fibre was in the range of 20 to 30%. In general, the thermal stability of the bio-retted kenaf *(Hibiscus cannabinus L.)* bast fibre can be considered as 240°C, which is comparable with *Cissus Quadrangularis root* (230°C) (Indran et al. 2014) and *Althaea Officinalis L.* (220°C) (Moshi et al. 2020). A comparison of the thermal properties of bio-retted kenaf (*Hibiscus cannabinus L.)* bast fibre is reported in Table 5. The thermal stability of bio-retted kenaf (*Hibiscus cannabinus L.)* bast fibre is comparable with water-retted and mechanical-retted kenaf and also comparable with jute, hemp, flax and coir as presented in Table 6. Due to their superior comparable thermal properties to synthetic fibres, natural fibre-reinforced polymer composites, like those made of jute or hemp have a wide range of uses as engineering materials in the automotive, aerospace and construction industries (Nurazzi et al. 2021).

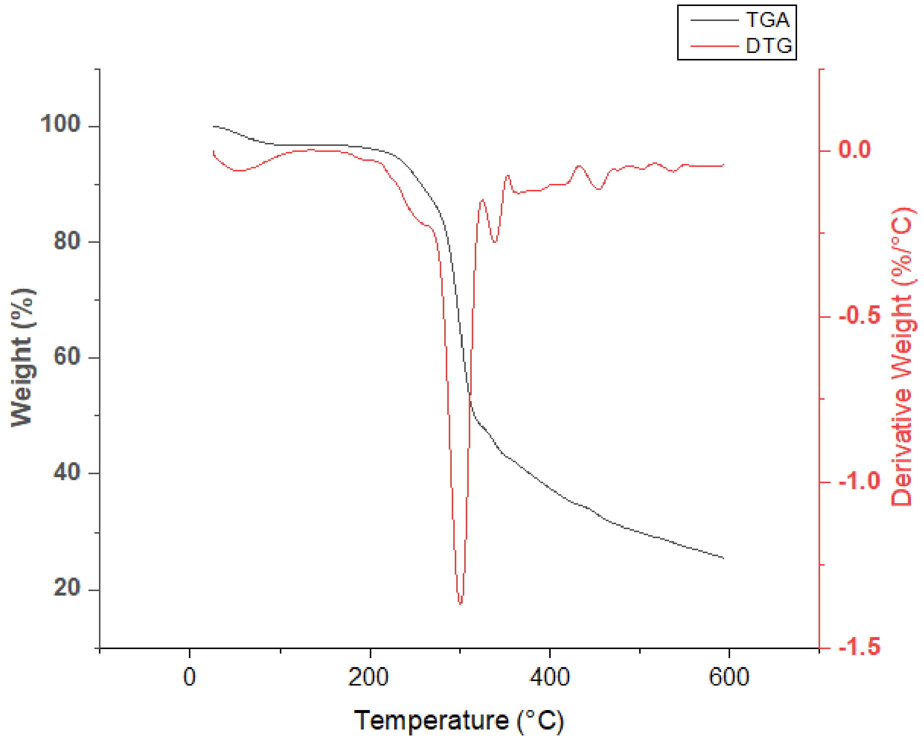

FIGURE 3. TGA curve for bio-retted kenaf (*Hibiscus cannabinus L.)* bast fibre

TABLE 5. Thermal properties of bio-retted kenaf (*Hibiscus cannabinus L.)* bast fibre with other retted kenaf bast fibres

Fibre	Temperature of Initial Decomposition (°C)	Maximum Decomposition Temperature (°C)	Reference
Bio-retted kenaf *(Hibiscus cannabinus L.)*	240	302	Current study
Water-retted kenaf *(Hibiscus cannabinus L.)*	259	337	(Hassan et al. 2018)
Mechanical-retted kenaf *(Hibiscus cannabinus L.)*	277.79	377.39	(Ismail et al. 2021)

TABLE 6. Thermal properties of bio-retted kenaf (*Hibiscus cannabinus L.)* bast fibre with other natural fibres

Fibre	Temperature of Initial Decomposition (°C)	Maximum Decomposition Temperature (°C)	Reference
Bio-retted kenaf *(Hibiscus cannabinus L.)*	240	302	Current study
Jute	205	340	(Jawaid et al. 2022)
Hemp	250	390	(Kamaruddin et al. 2021)
Flax	230-260	330	(Chaishome & Rattanapaskorn 2017)
Coir	200-250	313.4	(Ezekiel et al. 2011)

MORPHOLOGICAL CHARACTERISTICS

The mechanical and physical properties of the natural fibres, which indicate their potential as reinforcing material for composites, are significantly impacted by their shape. Figure 4 (a) and (b) show micrographs of the surface fibre in longitudinal view, obtained by observing the bio-retted kenaf (*Hibiscus cannabinus L.)* bast fibre's surface morphology at various magnifications. Figure 4 shows a scanning electron micrograph (SEM) of bio-retted kenaf (*Hibiscus cannabinus L.)* bast fibre, this fibre could reap benefits from suitable fibre treatments in order to reduce its bounded hemicellulose and cellulose content. Good interaction between the matrix and fibre would be facilitated by fibre treatments. (Moshi et al. 2020). The white layer in the micrograph represents the hemicellulose that was found on the surface of the fibre. The fibre surface also showed indicators of impurities, and it has a smooth texture. Therefore, achieving sufficient strength involves surface modification.

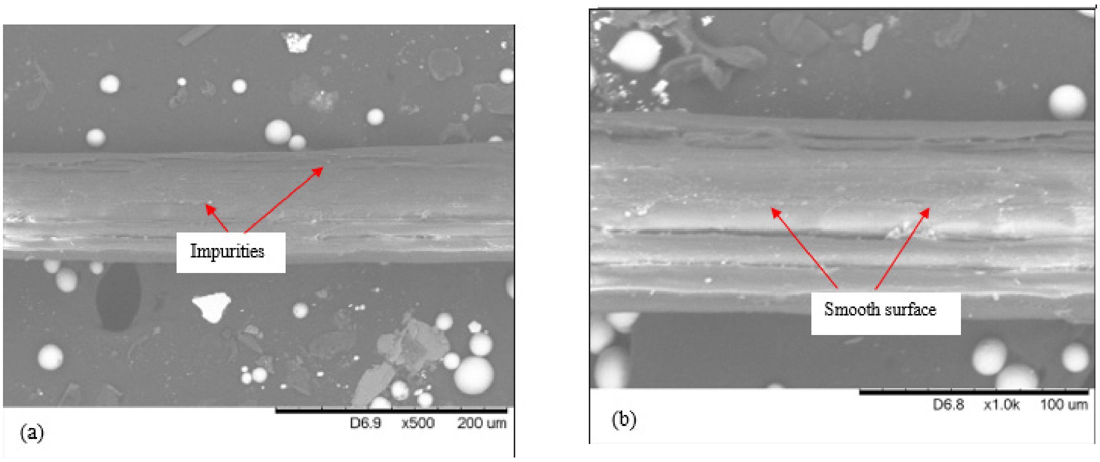

FIGURE 4. SEM images of the surface structure of bio-retted kenaf (*Hibiscus cannabinus L.)* bast fibre (a) 500X magnification and (b) 1000X magnification

FOURIER TRANSFORM INFRARED (FTIR) ANALYSIS

The Fourier Transform Infrared spectrum observed from bio-retted kenaf (*Hibiscus cannabinus L.)* bast fibre between the range of 4000 to 660 cm^{-1} which is shown in Figure 5. The presence of a hydroxyl (O-H) group in the cellulose contents was confirmed by the detection of a large concentrated peak at 3367 cm^{-1} (Moshi et al. 2020). The presence of cellulose and hemicellulose was demonstrated by the peak at 2792 cm^{-1}, which was related to the C-H stretching vibration of CH and CH_2 (Kamaruddin et al. 2021). The stretching vibration of carbonyl acid (C=O) / acetyl group linkage of lignin and hemicellulose in bio-retted kenaf (*Hibiscus cannabinus L.)* bast fibre was ascribed to the high sharp peak observed at 1626 cm^{-1} (Manimaran et al. 2020). The cellulosic polysaccharides constituents attributed to C-O stretching showed decreasing intensity is accountable for the wide peak at 1035 cm^{-1} (Hossain et al. 2021). The cellulose β

– glycosidic bonds linked with the lignin, are accountable for the peak at 826 cm^{-1} (Kathirselvam et al. 2019). The chemical bonds identified corresponding to the notable peaks and the relevant responsible components for the bio-retted kenaf (*Hibiscus cannabinus L.)* bast fibres with different bio-retting techniques are displayed in Table 7. From the comparison results, the FTIR spectrum imply that any functional group of bio-retted kenaf (*Hibiscus cannabinus L.)* does not introduce or eliminate due to different bio-retting techniques applied.

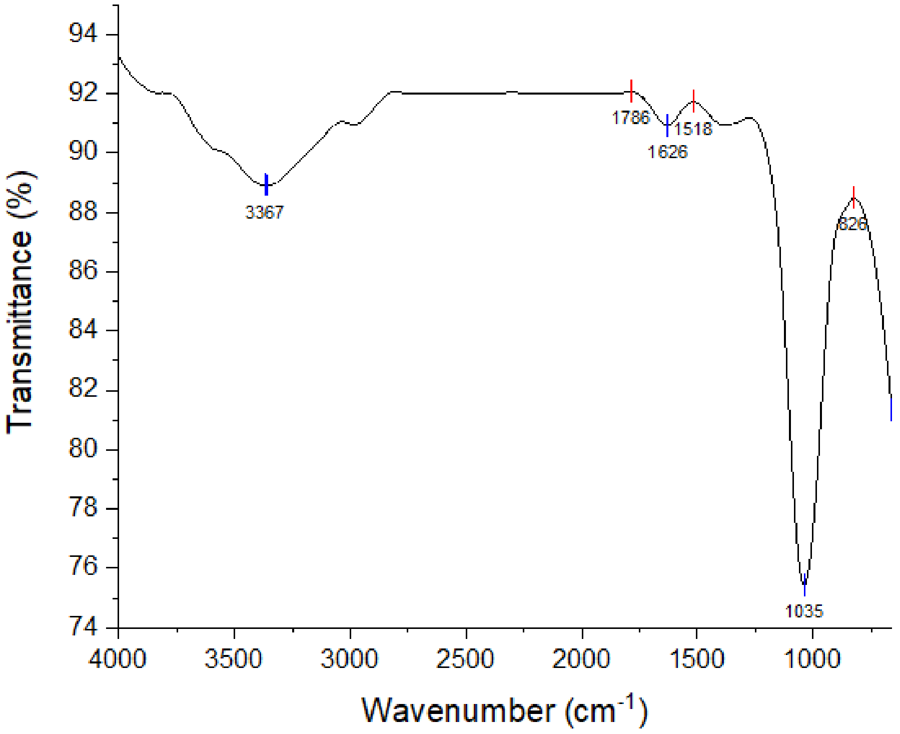

FIGURE 5. FTIR spectrum for bio-retted kenaf (*Hibiscus cannabinus L.)* bast fibre

TABLE 7. FTIR spectrum of bio-retted kenaf (*Hibiscus cannabinus L.)* bast fibre and other bio-retted kenaf bast fibres

Bio-retting technique	FTIR analysis result					Reference
	Peaks noted at the significant wave numbers (cm^{-1})					
	O-H	C-H	C=O	C-O	C-OH	
Freshwater and enzymes (3d)	3367	2792	1626	1035	826	Current study
Water and bacteria (10d)	*	*	*	1058	*	(Song & Obendorf, 2006)
Seawater (11d)	3308.17	2915	1638	1010	828	(Hossain et al. 2022)
Freshwater (11d)						
Municipal wastewater (11d)						
Fresh pond water (6d)	3308	2915	1638	1010	829	(Hossain et al. 2021)
Fresh pond water and Bacillus sp. KRB56(6d)						
Fresh pond water and Bacillus sp. KRB22 (6d)						

d denotes days
* denotes no reported result

X-RAY DIFFRACTION (XRD) ANALYSIS

The XRD spectra of the bio-retted kenaf (*Hibiscus cannabinus L.)* bast fibre is presented in Figure 6. The XRD spectra of bio-retted kenaf (*Hibiscus cannabinus L.)* bast fibre revealed two peaks at 2θ = 15.32° and 22.28°. The presence of the amorphous fraction in bio-retted kenaf (*Hibiscus cannabinus L.)* bast fibre was revealed by the first peak at 2θ =15.32°. The bio-retted kenaf *(Hibiscus cannabinus L.)* bast fibre demonstrated crystalline components at 2θ = 22.28°, where the second peak was observed. The excellent mechanical properties and molecular structure of the fibre were shown by its high crystallinity index value (Indran et al. 2014). A fibre's degree of crystallinity may be derived from its diffraction peak sharpness; a sharper peak indicates a greater degree of crystallinity in the fibre (Kamaruddin et al. 2021). By using the Segal empirical method, as shown in Equation 1 for the calculation, the crystallinity index value of the bio-retted kenaf (*Hibiscus cannabinus L.)* bast was 68.62%. While, by using Scherrer's formula as shown in Equation 2 for the calculation, the crystalline size was 19.65 nm. The crystalline index of bio-retted kenaf (*Hibiscus cannabinus L.)* bast fibre was higher than water-retted kenaf (*Hibiscus cannabinus L.)* bast fibre (49.91%) (Hassan et al. 2018). The crystalline index of bio-retted kenaf (*Hibiscus cannabinus L.)* bast fibres with different bio-retting techniques were compared and presented in Table 8. The higher crystalline index is attributed to the cellulose of the fibres. All bio-retting techniques were significantly showed higher crystalline index due to sufficient removal of non-cellulosic gums (NCGs). In general, the natural cellulosic fibres with higher crystallinity index considered premium quality fibres (Hossain et al. 2022).

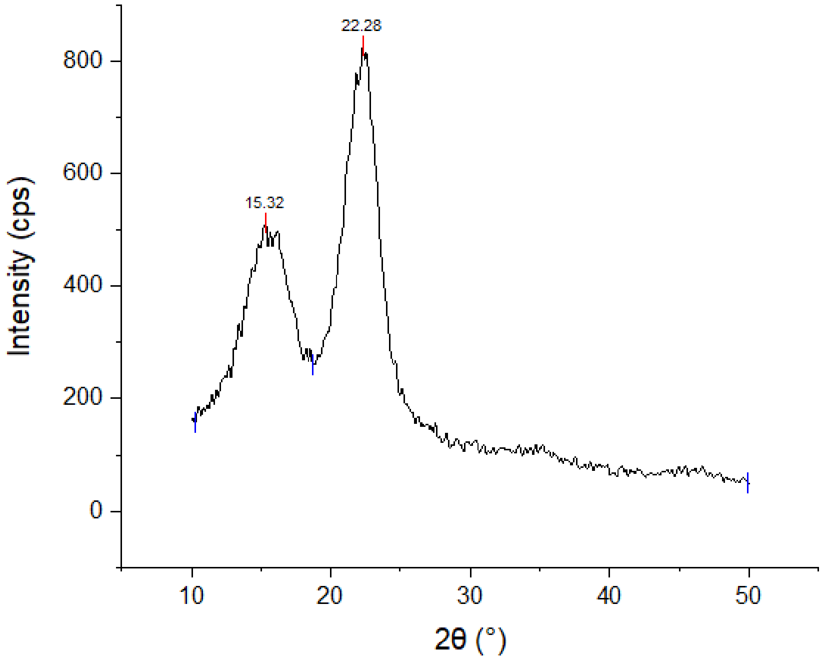

FIGURE 6. X-ray diffraction pattern of bio-retted kenaf (*Hibiscus cannabinus L.)* bast fibre

TABLE 8. Crystallinity index of bio-retted kenaf (*Hibiscus cannabinus L.)* bast fibre and other bio-retted kenaf bast fibres

Bio-retting technique	Crystallinity index (%)	Reference
Freshwater and enzymes (3d)	68.62	Current study
Seawater (11d)	64.78	(Hossain et al. 2022)
Freshwater (11d)	62.80	
Municipal wastewater (11d)	58.39	
Fresh pond water (6d)	57.80	(Hossain et al. 2021)
Fresh pond water and Bacillus sp. KRB56(6d)	69.52	
Fresh pond water and Bacillus sp. KRB22 (6d)	66.67	

CONCLUSION

The main objective of this study was to determine the characterization of the bio-retted kenaf (*Hibiscus cannabinus L.)* bast fibre. The density of bio-retted kenaf (*Hibiscus cannabinus L.)* bast fibre was determined 1.269 ± 0.26 g/cm^3, which confirms its potential as an acceptable reinforcement in lightweight composite applications. This study produced FTIR analysis which corroborate the findings of a great deal of the previous work, that have similar chemical groups (O-H), (C-H), (C=O), (C-O) and (C-OH). The tensile test indicated that the fibre had a high tensile strength (3876 ± 1122 MPa)

due to the presence of an adequate percentage of cellulose (58.72%), crystallinity index (68.62%) and crystalline size (19.65 nm) and suitable to be used in making high-strength composite structure. The thermogravimetric analysis result validated the bio-retted kenaf (*Hibiscus cannabinus L.)* bast fibre thermal stability (240°C) in spite of the high temperature generated during manufacturing process, which was expected in the range of 200°C to 350°C, and suitable to be used in high-temperature applications.

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DECLARATION OF COMPETING INTEREST

None.

REFERENCES

Abbas, A. G. N., Aziz, F. N. A. A., Abdan, K., Nasir, N. A. M., & Norizan, M. N. 2022. Kenaf fibre reinforced cementitious composites. *Fibers* 10(1): 1–24. https://doi.org/10.3390/fib10010003

Akil, H. M., Omar, M. F., Mazuki, A. A. M., Safiee, S., Ishak, Z. A. M., & Abu Bakar, A. 2011. Kenaf fiber reinforced composites: A review. *Materials and Design* 32(8–9): 4107–4121. https://doi.org/10.1016/j.matdes.2011.04.008

Arjmandi, R., Yıldırım, I., Hatton, F., Hassan, A., Jefferies, C., Mohamad, Z., & Othman, N. 2021. Kenaf fibers reinforced unsaturated polyester composites: A review. *Journal of Engineered Fibers and Fabrics* 16. https://doi.org/10.1177/15589250211040184

Ashori, A., Harun, J., Raverty, W., & Yusoff, M. N. M. 2006. Chemical and morphological characteristics of Malaysian cultivated kenaf (Hibiscus cannabinus) fiber. *Polymer - Plastics Technology and Engineering* 45(1): 131–134. https://doi.org/10.1080/03602550500373782

Bezazi, A., Belaadi, A., Bourchak, M., Scarpa, F., & Boba, K. 2014. Novel extraction techniques, chemical and mechanical characterisation of Agave americana L. natural fibres. *Composites Part B: Engineering* 66: 194–203. https://doi.org/10.1016/j.compositesb.2014.05.014

Chaishome, J., & Rattanapaskorn, S. 2017. The influence of alkaline treatment on thermal stability of flax fibres. *IOP Conference Series: Materials Science and Engineering* 191(1). https://doi.org/10.1088/1757-899X/191/1/012007

Chung, T. J., Park, J. W., Lee, H. J., Kwon, H. J., Kim, H. J., Lee, Y. K., & Yin Tze, W. T. 2018. The improvement of mechanical properties, thermal stability, and water absorption resistance of an eco-friendly PLA/kenaf biocomposite using acetylation. *Applied Sciences (Switzerland):* 8(3). https://doi.org/10.3390/app8030376

Duval, A., Bourmaud, A., Augier, L., & Baley, C. 2011. Influence of the sampling area of the stem on the mechanical properties of hemp fibers. *Materials Letters* 65(4): 797–800. https://doi.org/10.1016/j.matlet.2010.11.053

Ezekiel, N., Ndazi, B., Nyahumwa, C., & Karlsson, S. 2011. Effect of temperature and durations of heating on coir fibers. *Industrial Crops and Products* 33(3): 638–643. https://doi.org/10.1016/j.indcrop.2010.12.030

Hanana, S., Elloumi, A., Placet, V., Tounsi, H., Belghith, H., & Bradai, C. 2015. An efficient enzymatic-based process for the extraction of high-mechanical properties alfa fibres. *Industrial Crops and Products* 70: 190–200. https://doi.org/10.1016/j.indcrop.2015.03.018

Hassan, A., Isa, M. R. M., Ishak, Z. A. M., Ishak, N. A., Rahman, N. A., & Salleh, F. M. 2018. Characterization of sodium hydroxide-treated kenaf fibres for biodegradable composite application. *High Performance Polymers* 30(8): 890–899. https://doi.org/10.1177/0954008318784997

Hossain, M. M., Siddiquee, S., & Kumar, V. 2022. Water sources derived bio retting effect on kenaf fiber compositions. *Journal of Natural Fibers* 19(14): 9396–9409. https://doi.org/10.1080/15440478.2021.1982829

Hossain, M., Siddiquee, S., & Kumar, V. 2021. Isolation of alkalophilic pectinolytic bacteria and their bio retting effect on kenaf fiber compositions. *Alinteri Journal of Agriculture Sciences* 36(2): 156–165. https://doi.org/10.47059/alinteri/v36i2/ajas21129

Indran, S., Raj, R. E., & Sreenivasan, V. S. 2014. Characterization of new natural cellulosic fiber from Cissus quadrangularis root. *Carbohydrate Polymers* 110: 423–429. https://doi.org/10.1016/j.carbpol.2014.04.051

Ismail, N. F., Radzuan, N. A. M., Sulong, A. B., Muhamad, N., & Haron, C. H. C. 2021. The effect of alkali treatment on physical, mechanical and thermal properties of kenaf fiber and polymer epoxy composites. *Polymers* 13(12). https://doi.org/10.3390/polym13122005

Jawaid, M., Chee, S. S., Asim, M., Saba, N., & Kalia, S. 2022. Sustainable kenaf/bamboo fibers/clay hybrid nanocomposites: properties, environmental aspects and applications. *Journal of Cleaner Production* 330(November 2021): 129938. https://doi.org/10.1016/j.jclepro.2021.129938

Jebadurai, S. G., Raj, R. E., Sreenivasan, V. S., & Binoj, J. S. 2019. Comprehensive characterization of natural cellulosic fiber from Coccinia grandis stem. In *Carbohydrate Polymers* (Vol. 207). Elsevier Ltd. https://doi.org/10.1016/j.carbpol.2018.12.027

Kamaruddin, Z. H., Jumaidin, R., Rushdan, A. I., Selamat, M. Z., & Alamjuri, R. H. 2021. Characterization of natural cellulosic fiber isolated from Malaysian cymbopogan citratus leaves. *BioResources* 16(4): 7729–7750. https://doi.org/10.15376/biores.16.4.7729-7750

Kamaruddin, Z. H., Jumaidin, R., Selamat, M. Z., & Ilyas, R. A. 2022. Characteristics and properties of lemongrass (cymbopogan citratus): A comprehensive review. *Journal of Natural Fibers* 19(14): 8101–8118. https://doi.org/10.1080/15440478.2021.1958439

Kathirselvam, M., Kumaravel, A., Arthanarieswaran, V. P., & Saravanakumar, S. S. 2019. Isolation and characterization of cellulose fibers from Thespesia populnea barks: A study on physicochemical and structural properties. *International Journal of Biological Macromolecules* 129: 396–406. https://doi.org/10.1016/j.ijbiomac.2019.02.044

Kulandaivel, N., Muralikannan, R., & S, K. 2020. Extraction and characterization of novel natural cellulosic fibers from pigeon pea plant. *Journal of Natural Fibers* 17(5): 769–779. https://doi.org/10.1080/15440478.2018.1534184

Kumaar, A. S., Senthilkumar, A., Sornakumar, T., Saravanakumar, S. S., & Arthanariesewaran, V. P. 2019. Physicochemical properties of new cellulosic fiber extracted from Carica papaya bark. *Journal of Natural Fibers* 16(2): 175–184. https://doi.org/10.1080/15440478.2017.1410514

Manimaran, P., Pillai, G. P., Vignesh, V., & Prithiviraj, M. 2020. Characterization of natural cellulosic fibers from Nendran Banana Peduncle plants. In *International Journal of Biological Macromolecules* (Vol. 162). Elsevier B.V. https://doi.org/10.1016/j.ijbiomac.2020.08.111

Moshi, A. A. M., Ravindran, D., Bharathi, S. R. S., Padma, S. R., Indran, S., & Divya, D. 2020. Characterization of natural cellulosic fiber extracted from Grewia damine flowering plant's stem. *International Journal of Biological Macromolecules* 164: 1246–1255. https://doi.org/10.1016/j.ijbiomac.2020.07.225

Nurazzi, N. M., Asyraf, M. R. M., Rayung, M., Norrrahim, M. N. F., Shazleen, S. S., Rani, M. S. A., Shafi, A. R., Aisyah, H. A., Radzi, M. H. M., Sabaruddin, F. A., Ilyas, R. A., Zainudin, E. S., & Abdan, K. 2021. Thermogravimetric analysis properties of cellulosic natural fiber polymer composites: A review on influence of chemical treatments. *Polymers* 13(16). https://doi.org/10.3390/polym13162710

Perumal, C. I., & Sarala, R. 2020. Characterization of a new natural cellulosic fiber extracted from Derris scandens stem. *International Journal of Biological Macromolecules* 165: 2303–2313. https://doi.org/10.1016/j.ijbiomac.2020.10.086

Reddy, N., & Yang, Y. 2005. Biofibers from agricultural byproducts for industrial applications. *Trends in Biotechnology* 23(1): 22–27. https://doi.org/10.1016/j.tibtech.2004.11.002

Rozyanty, A. R., Zhafer, S. F., Shayfull, Z., Nainggolan, I., Musa, L., & Zheing, L. T. 2021. Effect of water and mechanical retting process on mechanical and physical properties of kenaf bast fiber reinforced unsaturated polyester composites. *Composite Structures* 257: 113384. https://doi.org/10.1016/j.compstruct.2020.113384

Selver, E., Ucar, N., & Gulmez, T. 2018. Effect of stacking sequence on tensile, flexural and thermomechanical properties of hybrid flax/glass and jute/glass thermoset composites. *Journal of Industrial Textiles* 48(2): 494–520. https://doi.org/10.1177/1528083717736102

Senthamaraikannan, P., & Kathiresan, M. 2018. Characterization of raw and alkali treated new natural cellulosic fiber from Coccinia grandis.L. *Carbohydrate Polymers* 186: 332–343. https://doi.org/10.1016/j.carbpol.2018.01.072

Song, K. H., & Obendorf, S. K. 2006. Chemical and biological retting of kenaf fibers. *Textile Research Journal* 76(10): 751–756. https://doi.org/10.1177/0040517506070520

Tholibon, D., Tharazi, I., Sulong, A. B., Muhamad, N., Ismail, N. F., Md Radzi, M. K. F., Mohd Radzuan, N. A., & Hui, D. 2019. Kenaf fiber composites: a review on synthetic and biodegradable polymer matrix. *Jurnal Kejuruteraan* 31(1): 65–76. https://doi.org/10.17576/jkukm-2019-31(1)-08

Vijay, R., Manoharan, S., Arjun, S., Vinod, A., & Singaravelu, D. L. 2021. Characterization of silane-treated and untreated natural fibers from stem of leucas aspera. *Journal of Natural Fibers* 18(12): 1957–1973. https://doi.org/10.1080/15440478.2019.1710651

Yang, H., Yan, R., Chen, H., Lee, D. H., & Zheng, C. 2007. Characteristics of hemicellulose, cellulose and lignin pyrolysis. *Fuel* 86(12–13): 1781–1788. https://doi.org/10.1016/j.fuel.2006.12.013

Yao, F., Wu, Q., Lei, Y., Guo, W., & Xu, Y. 2008. Thermal decomposition kinetics of natural fibers: Activation energy with dynamic thermogravimetric analysis. *Polymer Degradation and Stability* 93(1): 90–98. https://doi.org/10.1016/j.polymdegradstab.2007.10.012