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Unprecedented extraction and characterisation of *Yucca elephantipes* silver star plant fibre–An exploratory investigation

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A vegetable cellulosic fibre extracted from the leaves of the Yucca Silver Plant (YSP) through water retting process is presented in this paper. The study explores the diverse applications of the extracted fibre, conducting a thorough characterisation encompassing physical, mechanical, morphological, and thermal properties. Archimedes' principle-based density test of YSP fibres revealed its average density to be 1.23173 g/cc. Its average tensile strength, tensile modulus and strain rate were determined according to ASTM D3379-75 to be 454.68MPa, 18.744GPa and 2.216%, respectively. The surface and cross-section morphological analysis of YSP fibre revealed an uneven texture and a polygonal and multicellular honeycomb structure. The presence of free functional groups for Cellulose, Hemicellulose and Lignin has been identified by FTIR analysis. Exposure of powdered YSP fibre to XRD revealed acrystallinity index of 54.106% and a crystallite size of 4.70 nm. TG and DTA investigations of YSP fibre disclosed that these fibres are thermally stable up to 226ºC and the Kinetic activation energy to be 76 KJ/mol. Examining the findings suggests that the YSP plant possesses significant potential as a natural fibre resource. The fibres derived from this plant exhibit promise as reinforcement material in polymer matrix-based composites.

Keywords: FTIR analysis, Thermal stability, XRD analysis, *Yucca elephantipes* silver star, Yucca Silver plant

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Introduction

Synthetic fibres are widely embraced for reinforcing continuous mediums, serving as substitutes for various monolithic materials. When dispersed within a polymeric matrix, these fibres find diverse applications across structural engineering, space vehicles, automotive industries, furniture, sports equipment, domestic appliances, medical devices, and more. Their appeal lies in delivering a commendable strength-to-weight ratio and exceptional mechanical properties. Prominent examples of these commercial synthetic fibres include carbon, glass, and Kevlar. Despite their widespread use, it is essential to note that these fibres are derived from petroleum, rendering them inherently toxic. Apart from contributing to the depletion of fossil fuels, their utilisation poses a constant environmental threat. Furthermore, the recycling processes associated with these man-made fibres are not environmentally favorable[1].

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For centuries, natural fibres like cotton, jute, mesta, and flax were used mainly for textile purposes, but last couple of decades, many researchers worked in the direction of replacing synthetic fibres with natural fibres in diversified applications[2]. The reported results also encourage employing them for a few applications as they offer attractive benefits like renewable resources, abundance, biodegradability, selective superior properties, etc[3]. However, the increased consumption of natural fibres with increased population is alarming the shortage. Due to this, recent research towards discovering and analysing new natural fibre resources has increased drastically. In the last twenty years, a variety of plants have been investigated to find new sources of natural fibres as follows: Alfa fibre[4], Indian areca fruit husk Fibres[5], *Furcreaefoetida mediopicta* fibre[6], *Sansevieria cylindrical* fibre[7], *Prosopis juliflora* bark fibre[8] etc. This indicates the emphasis on investigating novel natural fibres.

The genus *Yucca* encompasses approximately 40 species of succulent plants, all of which possess fibrous

leaves[9]. This present research aims to study the potential of new Yucca Silver Plant (YSP) leaf fibres to discover their diversified applications, which has never been done before. This evergreen garden plant that withstands drought is native to hot and arid regions of the Americas and the Caribbean. This plant is also called yucca elephantipes, spineless yucca, yucca cane, dragon yucca, or banana yucca. It grows to about 30 cm in height with sword-like rosettes[10,11]. However, if these plants are harvested, unlike garden plants, then branches will grow like exact elephantipes plants, which can become a potential source of natural fibre. Currently, The YSP is valued for its ornamental appeal, with its tall flower stalks producing edible blooms high in calcium and potassium, suitable for use in salads, while its leaves contain significant amounts of ascorbic acid[12]. The leaves of the plant are found to have fibres and are currently being wasted of no use[9]. In a study conducted by Bell and King[13], it was observed that the distribution of fibrous bundles within Yucca leaves followed a specific pattern. These bundles were found to be organised into groups consisting of xylem and phloem tissues, with fibres serving as protective caps both above and below the bundles. In a study conducted by McLaughlin and Schunk[14], fresh Yucca leaves were exposed to a 5% potassium hydroxide treatment at a temperature range of 60-65°C for a duration of 40-48 hours. The researchers aimed to investigate the effects of this treatment on the characteristics of the Yucca leaf fibres, specifically focusing on fibre length, diameter, and cell wall thickness. The researchers successfully extracted fibres from the sample, which exhibited a diameter ranging from 13 to 18 mm and a cell wall thickness ranging from 4.5 to 6.2 mm. In their study, Azanaw *et al.*[15] employed two methods, namely water retting and chemical extraction, to extract fibres from Yucca Elephantine leaves. Water retting involved immersing the leaves in river water at room temperature for a duration of 26 days. On the other hand, chemical extraction was carried out using a sodium hydroxide (NaOH) solution with concentrations ranging from 3 to 20%. This process involved boiling the leaves for a period of 2 hours. Whenever a new natural fibre resource is discovered, it is essential to study the properties of all new natural fibres, as they vary widely depending on their chemical composition, density, ageing, defects, micro-fibril angle, cell dimensions, etc., which helps to identify its suitable engineering applications[5]. In this report, the YSP was presented, and the fibre extraction procedure was provided. Further, the physical, thermal, and mechanical properties of the YSP fibre were determined for the first time and compared to existing natural fibres.

Materials and Methods

Plant sample collection and identification

In July 2020, specimens of *Yucca elephantipes* "silver star," belonging to the Agavoideae family[10,11], were gathered from Bapatla in the Eastern Ghats of Andhra Pradesh, South India. Following collection, these plants were transplanted to a home garden in Hyderabad, Telangana, India. The Agriculture Department at Acharya N. G. Ranga Agricultural University (ANGRAU), Hyderabad, Telangana, India, employed a straightforward destructive technique to examine the leaves of these plants for the presence of fibres. Subsequently, these plants were utilised to extract lignocellulosic fibres.

Retting of YSP leaf

The YSP fibre extraction process utilised a straightforward water retting technique, as outlined by Madival *et al.*[16]. Fresh leaves from the plant were cut at the root and submerged in tap water, fully covering them from root to tail, for a duration of four weeks. Throughout this period, water changes were frequent, and the YSP leaves underwent a gentle beating to facilitate the removal of any impurities from the fibre strands. Following this, the resulting fibres were soaked in water for an additional two days, thoroughly rinsed, sun-dried for one week, and subsequently stored in a clean poly bag for future use.

Density of fibre

A calibrated Liquid Digital Density Meter (LDDM) was used to find the density of raw YSP fibre. In order to find the density using LDDM, YSP fibrewas cut into small pieces of varying lengths from 3–5 mm, weighed and introduced into the glass jar carrying working liquid (WL). Acetone, Xylene, and Toluene were the WLs, which were separately used to find the density of raw YSP fibre. The following formula has been used to determine the density:

$$\text{Density of YSP fibre} = \frac{\text{Mass of fibre in air}}{\text{Mass of fibre in air} - \text{Mass of fibre in WL}} \text{X density of WL}$$

Thermal characterisation of fibre

To investigate the thermal stability of YSP fibre, a comprehensive analysis was conducted using a single TG/DTA experiment. The Seiko TG/DTA 6200

apparatus, featuring sensitivities of 0.2 µg and 0.06 µV, was employed for this purpose. The experiment was executed in a Nitrogen gas atmosphere, and the temperature range spanned from 25 to 1500°C. Both the capacity and pan were constructed from Aluminum, serving as a secure holder for the weighed specimens of YSP fibre. The experiment incorporated a temperature ramp of 20°C/min, reaching a maximum temperature of 620°C, with a power supply of 15 kV. This setup facilitated a detailed exploration of mass changes in controlled atmospheric conditions, mass variations concerning temperature, and a comprehensive analysis of YSP fibre's thermal characteristics.

Scanned Electron Microscopy(SEM)

For a detailed examination of the cross-section and surface characteristics of YSP fibre, Scanning Electron Microscopy (SEM) was employed. Samples obtained from tensile tests, with fractured surfaces sputter-coated with gold, were meticulously analysed using the SEM equipment from ZEISS, Germany.

Crystallographic structure of fibre

YSP fibre's crystalline structure has been revealed using X-ray diffracted (XRD) analysis with PANalytical X-Pert Proequipment. Diffracted Spectrums have been recorded between 0 to 50°, Cu-Kα radiation (15kV) and a power supply of 20 mA. The percentage of crystallinity of the powdered YSP fibre has been determined using the following formulae[17]:

$$\% \text{ of crystallinity} = \frac{(I_{002} - I_{amr})}{I_{002}} \text{ X } 100$$

where I_{002} was maximum diffracted intensity of plane-002 and I_{amr} was the intensity of amorphous (2θ=18.5°). In addition to that, the following Scherrer's equation has been used for finding the crystallite size of YSP fibre:

$$L = \frac{K\lambda}{\beta\cos\theta}$$

where K was the dimensionless factor 0.89, β was the Full Width Half Maximum of the peak, λ was the wavelength of the radiation (m), and θ was the Bragg's angle in degree.

Fourier Transform Infrared Spectroscopy (FTIR)

FTIR analysis for identifying chemical components of YSP fibre was carried out at Kelvn Labs, Hyderabad. The powdered YSP fibre underwent testing using an FTIR spectrometer, specifically the Vertex 70 v with the Attenuated Total Reflectance (ATR) technique. The analysis comprised 64 acquisitions, employing a scanning speed with a spectrum range of 4000 to 650, a resolution of 16 cm^{-1}, and a triangular apodisation.

Tensile properties of fibre

The tensile properties of YSP fibre, including tensile strength, percentage of elongation, Young's modulus, and specific strength, were determined in accordance with the ASTM D3379-75 standard. Each YSP fibre specimen comprised ten fibre strands with a gauge length mounted on a piece of rigid cardboard measuring 55 mm. The filament ends were securely affixed to the cardboard using epoxy adhesive and subjected to testing at a crosshead speed of 0.2 mm per minute. Multiple tests were conducted to ensure robust confirmation of the results. For accurate property determination, the diameter of the extracted fibre was measured using SEM and Image J software. These measurements played a crucial role in comprehensively analysing YSP fibre properties.

Results and Discussion

Extracted fibres

The YSP plant and the fibres extracted from its leaves are shown in Fig. 1. The extraction technique influences the time for separating fibre strands from non-fibre tissues of leaf[18]. In this research, each leaf weighing approximately 5.3 mg was kept in a plastic tub filled with tap water. During the retting process, decaying of the leaf's surface layer was observed. This can be attributed to the enzymatic action reported by Lee *et al.*[19], which exposed fibre bundle structure in leaf within 15 days. Further, water has been replaced with fresh water on a regular basis to avoid unpleasant smell of retting. Most of the green-juicy content has been washed away while replacing water for 21 days and leftover green content has also been tried to remove by carefully beating, rinsing and retting several times for two more days. This entire retting process took 23 days to separate fibre strands, completely which are still having gum content adhered to them. This span is more than the reported retting spans of 14 days of *Prosopis juliphera*[8] and 5 days of Furcreofoeteda mediopicta[16] fibres. This indicates the delaying action of microbial degradation of natural gums in the case of YSP, which requires further intensive chemical analysis of its leaves. In addition, microbial degradation of pectineus matter

Fig. 1 — a) Yucca silver plant; and b) Extracted leaf fibre.

due to alone water retting is incomplete and may require additional mixed treatment techniques to improve the same. Research carried out by S. Banik *et al.*[20] agrees with this statement about using mixed treatment to remove non-tissue fibre content effectively and reduce the retting period.

After sun-drying the extracted fibre, the leaf fibre was weighed. The ratio of fibre weight to leaf weight indicated that YSP fibres account for approximately 4% of the total weight of the leaf. This discloses that the YSP fibres to leaf weight ratio is more than Furcreofoeteda mediopicta[8] and almost equal to Sisal[21]. Further, it has been noticed that each leaf consists of 80-120 Ivory coloured fibres of length 25-40 cm. For textile applications, this length is sufficient for the spinning process, according to Totong *et al.*[22]. Based on the measurement of staple fibre length, the YSP fibre can be classified as belonging to the category of 'extra-long staple fibre'. In manufacturing high-quality yarn, the preference lies with longer raw fibres as opposed to shorter ones. The observed phenomenon can be attributed to the inherent characteristics of longer fibres, which possess larger surface areas. This increased surface area enhances friction and reduces slipperiness between adjacent fibres during the spinning process. Consequently, the yarn produced exhibits greater strength, as evidenced by a higher tensile resistance[22]. According to Murthy[23], it has been observed that within each set of fibres, the utilisation of long staple yarns leads to the production of fabrics that possess enhanced strength, smoothness, and functionality. These fabrics are generally considered higher quality than those produced from short-staple yarns, albeit at a higher cost. According to recommendations, it is advised that the minimum length of the fibre should be no less than 5 mm[23].

Table 1 — Densities of working liquids used in LDDM and YSP fibre

Working liquid	Density of the liquid in g/cc	Average Density of YSP fibre in g/cc
Toluene	0.866	1.22041
Acetone	0.784	1.23956
Xylene	0.875	1.23523

Density of YSP fibre

Table 1 presents the densities of various working liquids alongside the density of Yucca Silver Plant (YSP) fibre. The average values are displayed from five samples for each working liquid. Remarkably, there is only a minor 2-3% variation in density values across the different working liquids, indicating consistent density results for YSP fibre. The computed average density of YSP fibre is recorded as 1.23173 g/cc. Upon comparison with commercial synthetic fibres, it is evident that akin to many natural fibres, the density of YSP fibre is lower than that of synthetic fibres. This lower-density characteristic positions YSP fibre favourably for applications requiring high specific strength in lightweight structural components.

Microstructural characteristics of fibre

Harvesting approach, fibre maturity stage, the type of soil, extraction process etc, are the factors which could result in disparities in fibre surface[5]. By keeping this in view, the sample is prepared with extreme care. SEM pictures of the surface and cross-section of the YSP fibre is firmly sputter coated with gold are

shown in Fig. 2. Few geometrical properties of YSP fibres can be noticed through microstructural investigation, including a diameter ranging from 6.700 to 38.998 µm, lumen diameter varying between 1.010 and 2.246 µm, and a cross-sectional area spanning from 37.390 to 1188.411 µm². It is evident from the longitudinal view (Fig. 2a, b and e) of the YSP fibre that its exterior is fully composed of both rough and smooth surfaces with many pores and impurities and few flaky regions. All these characteristics of the fibre play an important role in enhancing wettability and promoting bonding by mechanical interlocking with polymer matrix in composites application[24]. Further, Fig. 2c and e disclosed deep perforated cell structures known as trichomes on the fibre surface, which will also favour superior mechanical bonding than smooth-surfaced synthetic fibres, which come into play in stress transformation from matrix to fibres[5]. The water storage and passage pipes of fibre strands called lumens were spotted in Fig. 2d and e. In addition, Fig. 2c displayed a fair circular cross-section and a pentalobal type honeycomb structure of YSP fibre, which allows for partial filling of the lumen hallow spaces (Fig. 2d) with polymeric resin during the manufacture of composite material and enhances its mechanical performance[24]. It is also evident from Fig. 2f that the tail end of the fibre is also of circular cross-

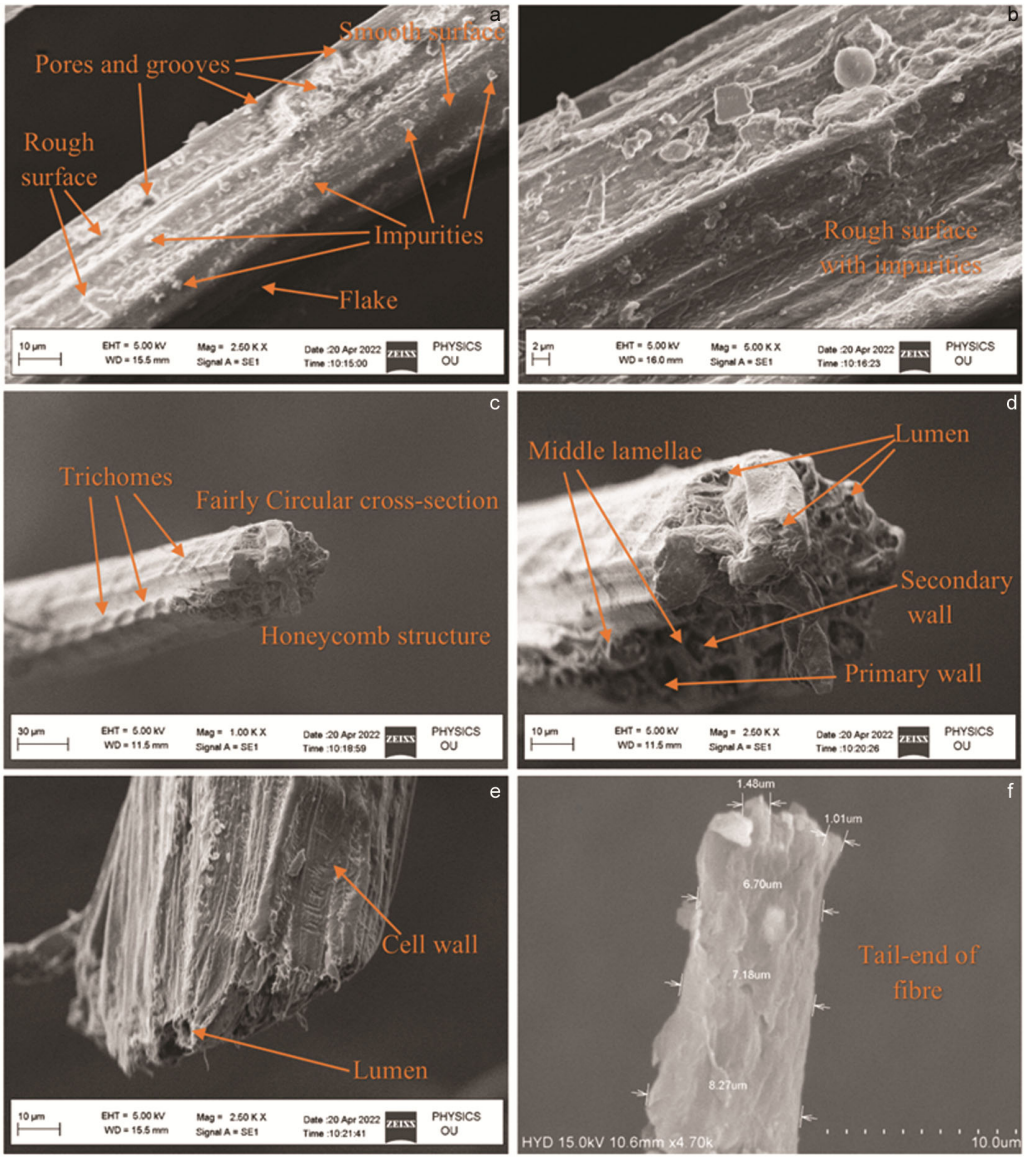

Fig. 2 — SEM images of Yucca silver plant fibre, a) Longitudinal view; b) Impurities on the Surface; c) Sectional view; d) Lumens, lamellae and walls of the fibre; e) Cell wall and uneven surface; and f) Tail end of the fibre.

section with a diameter of 6.70 µm. Like all other natural fibres, YSP fibre itself is a composite where cellulose microfibrils are embedded into soft and continuous primary and secondary walls primarily made of hemicellulose and lignin, which essentially provides fibre filament hardness[5,21]. This has been confirmed by observing Fig. 2d, which shows the Primary wall, secondary wall and middle lamellae. Compared to many natural fibres[8], the short diameter of the YSP fibre can be noticed, indicating it is finer. Root to tail end diameter ratio has been found to be 5.82 µm for this fibre.

FTIR analysis

FTIR has been used to examine the functional groups and chemical compounds of the extracted YSP fibre surface. The obtained spectrum is presented in Fig. 3. Similar to all natural fibres, cellulose presence has been identified in YSP fibre. The absorption band between the peaks of wave numbers 3295 to 3587 cm^{-1} can be observed from Fig. 3, which is mainly due to O-H (alcohols), i.e., hydrogen-bonded stretching of the vibration of cellulose[25]. Absorption bands specific to the peak at 3399 cm^{-1} of hydroxyl stretching band[4], 1031.19 and 1244 $cm^{-1}$of broad -C-OH stretching vibration presence due to polysaccharides components and –C-O stretching of alcohol groups[5,8,13,19], 745.5 cm^{-1} peak[7] and finally 1312 cm^{-1} peak[26] are the characteristics of cellulose, certainly. Absorption peaks found at 1244.93 cm^{-1} of C-O acetyl group stretching vibration[7] and 1543.11 cm^{-1} peak[5,26] is the conformation of the presence of Lignin. The little shoulder at 894.56 cm^{-1} in the spectrum corresponds to the monosaccharides' b-glycosidic linkages[27]. Furthermore, 1243 cm^{-1} and 1654.9 cm^{-1} absorption peaks can be attributed to Hemicellulose[5,10,13,27].

XRD analysis

A typical X-ray spectrum for YSP fibre is shown in Fig. 4. Like many raw-natural fibres, this fibre's spectrum also consists of two anticipated main peaks. These diffraction crests indicate that the YSP fibre is

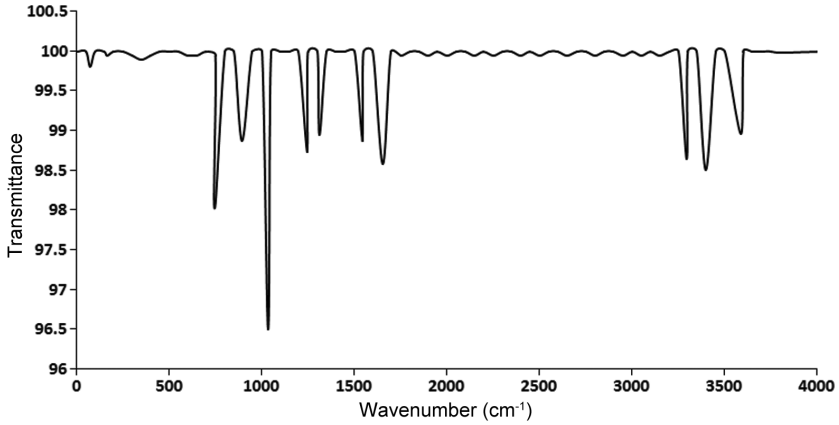

Fig. 3 — FTIR spectrum of Yucca silver plant fibre.

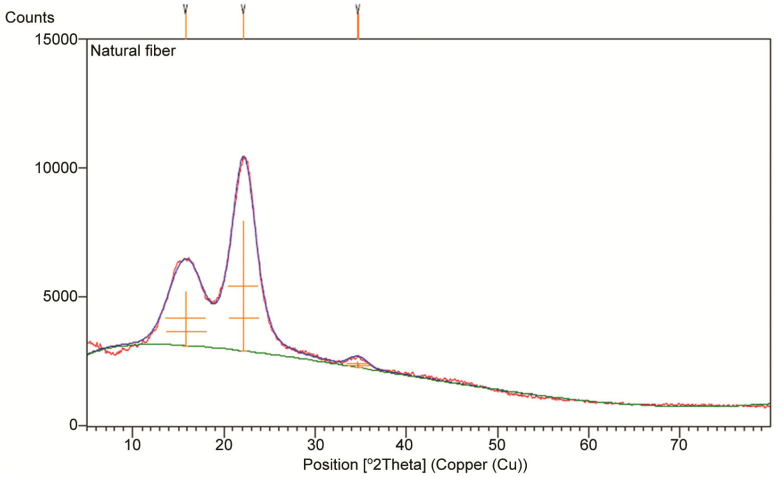

Fig. 4 — XRD spectrum of Yucca silver plant fibre.

semi-crystalline[14]. The percentage of crystallinity Index of YSP fibre has been calculated according to the same method followed by d'Almeida *et al.*[28] and Helbert *et al.*[29] from the XRD band, was found to be 54.106%. This value is lower than most of the common natural fibres, including cotton (60%), jute (71%), sisal (71%), and hemp (88%), ramie (58%) and higher than recently reported natural fibres *Wrightia tinctoria* seed (49.2%), oil palm fruit fibre (34.1%), *P. juliflora* (46%)[5,9,27,29]. This indicates that YSP fibre's crystallites are better ordered than *W. tinctoria* seed, oil palm fruit and *P. juliflora* fibres. Further, the two peaks formed at $2\theta=15.6^0$ and $2\theta=22.5^0$ can be attributed to cellulose I and IV, which displayed a monoclinic structure[28-30]. A small peak at $2\theta=34.5^0$ can also be attributed to cellulose I type[31]. The crystallite size determined using the Scherrer's[5] equation was found to be 4.70 nm for this YSP fibre, which is greater than flax (2.8 nm) and corn stalk (3.8 nm) fibres[14]. This large crystallite size of YSP fibre results in decreased chemical and water absorption capacities when embedded in polymeric matrices[18].

Tensile behaviour of fibre

The corrected stress vs strain curve of the YSP fibre is depicted in Fig. 5a. This exhibits a brittle behaviour with a two phases deformation curve similar to RC fibre[5], where the first phase corresponds to linear variation followed by the second phase of non-linear deformation due to the sudden drop of the load. Similar behaviour is also reported in the case of okra and *Sansevieria cylindrica*[14]. The occurrence of non-linear deformations after the linear phase can be linked to the breakage of weak primary cell walls and fibrecell delamination[32]. In addition to this, the tensile strength of 454.68 MPa with a 2.216% strain rate can be observed from Fig. 5a. These moderate strength values can be attributed to the moderate microfibril angle value of 11.54–17.14º determined using the global deformation equation[8,16]. The results pattern,i.e. tensile strength of 648+13.4 MPa and microfibril angle 9.46–12.8ºof *Albizia amara*[16], and tensile strength 558+13.4 MPa and microfibril angle 10.640+0.45º of *P. juliflora* fibre[8], are strongly supporting the result of the present work. However, a few fibres, including jute (8.1º), flax (5º), hemp (6.2 º) and banana (11º-12º) results are at slight variance with respect to their strength and are worth investigating for promising results[33,34]. According to the report of Baley C[35], the fibres under the traction loads cause the stress transfer from soft binding material to multiple microfibrils of cellulose, which eventually tends to reorient the load-bearing microfibrils, causing stretching along the fibre axis. Examination of the YSP fibre tensile test sample (Fig. 5b) revealed similarly extended microfibrils and their failure to resist the external axis loads in the case of the present YSP fibre. The active participation of microfibrils validates this tensile strength and supports the statement: longitudinal tensile strength and stiffness have both been demonstrated to be significantly impacted by Microfibril angle. As it rises, tensile strength and stiffness rapidly diminish[36]. The specific strength and specific modulus of YSP fibre were calculated to be 0.35659 MPa/(kg/m^3) and 14.722 MPa/(kg m^{-3}), respectively. These values indicate the YSP fibre fits within the usual range of natural fibres, and the properties are inferior to synthetic fibres[8]. Analysis of tensile properties confirms that YSP fibre is a potential candidate as reinforcement for polymeric composite materials and products.

Thermal resistance and stability of fibre

It is essential to investigate the thermal stability of all novel natural fibre as it can become one of the

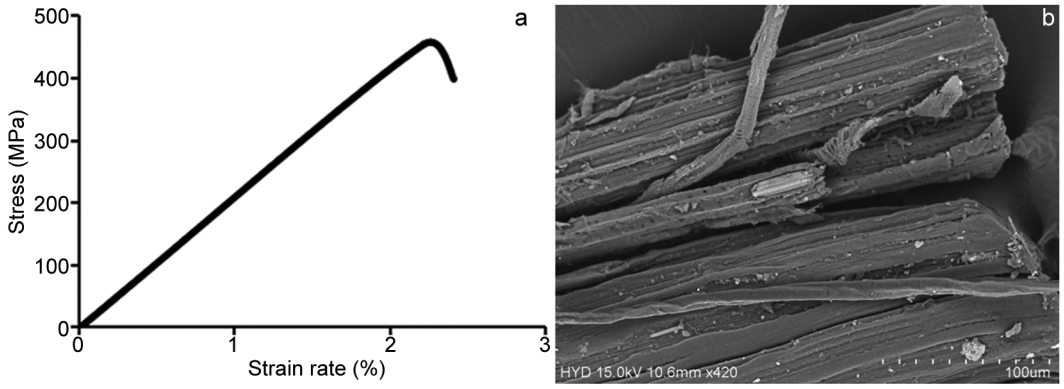

Fig. 5 — a) Corrected stress vs strain curve for Yucca silver plant fibre; and b) surface of the tensile test filament showing microfibrils.

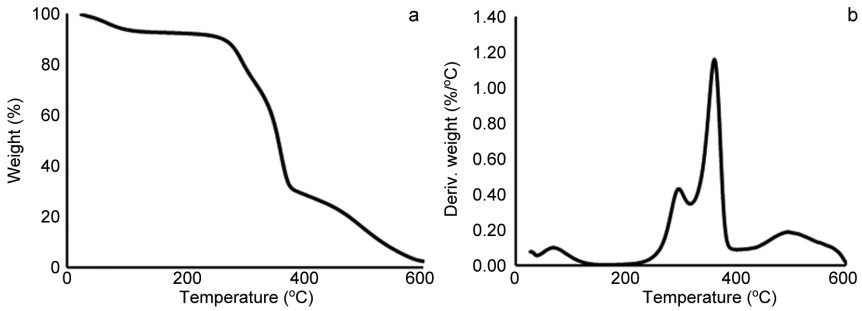

Fig. 6 — TG/DTA graphs for Yucca silver plant fibre.

limiting factors for reinforcement application of bio-composites. Therefore, degradation behaviour with respect to temperature variation of powdered YSP fibre was studied using the curves of TG and DTA, as depicted in Fig. 6. This TG curve is similar to the TG curves reported in the case of many natural fibres[5,8,25,37], which showed three weight loss segments with two decomposition stages. The initial minor loss of weight (≈7%) recorded between 25 and 105ºC is attributed to moisture content evaporation of YSP fibre. Afterwards, there is no appreciable weight loss (only ≈1%) is witnessed until 226ºC, which indicates the promising thermal stability of YSP fibre up to that temperature. An increase in temperature above this value caused the start of de-polymerization and first-stage degradation of the chemical compounds, i.e. hemicellulose and cellulose and continued with a weight reduction of 67% up to 434ºC[5,38]. The third weight loss segment is observed between 434 to 608ºC, occurring with 23% mass reduction by complex structured lignin decomposition[39]. The kinetic activation energy is also calculated to be 76KJ/mol, which is helpful in finding the kinetic parameter. This value ensures that YSP fibre has promising thermal stability to resist the de-polymerization against the thermal loads during the manufacture of polymer composites[5].

Conclusion

In conclusion, the study on YSP fibres reveals their promising potential as a natural resource for reinforcement in polymeric composites. Despite an extended retting period of 4 weeks, deviating from conventional fibres, YSP fibres exhibit enhanced strength and rigidity, validated through FTIR analysis of hemicellulose, lignin, and cellulose. Morphological scrutiny uncovers features conducive to mechanical interlocking with polymer matrices, suggesting heightened composite performance. With a density lower than synthetic fibers and favourable diameter characteristics, YSP fibres demonstrate potential advantages for high specific strength in lightweight structural applications. The larger crystallite size influences reduced chemical and water absorption capacities in polymeric matrices, while tensile properties affirm their viability for composite reinforcement. Additionally, YSP fibres exhibit remarkable thermal stability, with minimal weight loss up to 226ºC, favourable for polymer processing in composite manufacturing. Despite certain limitations compared to synthetic fibers, the comprehensive characterizations in this study establishes a foundational understanding of YSP fibers, paving the way for further exploration and application in diverse fields.

Conflict of interest

The authors declare that there is no conflict of interest regarding the publication of this manuscript. They have no financial or personal relationships that could influence the work or its interpretation. This includes any affiliations, funding sources, or financial interests that may have influenced the outcomes or presentation of the research. The content of this paper is solely the responsibility of the authors.

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