Comparative analysis of natural fibres characteristics as composite reinforcement

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ABSTRACT – REZUMAT

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Due to environmental concerns, natural fibre development is essential, and its utilization has recently attracted more attention. The use of jute, hemp, linen, sisal, and banana fibres in textile production is widespread around the world. Additionally, these fibres are widely accessible in many countries, including Pakistan, India, China, Turkey, and the United States. The objective of this study is to compare the physio-mechanical characteristics of the aforementioned natural fibres. All of these fibres were obtained locally. Scanning electron microscopy was used to examine the surface morphology of these natural fibres, and the results revealed that banana and sisal fibres are hollow in comparison to other fibres. A single fibre tensile testing apparatus was used to evaluate the mechanical characteristics. Banana and sisal fibres demonstrated the highest breaking strength and elongation, respectively. Fourier transform infrared spectroscopy was used to investigate the functional groups of these natural fibres. Differential scanning calorimetry and thermogravimetric analysis were used to investigate their thermal behaviour. Energy Dispersive X-Ray Analysis and Raman analysis were also carried out to ascertain the chemical composition.

Keywords: *natural fibres, mechanical properties, SEM, FTIR, TGA, DSC, EDX, Raman analysis*

Analiza comparativă a caracteristicilor fibrelor naturale ca armătură pentru compozite

Datorită preocupărilor legate de mediu, dezvoltarea fibrelor naturale este esențială, iar utilizarea lor a atras recent şi mai multă atenție. Utilizarea fibrelor de iută, cânepă, in, sisal și banane în producția de textile este larg răspândită în întreaga lume. În plus, aceste fibre sunt accesibile pe scară largă în multe țări, inclusiv Pakistan, India, China, Turcia și Statele Unite. Obiectivul acestui studiu este de a compara caracteristicile fizio-mecanice ale fibrelor naturale menționate mai sus. Toate aceste fibre au fost obținute local. Microscopia electronică cu scanare a fost folosită pentru a examina morfologia suprafeței acestor fibre naturale, iar rezultatele au arătat că fibrele de banană și sisal prezintă lumen, în comparație cu alte fibre. Pentru a evalua caracteristicile mecanice a fost utilizat un singur aparat de testare a rezistenţei la rupere a fibrelor. Valorile cele mai mari ale rezistenţei şi alungirii la rupere au fost înregistrate pentru fibrele de banană și, respectiv, de sisal. Spectroscopia în infraroșu cu transformare Fourier a fost utilizată pentru a investiga grupele funcționale ale acestor fibre naturale. Scanarea calorimetrică diferențială și analiza termogravimetrică au fost utilizate pentru a investiga comportamentul lor termic. Spectroscopia de raze X cu dispersie de energie și analiza Raman au fost, de asemenea, efectuate pentru a stabili compoziția chimică.

Cuvinte-cheie: *fibre naturale, proprietăți mecanice, SEM, FTIR, TGA, DSC, EDX, analiză Raman*

INTRODUCTION

Due to environmental concerns [1–4], the development of natural fibres is essential, and the usage of natural fibres has recently drawn more attention [5, 6]. The utilization of natural plant fibres as reinforcing elements has drawn more attention in recent years. Currently, India produces about 14.5 million tons of natural fibres annually, compared to a global production of 45.5 million tons. These fibres were strengthened using a variety of matrixes, including polyester, epoxy, vinyl ester, phenol-formaldehyde, and others. It was utilized as a substitute for synthetic fibres. Natural fibres can be categorized into a wide range of categories, including bast, leaf, seed, fruit, and wood fibre. Conventional applications of these fibres included rope, roofing, home furnishings, bandages, and a variety of other things. Composites with natural fibre reinforcement have good mechanical and thermal characteristics. Natural fibres have replaced synthetic polymers for composite materials used in the automotive, packaging, and aerospace industries. Natural fibres are becoming more popular as their use is expanded into engineering end uses like construction materials. High specific strength and modulus, availability, low cost, lightweight, recyclability, biodegradability, lack of health risks, and non-abrasive nature are the key benefits of these natural fibre-reinforced composites [7–9].

Glass fibre in composite materials might be replaced with natural fibres. Natural fibres are a desirable option because of their inherent benefits, which include low density, biodegradability, and mechanical

qualities that are comparable to those of glass fibre composites [10, 11]. Stalks, stems, seeds, roots, leaves, and fruit husks are just a few examples of the natural fibres that have been taken from various plant parts and employed as reinforcement in polymer-based composites to create industrial components. The natural fibres that are frequently used include jute, flax, kenaf, hemp, ramie (extracted from bast), sisal, pineapple, palf (extracted from leaf), cotton, kapok (extracted from seed), coir (extracted from fruit), bamboo, elephant grass (extracted from stalk), etc.
These fibres have also evolved into the primary determining elements in the choice, creation, and design of the components [12–14]. Natural fibres are employed in a variety of ways, including short, continuous, and randomly oriented. Due to their exceptional integrity and conformability for cutting-edge structural applications, woven fabric mats have recently attracted the most attention and are thought to be the most appealing [15, 16]. Compared to reinforcements that are short and randomly oriented, some studies have shown how the functional qualities of composites using woven fabric reinforcement from natural fibres are improved [17, 18].
Some researchers have looked into the combined effects of using various natural fibres as reinforcement to create composites made of hybridized natural fibres [19, 20]. Hybrid composites made from banana and kenaf have been studied for their impact on mechanical characteristics. It was claimed that as compared to composites made of separate fibres, the hybridization of kenaf and banana fibres increased the mechanical strength. In line with this, a different study claims that sisal and oil palm were hybridized to enhance the composites' mechanical properties [21].
Natural fibres have a high cellulose content, are inexpensive, easily renewable, and have the potential to be reinforced with polymers. Natural fibres are becoming more popular since they are affordable, light, strong, and biodegradable. They are also environmentally friendly due to ease of recyclability [22, 23]. Additionally, natural fibre composites are said to have environmental benefits such as decreased reliance on non-renewable energy and material sources, lower pollutant and greenhouse gas emissions, improved energy recovery, and end-of-life component biodegradability [24]. The natural fibre is thought to be an environmentally friendly material used as reinforcement for creating biocomposites, suitable for many industrial applications because of its high hydroxyl content of cellulose, which makes it susceptible to water absorption and affects the mechanical properties of materials [25, 26]. The fundamental function of cellulose, which makes up a bigger portion of natural plant fibres, is to absorb moisture. Some spirally coiled cellulose microfibres are joined by an amorphous lignin matrix. However, it contributes to defending against biological assaults and giving strength [27].
Using natural fibre in reinforced composites has numerous technological and environmental advantages. Numerous natural fibres, such as jute, straw, Flax, hemp, wood, sugarcane, bamboo, grass, kenaf, sisal, coir, rice husks, wheat, barley, oats, kapok, bagasse, cotton, mulberry, banana fibre, raphia, pineapple leaf fibre, and papyrus, have been investigated for use in plastics. The matrix materials used to reinforce the fibres are categorized as thermosets, thermoplastics and elastomers [28].
In addition, numerous natural fibres, including kenaf, hemp, flax, jute, sisal, banana, coir, and pineapple leaf fibres, are gaining increased prominence as environmentally friendly reinforcement for composite materials [29, 30]. The building and automobile industries utilize sisal more than any other fibre. This could be due to its remarkable mechanical properties, which are why it is utilized to produce concrete buildings and automotive car parts [31]. Similar to other plant-based natural fibres, hemp is a lignocellulosic reinforcing component used in composites. It is employed in a variety of structural applications, and the European Industrial Hemp Association (EIHA) organizes an annual hemp conference to share information on the most recent advancements in the use of hemp fibres in the food, food supplements, textile, automotive, and pharmaceutical industries. Studies on sisal plants in Kenya, China, and India have shown the mechanical properties of sisal fibres. Sisal fibre has a tensile strength of 347 MPa in Kenya and China and 400–700 MPa in India. Compared to untreated fibres, treated fibre-reinforced composites typically have a higher tensile strength [32, 33].
Natural fibre composites have the potential to take the place of expensive glass fibre in applications requiring little load-bearing capacity. Plant fibres provide several well-known benefits, including low cost and reduced tool wear during processing [34]. When it comes to transportation, such as cars, trains, and aircraft, natural fibres have proven to be more durable than synthetic fibre. For ceiling panelling and partition boards, other industries that use them are construction, building, packing, consumer goods, and military [35]. The automobile industry greatly benefits from the low density of natural fibres. According to a study, replacing 30% of glass fibres with 65% of hemp fibres results in a net energy savings of 50,000 MJ, or 3 tons less emissions [36, 37]. Many studies have been conducted on natural fibres, including topics like extraction, morphology, mechanical properties, etc. The comparison of five distinct natural fibres, including jute, hemp, linen, sisal, and banana fibres, is carried out in this research project.
The relevant composites were subjected to Scanning electron microscopy (SEM), tensile testing, Fourier-transform infrared spectroscopy (FTIR), Thermogravimetric Analysis (TGA), Differential scanning calorimetry (DSC), Energy Dispersive X-Ray (EDX), and Raman analysis to describe their thermal and physio-mechanical characteristics.

MATERIALS AND METHODS

Materials

Five distinct kinds of natural fibres, including jute, hemp, linen, sisal, and banana fibres, are provided by Elvin Textile Bursa, Turkey. All fibres are applied in their unprocessed, raw state.

Methodology

Morphological observations and fibre characterizations have been carried out at Bursa Technology Coordination and R&D Center (BUTEKOM).

Scanning Electron Microscopy (SEM)

Surface morphology of the fibres analysed by SEM (HITACHI TM3030 PLUS, Japan).

Tensile properties

According to ISO 5079, the mechanical characteristics, such as tensile strength (maximum breaking force), maximum elongation, and maximum elongation percentage, were assessed using a SHIMADZU AGS-X tensile tester.

Fourier Transform Infrared Spectroscopy (FTIR) analysis

To identify the functional groups of the fibres, FTIR analysis was done. Utilizing a Shimadzu IR-Tracer100 device in ATR mode and accordance with ASTM E1252, infrared spectroscopy (FTIR) analysis was carried out.

Thermogravimetric analysis (TGA)

TGA is applied to measure sample mass loss caused by temperature. It is employed to describe the thermal stability of the sample and the rate of decomposition. TGA of fibres was carried out using a SDTQ600 TA Thermogravimetric analyser in accordance with ISO 11358-1. The temperature range of the TGA was 20 °C to 600 °C with a heating rate of 20 °C/min under a nitrogen atmosphere and then heated from 600 °C to 900 °C under an oxygen atmosphere.

Differential scanning calorimetry (DSC) analysis

DSC is a popular thermal analysis used to identify temperature-related changes in the polymer, such as melting, crystallization, degradation, and glass transition. Q 2000 TA Instruments differential scanning calorimeter was used for DSC measurements in accordance with ASTM E1952 (ISO 11357-3). Under a nitrogen atmosphere, samples were heated from 40 °C to 280 °C at a rate of 10 °C/min.

Raman analysis

With a laser excitation wavelength of 785 nm, 1 mW laser intensity, and a 50X objective, Raman spectra were captured by a Renishaw inVia Reflex Raman microscope. Each sample was positioned on a piece of glass. Molecular vibrations can be determined using Raman spectroscopy. There is a significant capacity for water absorption due to the hydrophilic surface of the lignocellulosic structure. Water-related absorption bands do not significantly affect Raman spectroscopy as they do in FTIR.

RESULTS AND DISCUSSION

Scanning Electron Microscopy (SEM)

The longitudinal and cross-sectional views of the natural fibres are given below from figure 1 to figure 5. These can be compared with each other cross-sectionally and longitudinally. Hemp and linen resemble solid rods, while banana, sisal, and jute are straight, cylindrical fibres. In contrast to hemp and linen fibres, SEM micrographs demonstrated that banana, sisal, and jute fibres are hollow. Hemp, jute, and linen fibres all have some notches on their surfaces, according to SEM results. The smooth surface of linen fibre provides it with more softness than other natural fibres, and this makes it popular in clothing fabrics, particularly for women. Unlike other fibres, which have a smoother surface, banana fibres feature grooves on their surface. Banana fibres are a more practical solution for better moisture management and air permeability due to the surface grooves. However, banana fibres cannot be used as a substitute for linen fibre in clothing fabrics because of their rough surface.

Tensile strength and elongation

The capacity of a material to withstand the tensile load before failing is explained by its tensile characteristics. To ascertain the breaking strength or tensile strength, maximum elongation, and elongation % of natural fibre, the tensile test was carried out using the tensile tester. Finding the right application for the particular fibre is made much easier by these characteristics. The results of tensile tests are presented in table 1.

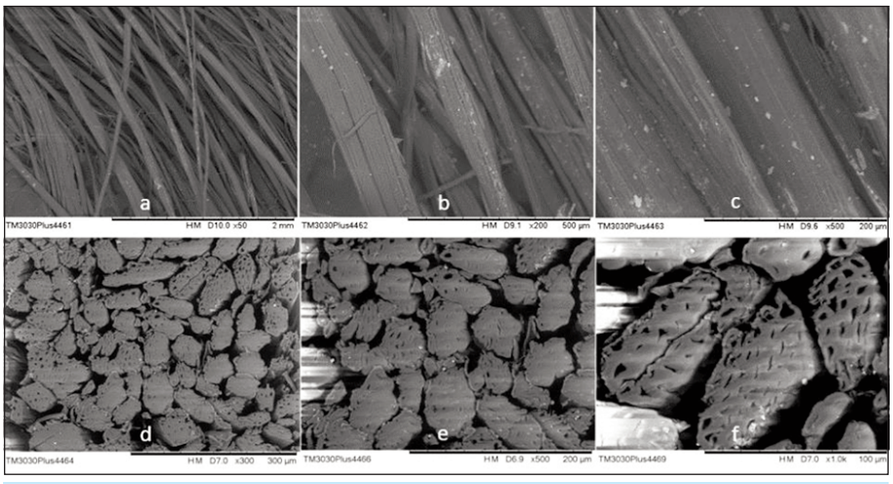

Fig. 1. SEM images for jute fibres: *a* – longitudinal view at x50; *b* – longitudinal view at x200; *c* – longitudinal view at x500; *d* – cross-sectional view at x300; *e* – cross-sectional view at x500; *f* – cross-sectional view at x1000

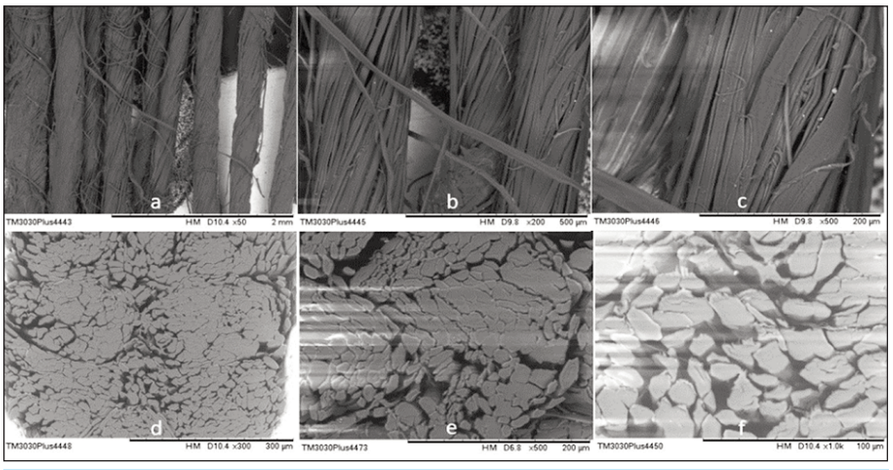

Fig. 2. SEM images for hemp fibres: *a* – longitudinal view at x50; *b* – longitudinal view at x200; *c* – longitudinal view at x500; *d* – cross-sectional view at x300; *e* – cross-sectional view at x500; *f* – cross-sectional view at x1000

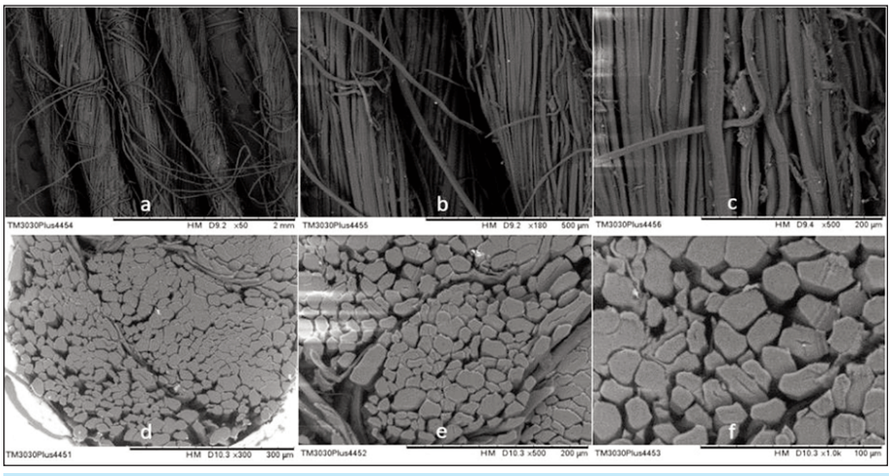

Fig. 3. SEM images for linen fibres: *a* – longitudinal view at x50; *b* – longitudinal view at x180; *c* – longitudinal view at x500; *d* – cross-sectional view at x300; *e* – cross-sectional view at x500; *f* – cross-sectional view at x1000

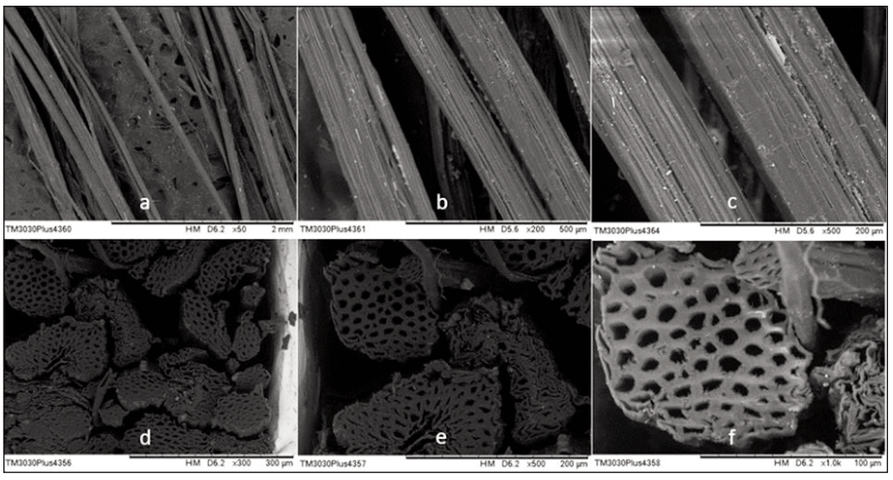

Fig. 4. SEM images for Sisal fibres: *a* – longitudinal view at x50; *b* – longitudinal view at x200; *c* – longitudinal view at x500; *d* – cross-sectional view at x300; *e* – cross-sectional view at x500; *f* – cross-sectional view at x1000

Figure 6 illustrates the tensile strength graphs. These graphs contain two separate regions: the first is an elastic zone, and the second is a plastic region. The area in which the curve deviates from its intended direction is addressed as the elastic region of the fibre. The permanent strain in the fibres can be seen when the curve descends and approaches the x-axis. This point displays the ultimate point of rupture of the fibre. Sisal fibre has the highest breaking strength, according to the graphs. Sisal fibre has a larger diameter or thickness than the other fibres, as demonstrated in the SEM examination, and as a result, it has a higher tensile strength.

Since hemp fibre has the smallest thickness and diameter among the fibres, it also has the worst tensile strength. Since hemp and linen fibres appear to have the same internal structure from a cross-sectional perspective and linen fibre has a slightly higher thickness than hemp fibre, it exhibits slightly better tensile strength.

FTIR measurements

Results from 20 scans with 8 cm^{-1} resolution between 600 cm^{-1} and 4000 cm^{-1} were acquired using FTIR. FTIR spectra of jute, hemp, linen, sisal, and banana fibres are presented in figure 7. The basic components of natural fibres are cellulose, hemicellulose, and lignin, which define their physical characteristics. Pectin and waxes are also present in some natural fibres, albeit in very small concentrations. Table 2 shows the chemical composition of jute, hemp, linen, sisal, and banana fibres. The vibrational bands of these elements are represented by the peaks in the FTIR spectra. The O-H stretching vibration is related to the broad peak at 3000–3600 cm^{-1}. The emergence of very strong and widespread absorption was a blatant sign that the fibre contained several hydroxyl groups. The asymmetric and symmetric C-H vibrations from the -CH_2

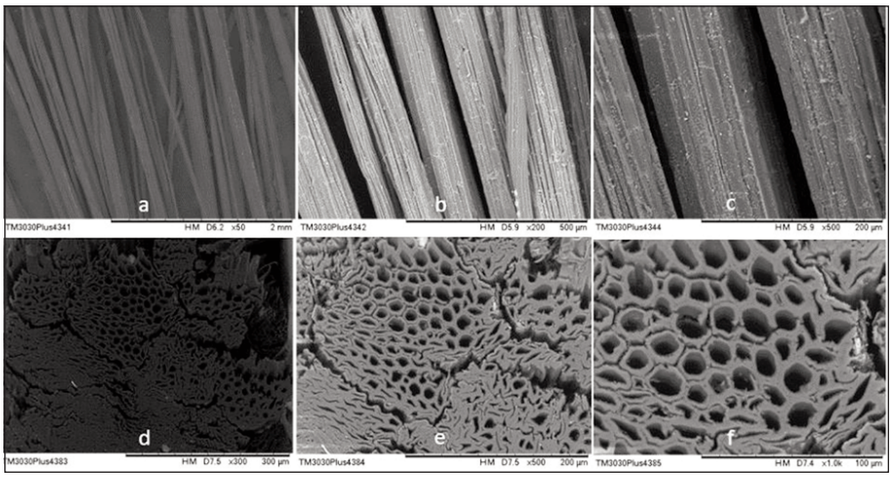

Fig. 5. SEM images for banana fibres: *a* – longitudinal view at x50; *b* – longitudinal view at x200; *c* – longitudinal view at x500; *d* – cross-sectional view at x300; *e* – cross-sectional view at x500; *f* – cross-sectional view at x1000

group of cellulose and hemicellulose, respectively, are represented by the peaks at about 2900 cm^{-1} and 2850 cm^{-1}. The carbonyl C=O bond of stretching vibration of carboxylic acid in lignin or ester group in hemicellulose causes the peak at 1639 cm^{-1} to appear [38]. -CH_2 symmetric bending in lignin, cellulose, and hemicellulose and -CH symmetric deformation of cellulose and hemicellulose are related to the peaks near 1427 cm^{-1} and 1369 cm^{-1}, respectively [39]. No new peaks were found in the FTIR spectra of the fibres. The heterogeneous structure of natural fibres is what causes the intensity variations in identical peaks that were found in different samples by FTIR and the presence of various peaks with low intensity.

Thermogravimetric analysis (TGA)

Figure 8 illustrates the fibres' TGA curves, which consist of four stages. The amount of mass that is retained as the temperature rises is shown in the graphs as the weight percentage (wt%). For jute, hemp, linen, sisal, and banana fibres, weight reduction in the first stage was 6.9%, 6%, 5.6%, 6.7%, and 6.4%, respectively. All natural fibres exhibit this tendency as a result of presented moisture. The FTIR study also demonstrates that the jute fibre absorbs the most water overall. The quantity of residual mass in the TGA curve abruptly drops between 148 and 416 °C. Depending on the

Table 1

TENSILE PROPERTIES OF NATURAL FIBRES

No.	Fibre	Max force (cN)	Max elongation (%)
1	Jute	114.88 ± 0.62	3.13 ± 0.37
2	Hemp	14.25 ± 0.47	10.71 ± 0.93
3	Linen	23.03 ± 0.30	16.06 ± 0.75
4	Sisal	818.08 ± 0.42	6.2 ± 0.29
5	Banana	371.44 ± 0.55	3.50 ± 0.37

Table 2

COMPARISON OF THE CHEMICAL STRUCTURE OF SOME NATURAL FIBRES

No.	Portion/ Fibre	Hemp	Sisal	Linen	Jute	Banana
1	Cellulose	70–75	78	81	61–75	60–65
2	Hemi-cellulose	16	10	14	13–20	6–9
3	Pectin	18	-	4	-	3–5
4	Lignin	4	8	3	5–13	5–10
5	Wax	-	2	-	-	-

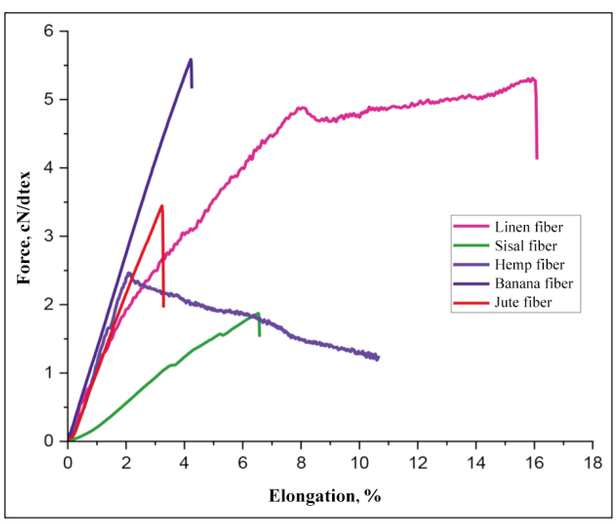

Fig. 6. Single fibre strength, the force-elongation curve of jute, hemp, linen, sisal, and banana fibres

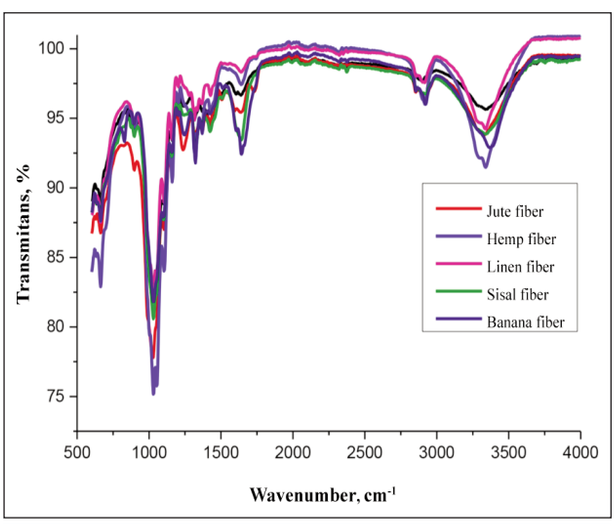

Fig. 7. FTIR spectra of jute, hemp, linen, sisal, and banana fibres in the range of 600 and 4000 cm^{-1}

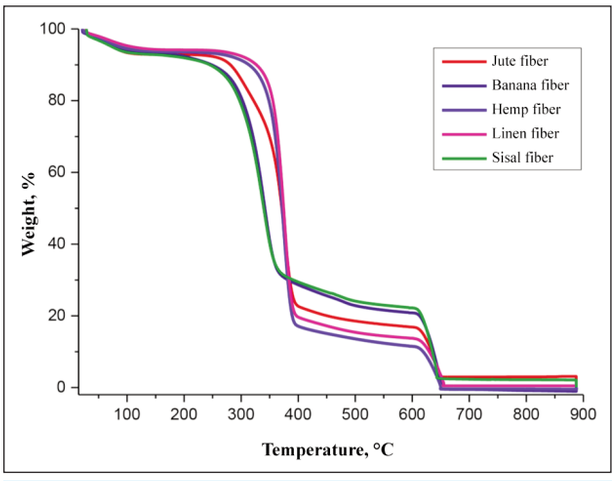

Fig. 8. TGA curves of jute, hemp, linen, sisal, and banana fibres

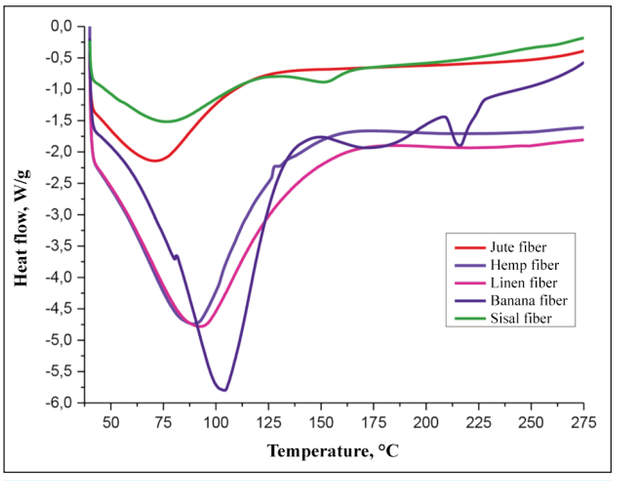

Fig. 9. DSC curves of jute, hemp, linen, sisal, and banana fibres

type of fibre, the second stage, which involves the majority of the weight loss, begins at 148 °C to 234 °C and ends at 395 °C to 416 °C. The temperature range where the rate of weight loss reaches its maximum value is thought to be when hemicellulose and cellulose start to break down [40]. The main weight loss, in this case, occurs between 148 °C and 416 °C. Compared to other fibres, linen fibre contains the most cellulose. The linen fibre has the highest value when the thermal decomposition temperatures are taken into account. Due to the breakdown of cellulose and lignin, a slight weight loss is seen in the third stage. Further raising the temperature does not affect the quantity of residual mass once the weight loss reaches about 75–80%. Their differing cellulose contents are the cause of this disparity. Coal oxidizes and breaks down into gaseous products with smaller molecular weights in the final step of the process.

Differential Scanning Calorimetry (DSC) analysis

The DSC method measures the enthalpy change brought on by variations in the sample's chemical and physical properties concerning temperature. According to the DSC curves shown in figure 9, the evaporation of water from the fibre structure results in a large endothermic peak that emerges between 30 °C and 135 °C. There is a large endothermic peak because water molecules have such a wide range of binding energies to the cellulose backbone.

According to TGA analysis, this peak corresponds to a weight reduction of 5.6% to 6.9%. The deepest peak, which indicates the existence of a highly moistened part of lignin, was seen in banana fibre.

Energy Dispersive X-Ray Analysis (EDX)

EDX is a technique that reveals an element's composition by analysing its different constituents. In figure 10, the ionization energy is displayed along the x-axis of the EDX spectrum, while the element counts are displayed along the y-axis. A larger presence in particular places indicates higher counts of that element. Carbon and oxygen were found in all of the natural fibres according to the EDX results in table 3, while additional elements were found in varying amounts in each fibre. The results revealed that all five natural fibres include calcium, iron, carbon, and oxygen.

Raman analysis

The Raman spectra of jute, hemp, linen, sisal, and banana fibres are displayed in figure 11. According to Eichhorn et al., the major peak at 1096 cm^{-1} is indicative of the C-O stretching mode of cellulose, but the peak at 380 cm^{-1} is only associated with crystalline cellulose I and is not present in amorphous cellulose [41]. Only the strongest bands in the spectrum

Table 3

EDX RESULTS

Elements	Jute		Hemp		Linen		Sisal		Banana	
	wt%	σ	wt%	σ	wt%	σ	wt%	σ	wt%	σ
C	54.8	0.2	52.4	0.2	49.6	0.2	54.2	0.8	46.8	1.3
O	43.7	0.2	47.1	0.2	49.9	0.2	44.4	0.8	46.2	1.3
Fe	0.6	0.1	0.2	0	0.4	0.1	0.8	0.2	-	-
Ca	0.3	0	0.1	0	0.2	0	0.4	0.1	-	-
Si	0.3	0	-	-	-	-	-	-	-	-
Al	0.2	0	-	-	-	-	-	-	-	-
K	0.1	0	-	-	-	-	0.3	0.1	6.1	0.4
S	0.1	0	-	-	-	-	-	-	-	-
Cl	-	-	0.1	0	-	-	-	-	-	-
Mg	-	-	-	-	-	-	-	-	0.9	0.2
Na	-	-	-	-	-	-	-	-	-	-

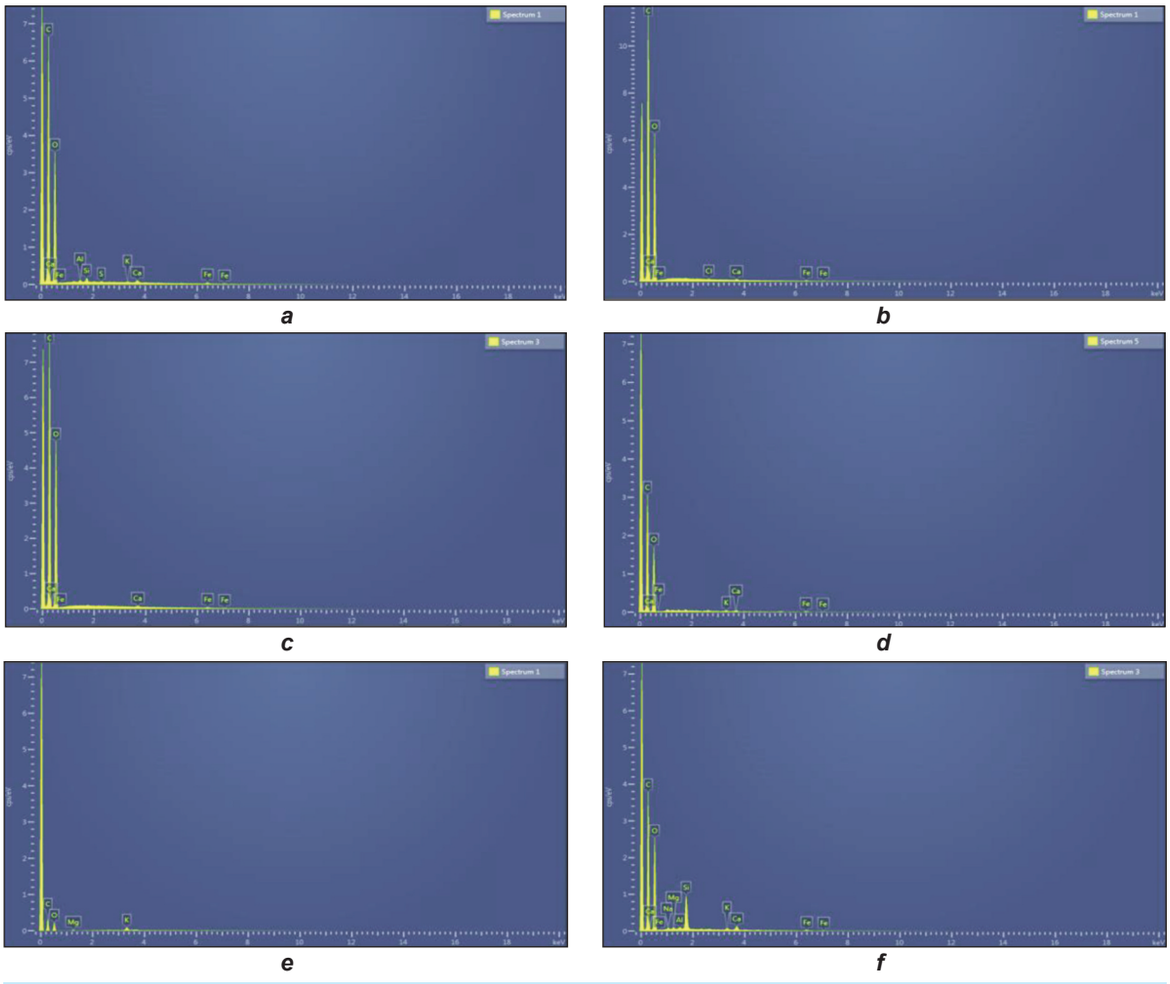

Fig. 10. EDX analysis: *a* – jute fibre; *b* – hemp fibre; *c* – linen fibre; *d* – sisal fibre; *e* – banana fibre

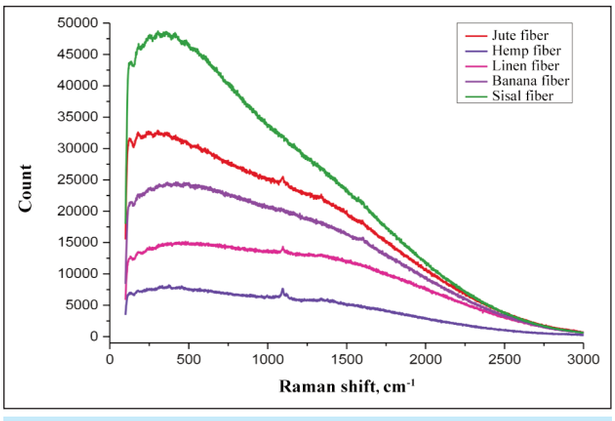

Fig. 11. Raman spectrum of jute, hemp, linen, sisal, and banana fibres

may be observed in Raman spectra because of the luminous background.

CONCLUSION

The objective of this study was to compare the physio-mechanical characteristics of the selected natural fibres. The natural fibres including jute, hemp, linen, sisal, and banana fibres were obtained from Elvin Textile Bursa. SEM, tensile testing, FTIR, Thermogravimetric analyzer, Differential calorimetry spectroscopy, Energy dispersive X-ray analysis, and Raman analysis were used to investigate the thermal and physio-mechanical properties of the fibres. The longitudinal and cross-sectional morphology of the natural fibres was revealed by the SEM. The SEM images demonstrated that whereas some fibres have filled cores, sisal and banana fibres had hollow internal structures. The highest breaking strength was demonstrated by sisal fibre (818.08 ± 0.42 cN), the lowest by hemp fibre (14.25 ± 0.47 cN), and the highest by linen fibre in terms of elongation before rupture. There are four stages in the TGA curves of fibres. The amount of mass that is retained as the temperature rises is indicated by the wt% in the graphs. In the case of jute, hemp, linen, sisal, and banana fibres, weight reduction in the first stage was 6.9%, 6%, 5.6%, 6.7%, and 6.4% respectively. All of the fibres are thermally stable below 148 °C, according to the graphs. The DSC curves, which showed the very least heat change after 150 °C, are likewise related to the TGA data. All of the natural fibres under consideration have carbon, oxygen, iron, and calcium present in their chemical structures, according to EDX and Raman analyses. According to the Raman spectra of jute, hemp, linen, sisal, and banana fibres [41], the main peak at 1096 cm^{-1} is indicative of the C-O stretching mode of cellulose, while the peak at

380 cm^{-1} is only associated with crystalline cellulose I and is not present in amorphous cellulose. Only the strongest bands in the Raman spectrum are visible due to the luminous background.

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