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Improvement of Mechanical, Thermal, and Physical Behaviors of Jute/Cotton Biocomposites Reinforced by Spent Tea Leaf Particles

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Abstract: Natural fibers such as jute, cotton, and bamboo composites are becoming alternative materials to synthetic fiber composites, as their use raises awareness of environmental protection. Among natural fibers, jute and cotton fibers were used in this research to fabricate six-layered composites reinforced by spent tea leaves. Varying amounts (0, 5, 10, and 15 g) of spent tea leaf powder were incorporated as reinforcement with resin to improve and observe properties and determine usability. The prepared composites were investigated comparatively in terms of mechanical, microstructural, morphological, and thermal properties. As regards mechanical characterization, tensile, compression, and bending properties were tested in this research to compare the obtained data with the data available in the literature to show its practical application. The results indicated that significant improvements in mechanical properties were obtained from the composites up to a certain proportion of reinforcement. The addition of 10 g reinforcement of spent tea leaves improved tensile strength by 33.46% and compressive strength by 38.86%. In terms of microstructural, morphological, and thermal characterization, in-depth SEM, EDS, XRD, UV, FTIR, TGA, and DSC analyses were performed. The results revealed that advanced microstructural, morphological, and thermal properties were improved with a certain proportion of spent tea leaf reinforcement.

Keywords: biocomposite; jute fiber; cotton fiber; spent tea leaves; resin

1. Introduction

Two or more dissimilar elements embedded together in a continuous phase and comprised physically or chemically produce composite materials [1]. The growth in the use of composite materials is seen in different industrial sectors because of their superior properties. Due to increased environmental consciousness, awareness, and the need for sustainable development, interest in natural fibers as reinforcements, used as alternatives to synthetic fibers in preparing composite materials, has increased [2,3]. In recent years, composites reinforced by biodegradable and sustainable natural fibers have gained significant attention because of low carbon emission, less fossil fuel consumption, lower cost and

density, and ease of fabrication [4,5]. Superior characteristics such as strength, stiffness, and toughness at lower density could be achieved from these fibers [6]. Therefore, natural fibers could serve as alternatives to synthetic fibers such as glass or carbon for the fabrication of composites in numerous industries such as construction, household, and automotive. Flax, banana, jute, ramie, hemp, and kenaf are the most commonly used fibers for the manufacture of composites. Among them, Jute fibers and cotton fibers are the most popular fibers, commonly produced in Central Asia and South East Asia, and are cheaper than flax [7].

Jute is an eco-friendly, non-synthetic, biodegradable, renewable, non-abrasive natural fiber extracted from plants [8]. It is an inexpensive natural fiber that shows good thermal conductivity and strength and has low density. The applications of jute fibers are widely seen in different engineering sectors including the structural and automotive industries, which make jute an attractive subject for researchers to study the strengthening of composites in terms of their mechanical and thermal properties with the use of different fiber orientations. The nature of the plant, the locality where it is grown, the used extraction methods, age, and the cultivation environment determine the mechanical properties of jute fibers [9]. Some important mechanical properties of jute and cotton fibers are presented in Table 1.

Table 1. Mechanical properties of jute and cotton fiber.

Tensile Strength (MPa)	Young's Modulus (GPa)	Elongation at Break (%)	Reference
Jute fiber			
393–773	26.5	1.5–1.8	[10]
400–800	10–30	1.5–1.8	[11]
393–773	13–26.5	1.16–1.5	[12]
393–773	19.0–26.5	1.16–1.8	[13]
Cotton fiber			
287–597	5.5–12.6	9.7	[14,15]

Cotton is a soft, fluffy, and staple fiber of cotton plants that grows in a boll or protective case around seeds. Cotton fiber is almost pure cellulose and contains some other percentage of fats, waxes, pectin, and water [16].

Spent tea leaves are low-cost green materials that can be used as reinforcement to develop composite materials and are dried and processed from *Camellia sinensis* [17]. Cellulose and hemicelluloses, lignin, condensed tannins, and structural proteins constitute the insoluble cell wall of tea leaves. Tea leaves have good potential in developing the properties of composite materials, as these constituents possess various functional groups.

In this research paper, we introduce jute and cotton fiber as reinforcement with spent tea leaf powder to manufacture a hybrid composite. We performed necessary experiments with proper analyses to prove the usability of the composite as a replacement for natural fibers.

2. Materials and Methods

2.1. Materials

2.1.1. Spent Tea Leaves

Tea leaves were collected from a local market, washed thoroughly three times with distilled water, and dried. Dried leaves were crushed and used for specified degrees of reinforcement to manufacture the composites shown in Figure 1a.

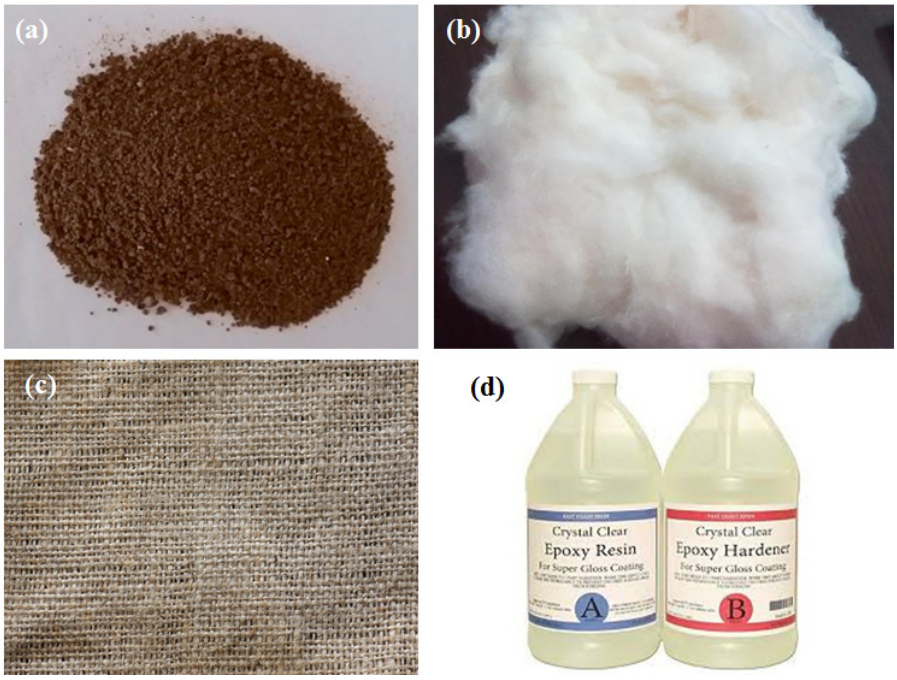


Figure 1. (**a**) Spent tea leaves, (**b**) cotton, (**c**) jute, and (**d**) epoxy resin and hardener.

2.1.2. Jute Fiber

Jute fiber was collected from a local market, washed properly with acetone to remove wax and other impurities, and cut into pieces to develop the composite. Jute is a natural, eco-friendly, and recyclable, and is the second-most biodegradable fiber, which belongs to the *Tiliaceae* family [18]. Currently, research interest in jute fiber composites is in high demand [19,20]. Jute fiber is, in general, used in the textile manufacturing industry for its low cost. Jute is composed of cellulose, lignin, and hemicelluloses [21,22]. Lignin contains many aromatic rings, which serve as mechanical support to the jute fiber. White jute and Tossa jute are the most extensively grown jute fibers. Table 2 shows the chemical composition of jute fiber.

Table 2. Chemical composition of jute fiber [23].

Elements	Content (%)
Cellulose	64.4
Hemicellulose	12
Lignin	11.8
Pectin	0.2
Water soluble	1.1
Water	10
Wax	0.5

2.1.3. Cotton Fiber

For this research, cotton fiber was collected from a local market and washed properly with acetone so that impurities such as wax and dirt can be removed. Cotton fiber is a versatile textile fiber from the *Malvaceae* family consisting mainly of cellulose. Cellulose has a long-chain molecular structure made by hydrogen bonds, which give higher tensile strength and dimensional stability to its fiber-forming spiral shape. Compared with synthetic fibers, cotton fiber has a low density but higher impact resistance [24]. Moreover, cotton fiber is durable, biodegradable, and has higher strength and absorbency [25].

2.1.4. Epoxy Resin

The cured end product of epoxy resin, as well as the basic components of epoxy resin, is known as epoxy. It is also a colloquial name for the functional group epoxide. Epoxy resin contains an epoxide group and a class of reactive prepolymers. Today, epoxy is used in numerous applications and sold in local hardware stores to be used as binders. Industries are developing different varieties of epoxies in response to the increasing number of applications. The epoxy resin used in this research was collected from Magic Corporation (Pvt) Ltd. (Dhaka, Bangladesh).

2.1.5. Epoxy Hardener

Certain types of mixtures were used to make hardeners to increase the resilience of the applied mixture. In the mixing process, the hardener can act either as a reactant or catalyst. A chemical reaction is initiated by mixing resin and hardener together, and the liquid is transformed from solid to liquid. Magic Corporation (Pvt) Ltd. provided the epoxy hardener for this research.

2.2. Synthesis of Composite

To prepare the composites, a total of six layers of fibers were used, three of which were cotton fibers, and the remaining three layers from jute fibers. The layers were maintained in the order of jute/cotton/jute/cotton. The composites were prepared in a hand layup process, shown in Figure 2. Wax-coated polyethylene was used as a surface to avoid the sticking of the laminates. Four types of composites were prepared with varying amounts of spent tea leaves—0 gm, 5 gm, 10 gm, and 15 gm. Spent tea leaf powder was mixed with epoxy resin and epoxy hardener by applying a magnetic stirrer to obtain an even distribution. The ratio between epoxy resin and hardener was maintained at 10:3 during the whole process. Uniform cross-sections were obtained by applying constant load on the fabricated composites. The same process was repeated to develop all composites. The composition of natural-element-based composites is given in Table 3.

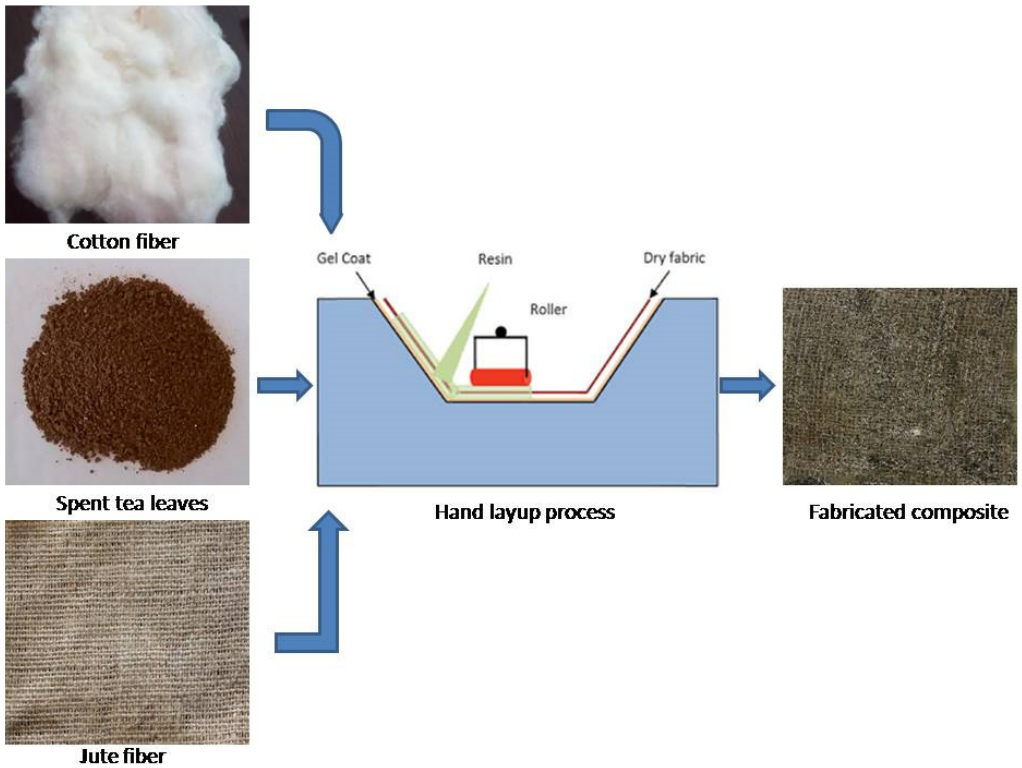


Figure 2. Fabrication process of the natural fibers and reinforced-particle-based composites.

Table 3. Compositions of natural-element-based composites.

Sample No	Layer	Spent Tea Leaves (gm)	Epoxy Resin (%)	Hardener (%)
S1	J/C/J/C	0	70	30
S2	J/C/J/C	5	70	30
S3	J/C/J/C	10	70	30
S4	J/C/J/C	15	70	30

2.3. Mechanical Studies

CMT-10 computer control electronic universal testing machine was employed to characterize the tensile properties of the synthesized composite. All samples were tested for mechanical testing based on the ASTM Standards at room temperature. Tensile and compression tests were performed using Universal Testing Machine (UTM) based on the ASTM D638 Standard, and a bending test was performed based on the ASTM D790 Standard. The length of each sample was 250 mm, and the width was 25 mm.

2.4. Characterization

2.4.1. FTIR

The FTIR spectra were measured with a PerkinElmer Spectrum One spectrometer coupled to an auto-image light microscope. The functional groups present in the composite were determined using wavenumbers ranging from 4000 to 500 cm^{-1}.

2.4.2. SEM

The microstructure of the composites was analyzed using a scanning electron microscope (Hitachi company, Tokyo, Japan). The composite samples were cut into 0.5 cm^2 sizes and immersed in liquid nitrogen, followed by cryogenic fracturing, and then were randomly broken to investigate the surface of the samples. The cryo-fractured samples were fixed on a support by a double-sided adhesive tape mounting on aluminum stubs. Using an accelerating voltage of 10 kV, the samples were observed, coated with gold–palladium at a working distance of 10 mm.

2.4.3. XRD

An X-ray diffractometer 2500 (Rigaku, Tokyo, Japan) was employed for the X-ray diffraction analysis of composite samples, with a speed of scattering at 0.02 (θ) s^{-1} over a range of angles between 5 and 60° (2θ). For this process, 35 mA and 40 kV were the operating current and voltage, respectively. Crystalline area (Ic), relative crystallinity (Xc), and amorphous area (Io) were the outcomes of XRD analysis.

2.4.4. Thermal Analysis

TGA and DSC analyses were performed using a TA Instrument SDT 650. The weight range and heating increment were set at 10–25 mg and 5 °C, respectively, from room temperature to 500 °C.

3. Results and Discussion

3.1. Mechanical Properties of JCPC Spent Tea Leaves Composites

Computer-generated data for the tensile properties of the composites can be seen in Figure 3a–d and Table 4. The comparison data of tensile strength, tensile strain, and elastic modulus can be seen in Figure 4a–c. It is seen that with the addition and increase in the amount of spent tea leaves, the tensile strength of the composites increased but decreased when the amount of spent tea leaves increased from 10 gm to 15 gm. This indicates that tensile strength decreased after the addition of a certain amount of powder due to the agglomeration of spent tea leaves [26]. However, the tensile strain fluctuated with the

addition and increase in the percentage of spent tea leaves. The value of elastic modulus fluctuated with the addition and increase in the amount of spent tea leaves.

Table 4. Tensile properties of JCJC spent tea leaf composites.

Sample Name	Tensile Strength (MPa)	Strain (%)	Elastic Modulus (GPa)
S1	22.43	2.48	9.02
S2	26.14	4.11	6.36
S3	34.89	3.40	10.26
S4	25.64	3.47	7.04

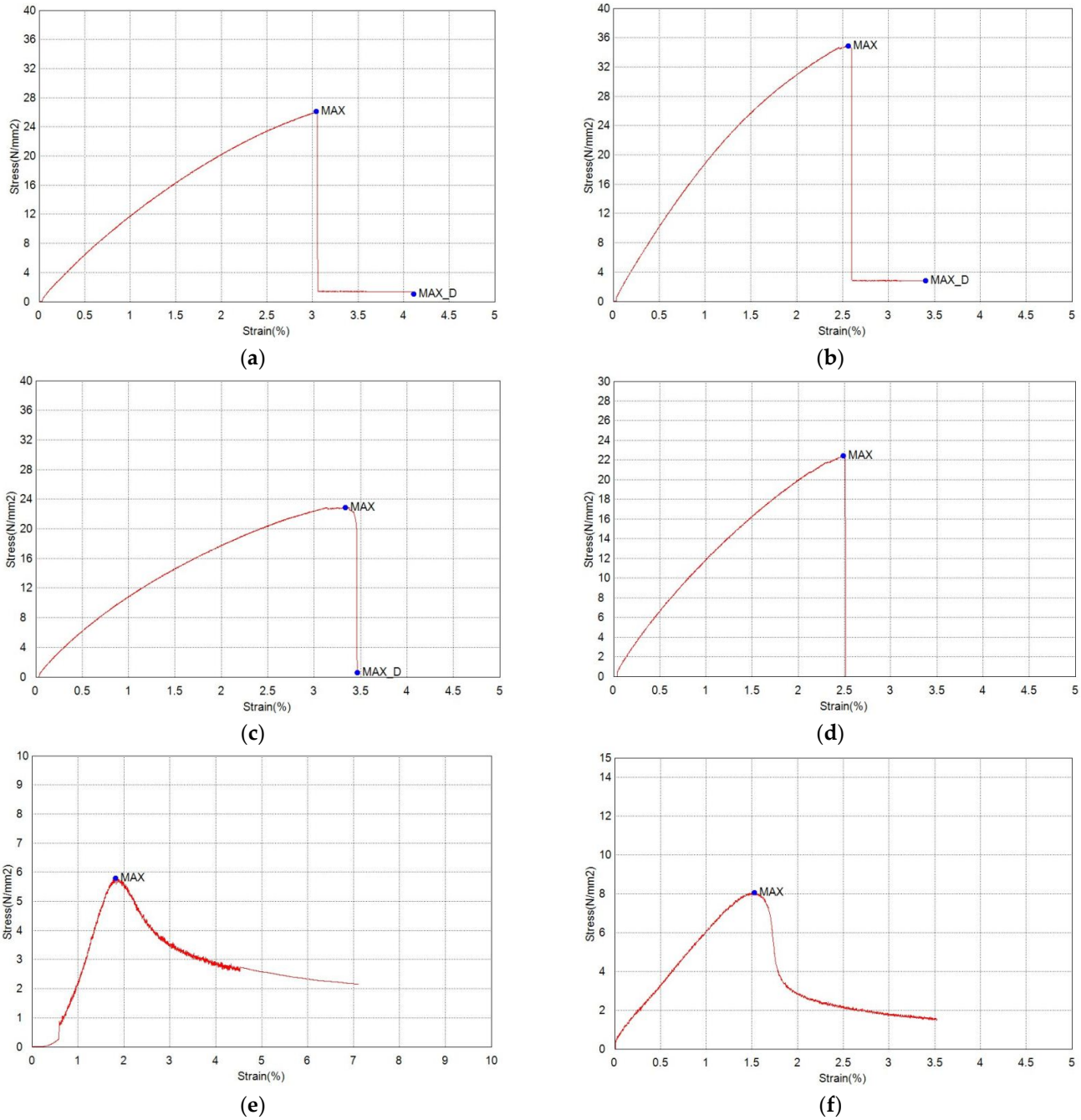


Figure 3. *Cont.*

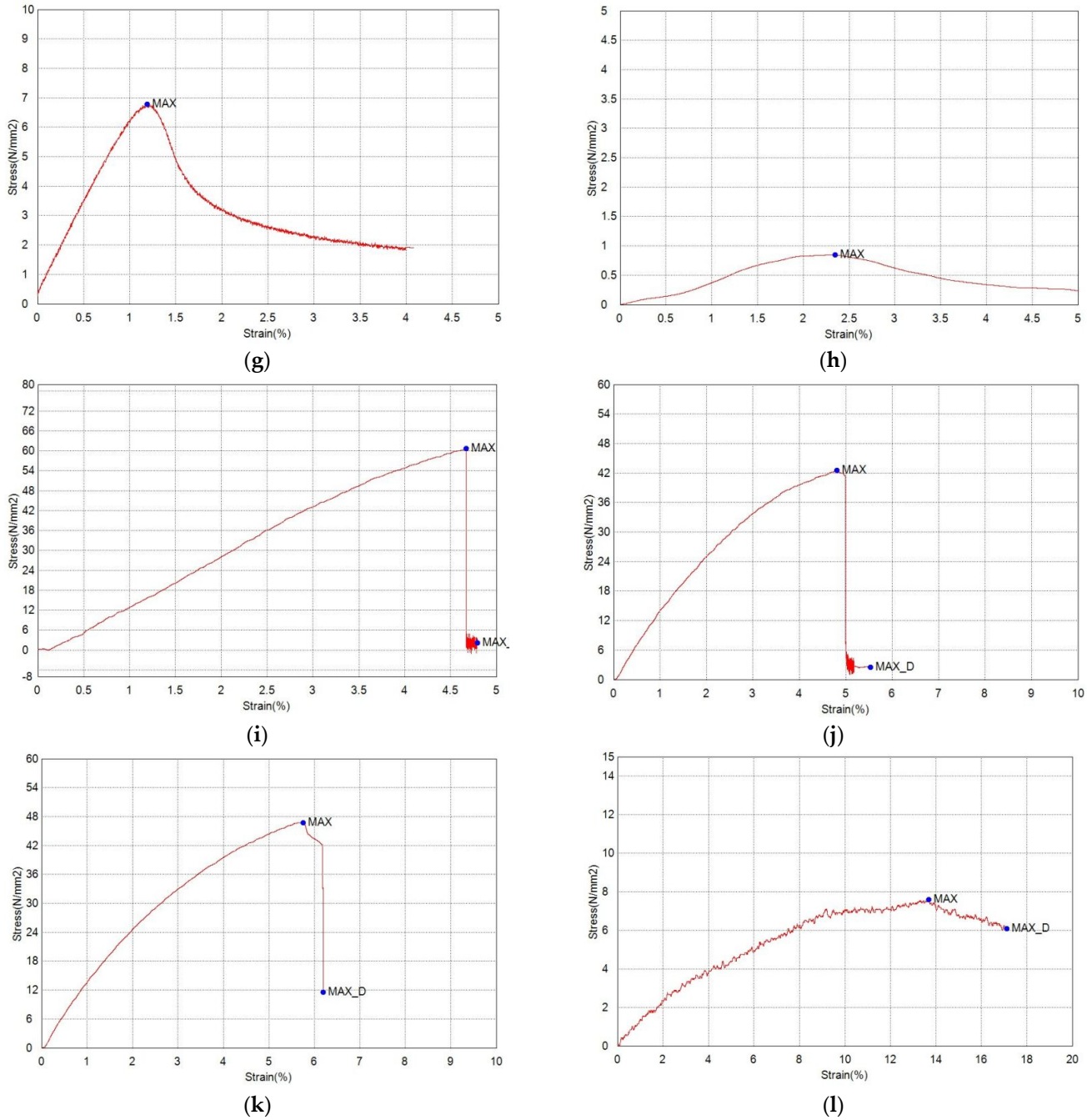


Figure 3. (**a**–**d**) Tensile strength graph of JCJC spent tea leaf composites: (**a**) S1, (**b**) S2, (**c**) S3, and (**d**) S4; (**e**–**h**) compression strength graph of JCJC spent tea leaf composites: (**e**) S1, (**f**) S2, (**g**) S3, and (**h**) S4; (**i**–**l**) bending strength graph of JCJC spent tea leaf composites: (**i**) S1, (**j**) S2, (**k**) S3, and (**l**) S4.

Figure 3e–h show computer-generated data (Table 5) of the compression strength of the composites. Figure 4d–f show the comparison data of compressive strength, compressive strain, and elastic modulus. These figures show that the compressive strength increased with the addition and increase in the amount of spent tea leaves but decreased when spent tea leaves were added and the amount increased from 10 gm to 15 gm. These results indicate that the addition of more than 10 gm spend tea leaves decreased the tensile strength of the composite [27]. However, the compressive strain decreased with the addition and increase in the amount of spent tea leaves. Comparing the compressive properties [28] for the spent tea leaf particles, it can be seen that tensile yield strength was much higher than compression strength. This may be due to fracture failure in the compressive modulus.

Aggregation and bubbles in the matrix may lead to stress concentration. Elastic modulus increased with the addition and increase in the amount of spent tea leaves.

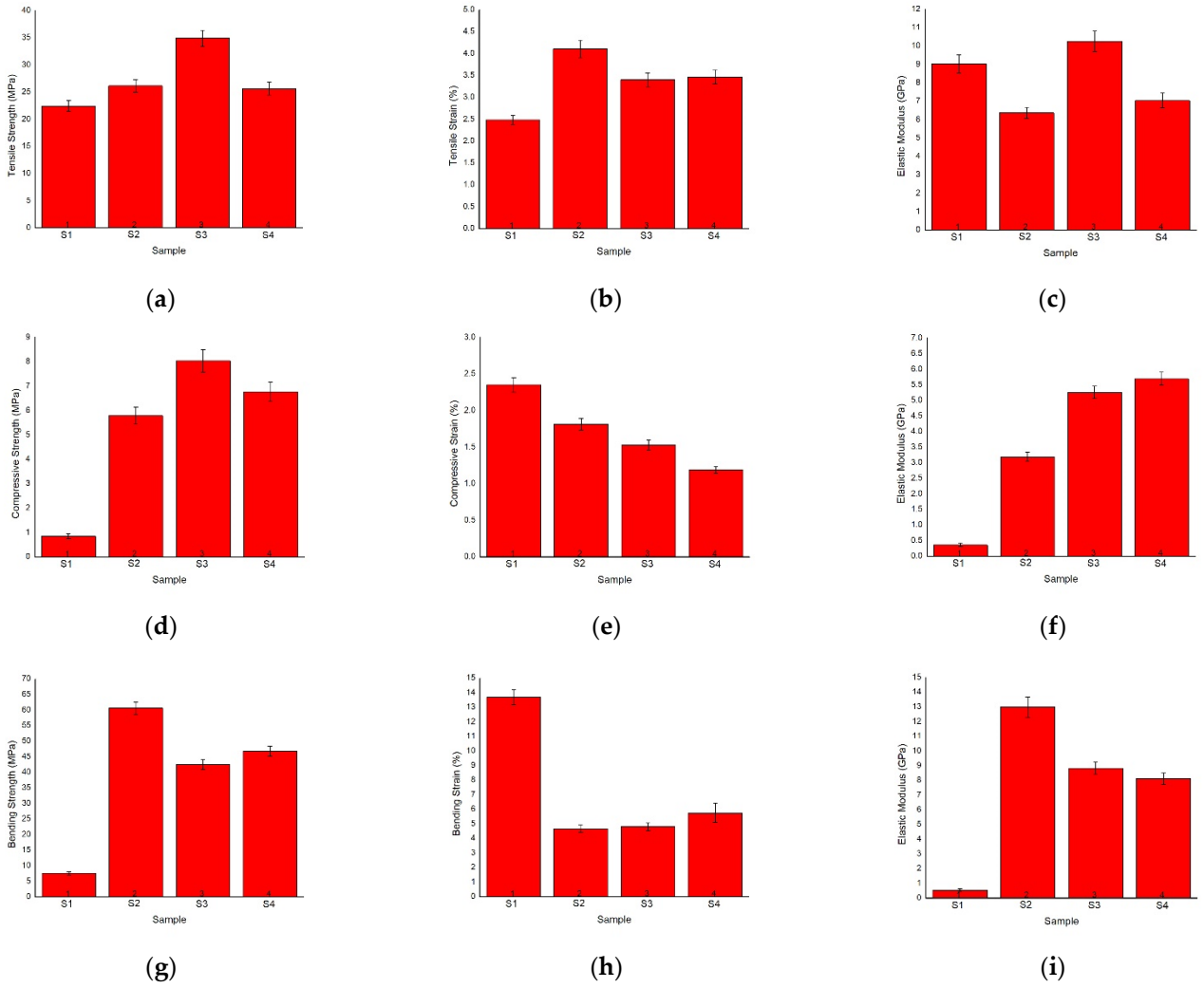


Figure 4. Comparison of mechanical properties in: (**a**) tensile strength, (**b**) tensile strain, (**c**) elastic modulus, (**d**) compression strength, (**e**) compression strain, (**f**) elastic modulus, (**g**) bending strength, (**h**) bending strain, and (**i**) elastic modulus.

Table 5. Compression properties of JCJC spent tea leaf composites.

Sample Name	Compression Strength (MPa)	Strain (%)	Elastic Modulus (GPa)
S1	0.84	2.35	0.36
S2	5.79	1.81	3.19
S3	8.03	1.53	5.26
S4	6.77	1.19	5.69

Figure 3i–l illustrate the computer-generated data of the bending strength of the composites (Table 6). Figure 4g–i illustrate the comparison of bending strength and bending strain. These figures reveal that bending strength increased with the addition of spent tea leaves but decreased with the increase in the amount of spent tea leaves, as spent tea leaves reduced the matrix strength [29] but increased it when the amount of spent tea leaves increased from 10 gm to 15 gm. However, the bending strain decreased with the addition

of spent tea leaves but increased with the increase in the amount of spent tea leaves. Elastic modulus increased dramatically with the addition of spent tea leaves but decreased when the amount increased.

Table 6. Bending properties of JCJC spent tea leaf composites.

Sample Name	Bending Strength (MPa)	Strain (%)	Elastic Modulus (GPa)
S1	7.59	13.68	0.55
S2	60.61	4.66	12.98
S3	42.49	4.80	8.83
S4	46.76	5.75	8.12

3.2. Characterization of JCJC Spent Tea Leaf Composites

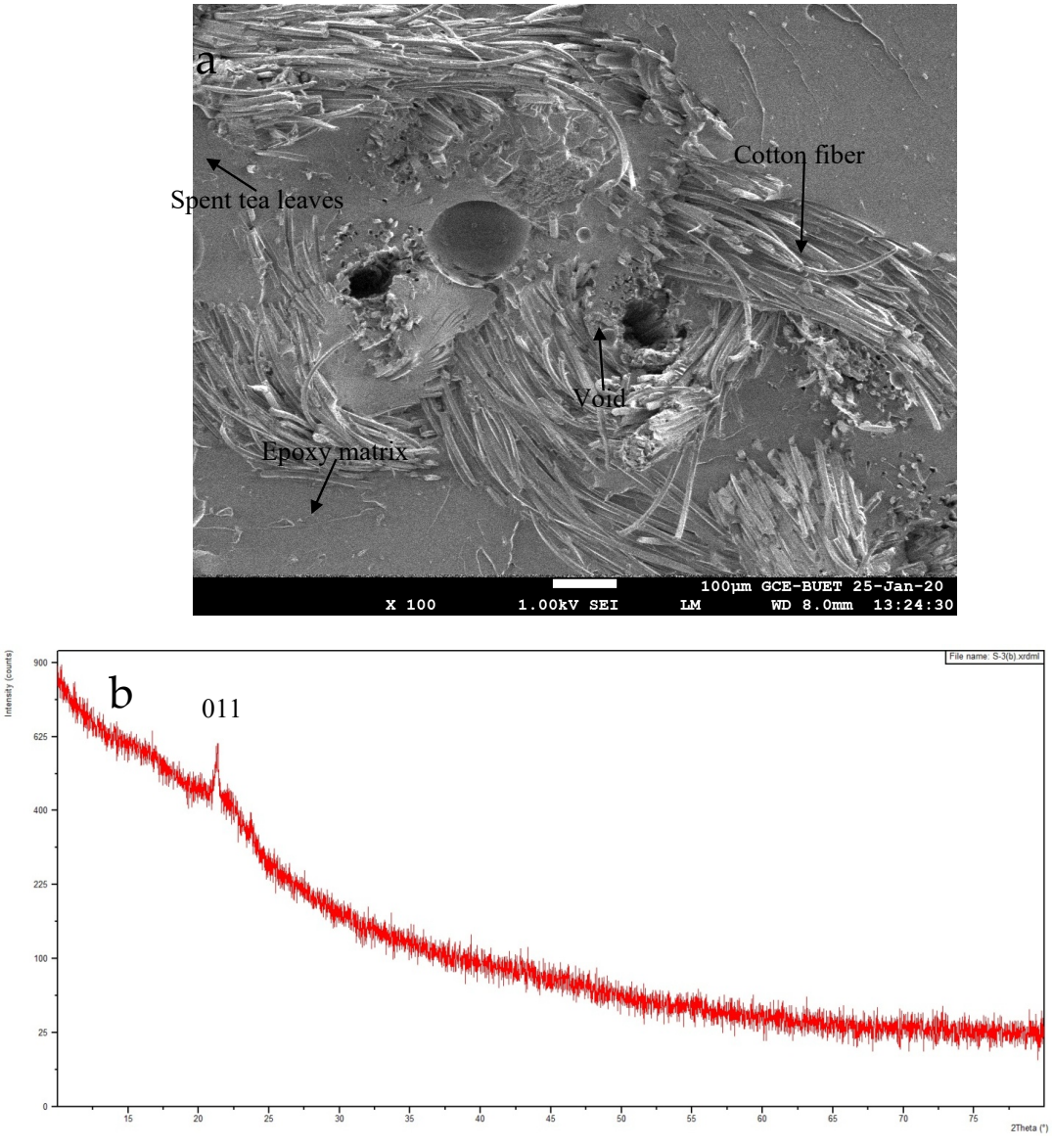
SEM analysis was conducted to analyze the epoxy–fiber–spent tea leaf interaction of the composites, shown in Figure 5a. From the figure, it can be seen that cotton fiber orientation in the matrix was agglomerated. Some voids and cracks were identified, as shown in SEM results, which are caused by a lack of fiber–matrix adhesion [30]. No pullout was seen in the fiber matrix, indicating that there was a strong fiber–matrix bond. The image shows an even distribution of spent tea leaf powder on the surface of the composite. The sample was characterized by homogenous dispersion of fibers, resulting in begat effective stress transfer. Improvement in mechanical properties is observed due to good interfacial adhesion observed between matrix and reinforcement. The X-ray diffraction pattern of JCJC Spent Tea Leaves Composites is shown in Figure 5b. A peak value at approximately $2\theta = 22^{\circ}$ corresponds to (0 1 1), and this indicates that crystalline was fixed for these composites. The crystalline grain size was calculated to be 26.51 nm based on the Scherrer equation. Figure 5c shows three different strong absorption peaks for the JCJC spent tea leaf composites. The different peaks were identified at 345.22 nm, 553.03 nm, and 730.75 nm, which indicates that the spectra transferred from the ultraviolet region to the visible region. The peak at 345.22 nm slightly shifted toward 553.03, and the peak at 730.75 nm indicated the conversion from a localized polaron band to a delocalized polaron band, meaning free-carrier tail absorption [31]. Figure 5d represents Fourier transform infrared spectroscopy (FTIR) graph, and actual spectra were analyzed in the range of wavenumber 650–4000 cm^{-1}. The FTIR data were examined to characterize the chemical bonds present in the composite. By identifying the band-position shifting of the spectra, the interactions between individual components in the composite were determined. The chemical structure and polymer chains were observed through the FTIR analysis. The presence of characteristics peaks, chemical functional groups, assignments, and vibration types are shown in Table 7. The peak 2966 cm^{-1} shifted to 2916 cm^{-1} and 2646 cm^{-1}, which correspond to hydrogen, methylene, and aldehyde groups, and vibration types of stretching, asymmetric stretching, and stretching, respectively [32]. The peaks 1472 cm^{-1} and 1452 cm^{-1} attributed to C-H (methylene) functional class, with bend-type vibration [33]. Aromatic rings were found at 1179.93 cm^{-1} and 1068.08 cm^{-1}, which attributed to plane bend.

Table 7. Characteristics, chemical properties, functional groups, and vibration types of JCJC spent tea leaf composites.

Band	Functional Class	Assignment	Vibration Type
2966	Hydrogen	C-H	Stretching
2916	Methylene	C-H	Asymmetric stretching
2646	Aldehyde	C-H	Stretching
1659	Alkenyl	C=C	Stretching
1507	Aromatic ring	C=C-C	Stretching

Table 7. *Cont.*

Band	Functional Class	Assignment	Vibration Type
1472	Methylene	C-H	Bend
1462	Methylene	C-H	Bend
1378.54	Gem-Dimethyl	–CH3	Doublet
1260.57	Vinylidene	C-H	Bend
1179.93	Aromatic	C-H	In-plane bend
1068.08	Aromatic	C-H	In-plane bend
1023.56	Alkyl-substituted ether	C-O	Stretching
874.91	Peroxides	C-O-O-	Stretching
803.39	Alkene	C-H	Bend
729.61	Alkene	C-H	Bend
719.26	Aromatic	C-H	Bend



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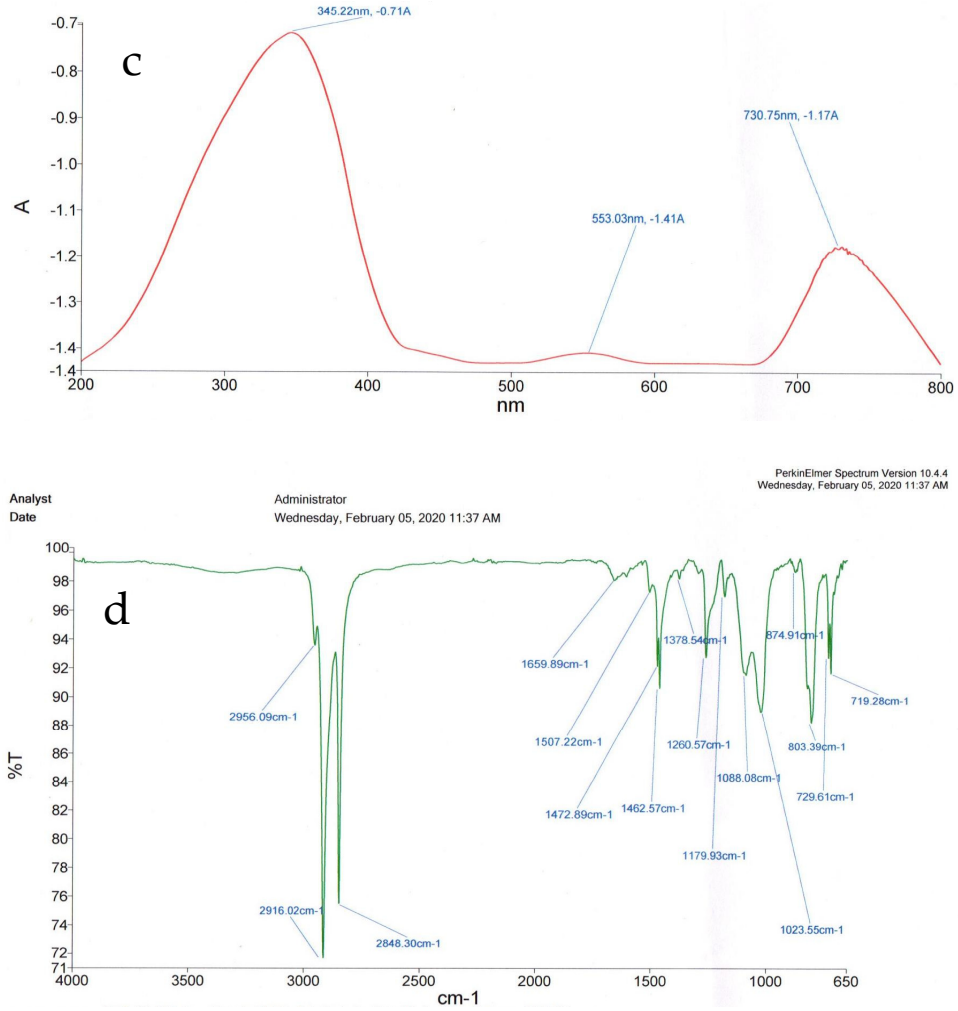

Figure 5. Characterization of JCJC spent tea leaf composites: (**a**) SEM image, (**b**) XRD analysis, (**c**) UV–Vis spectra, and (**d**) FTIR graph.

The chemical elements were identified using energy-dispersive spectroscopy (EDS), as shown in Figure 6. C, O, N, and P concentrations were determined in the JCJC spent tea leaf composites. The highest percentage element was carbon, which amounted to 58.63% of the total mass, and the second-highest element was oxygen (O), amounting to 23.96%. Surface morphology was analyzed in terms of surface topology and 3D surface topology. As shown in Figure 7, 3D surface topology was almost homogenous in the distribution of matrix, fibers, and particles, and surface topology was formed at 50 µm. Figure 8a shows particle size detection and distribution, according to which the average particle size was identified at 31.91 µm and almost homogeneously distributed. Surface roughness was identified to be 47 µm, as seen in Figure 8b. For the projected area, the standard deviation was obtained at 7.82 μm^2 and for equivalent diameters, the standard deviation was obtained at 0.031 mm.

Thermogravimetric analysis (TGA) was performed to investigate the thermal stability of the JCJC spent tea leaf composites containing 5 g spent tea leaf powder. Figure 9a,b show thermal degradation as a function of heat flow rate and mass loss. It is well established that, in TGA, when the material is degraded, weight loss ocurrs in the form of volatiles [34]. From Figure 9c, it can be deduced that glass transition temperature was obtained at 127 °C, and at 300 °C, dramatic weight loss occurred. The glass transition temperature of natural fiber was lower than that of the glass-fiber-based composites. The major degradation temperature was found at 330 °C, and almost the same data were obtained from hybrid composites and sisal-fiber-based composites. Differential scanning calorimetry (DSC) was used to investigate heat flux and temperature, with material phase transition as a function of

temperature and time. Endothermic and exothermic peaks for qualitative and quantitative analyses were provided through DSC analysis. The endothermic peak was found to range from room temperature to 130 °C, which is related to the dehydration process of the natural composites [30]. This may be due to the fact that the natural fiber contents in JCJC spent tea leaf composites exhibit higher water absorption [35]. In addition, an exothermic peak was identified at around 320 °C, which signifies the decomposition of cellulose of the fibers in the matrix.

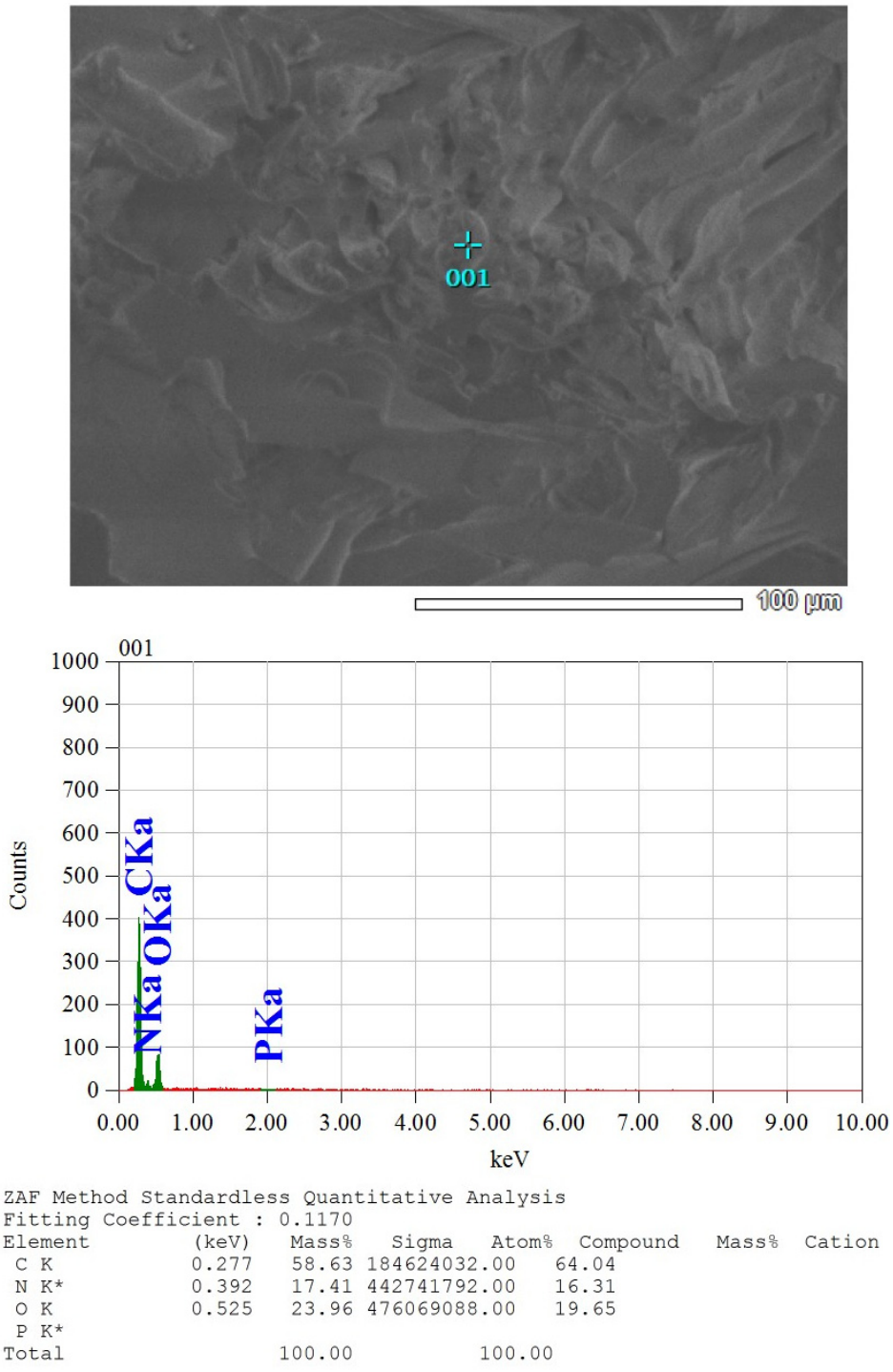

Figure 6. Explain of Energy-dispersive spectroscopy analysis of JCJC spent tea leaf composites.

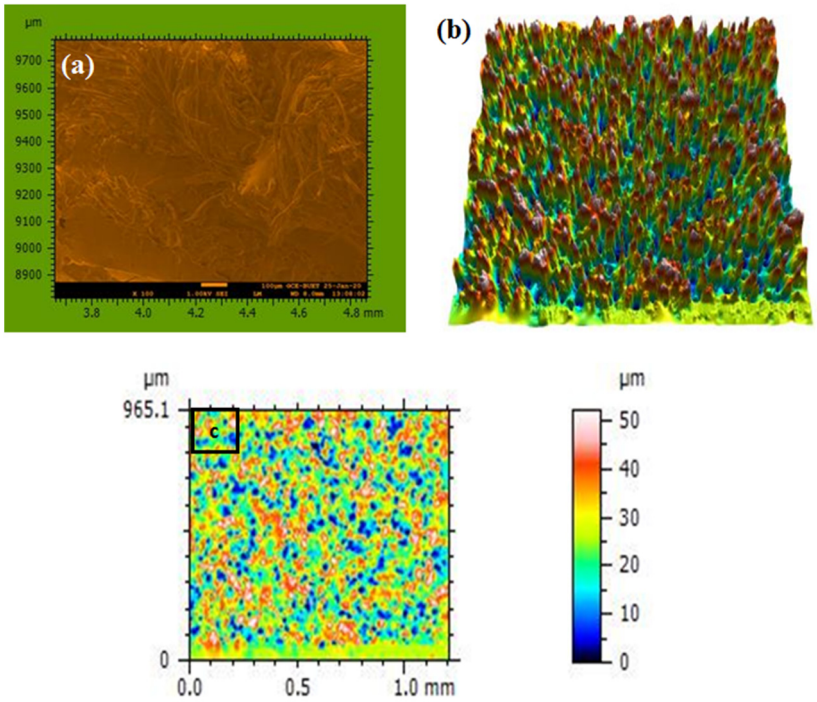

Figure 7. Surface morphology of JCJC spent tea leaf composites: (**a**) SEM pseudo-colored image, (**b**) 3D topology, and (**c**) surface topology.

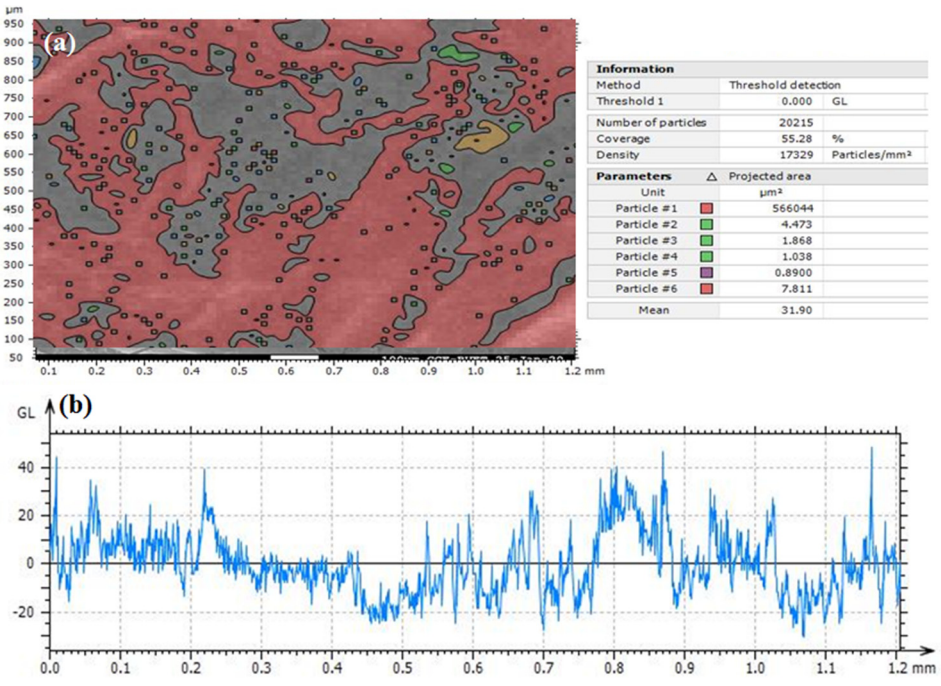

Figure 8. Particle analysis of JCJC spent tea leaf composites, (**a**) particle size detection and (**b**) surface profile.

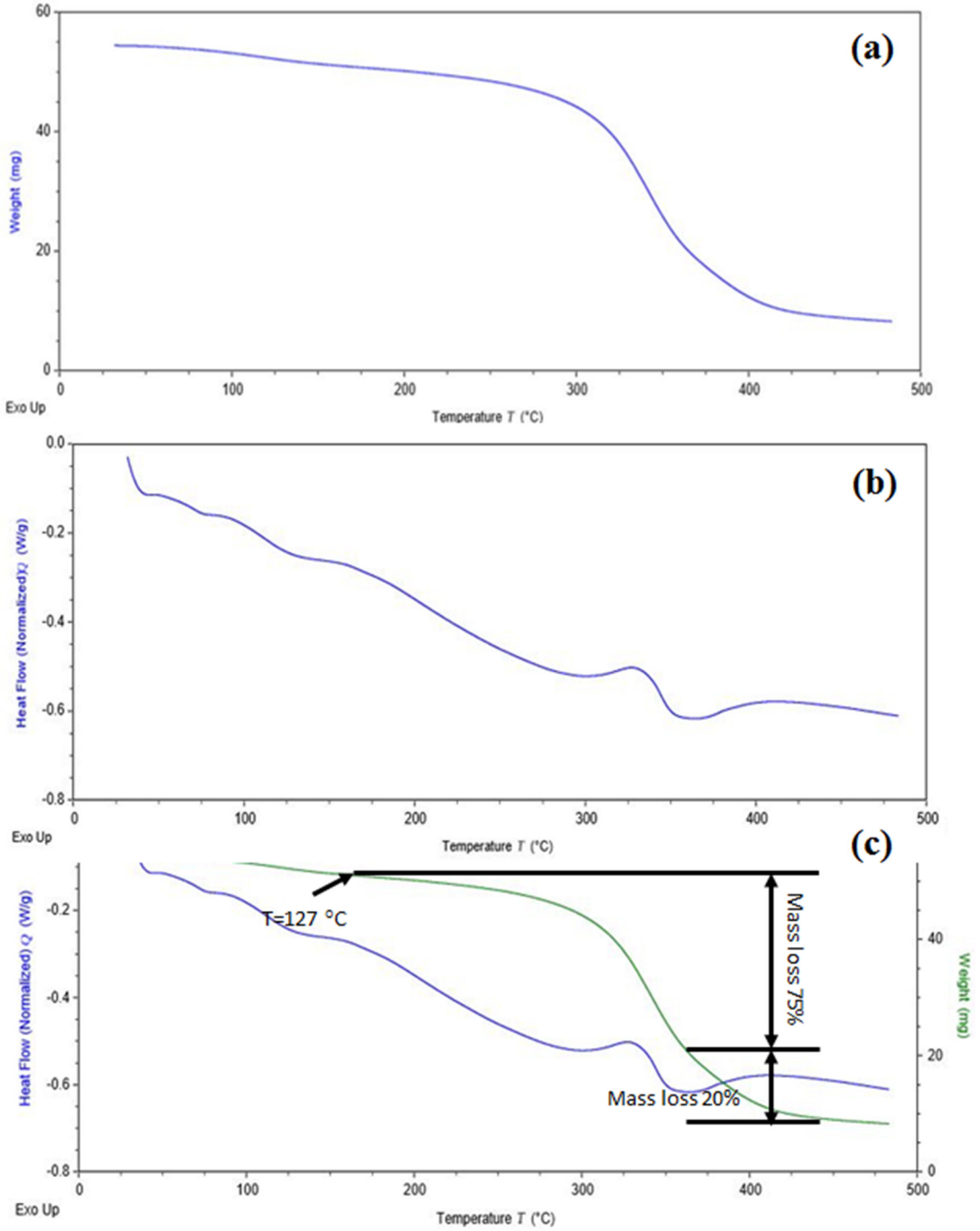

Figure 9. Thermal analysis of the JCJC spent tea leaf composites: (**a**) TGA curves, (**b**) DSC curves, and (**c**) TGA and DSC combined curves.

4. Conclusions

In this study, composites were fabricated using jute and cotton fibers in six layers, reinforced by spent tea leaf powder at varying proportions with the resin matrix. The effects of reinforcement in terms of mechanical, microstructural, morphological, and thermal properties were investigated. The results showed improvement in tensile and compressive strength by 33.46% and 38.86%, respectively, by the addition of 10 gm reinforcement of spent tea leaves. Moreover, 15 gm reinforcement of spent tea leaves increased bending strain by 23.23%. The presence of fiber, reinforcement, and resin was identified with the SEM analysis. Furthermore, 26.61 nm size grain structures were identified in the XRD analysis. Peaks were identified at 345.22 nm, 553.03 nm, and 730.75 nm based on the UV

analysis. FTIR analysis identified the presence of polymer chains. The presence of C, O, N, and P elements were observed with the EDS analysis. Glass transition temperature was found at 127 °C, and major degradation temperature was found at 330 °C. Further research can be carried out by reinforcing more natural and eco-friendly resources.

Author Contributions: Conceptualization, M.A.C.; methodology, A.R.; software, A.R.; validation, N.H.; formal analysis, N.H.; investigation, M.B.A.S.; resources, M.B.A.S.; data curation, M.A.K.; writing—original draft preparation, N.H.; writing—review and editing, M.B.A.S.; visualization, M.R.R.; supervision, M.T.S.C.; funding acquisition, K.K.K.; project administration, M.M.R. All authors have read and agreed to the published version of the manuscript.

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Conflicts of Interest: The authors declare no conflict of interest.

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