Influence of Washing with Sodium Lauryl Sulphate (SLS) Surfactant on Different Properties of Ramie Fibres

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Green composite materials are a means of reducing reliance on synthetic and especially single-use plastics (SUP) and raising public awareness of the need for urgent action to protect the planet. Natural (lignocellulosic) fibres are increasingly utilized as the reinforcement in polymer matrix composites, in search for increased renewability and sustainability. This work concerns the effect of washing ramie (*Boehmeria nivea*) fibres using sodium lauryl sulphate (SLS) surfactant. The SLS-treated ramie fibres were examined for their morphological, physical, thermal, structural, and mechanical properties by powder X-ray diffraction (XRD), Fourier transform infrared (FTIR) spectroscopy, thermogravimetric analysis (TGA), scanning electron microscopy (SEM), and tensile testing. SLS treated ramie fibres density and crystallinity index values were 1.23 g/cc and 84.5%, respectively, with a very high cellulose content of 81.3%, because hemicellulose and loose particles were dissolved. SEM images depicted the relevant changes, with no significant damage on treated fibre surfaces. With some assistance from the treatment, fibres initiated their degradation only above 250 °C, culminating at 327 °C, which appears suitable for the manufacturing of composites with the most common matrices.

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INTRODUCTION

A rise in environmental legislation has paved the way for studies into renewable, recyclable, and biodegradable fibres and biocomposites (Bhuvaneshwaran *et al*. 2019b, 2021; Almeshaal *et al*. 2022; Mohan *et al*. 2023). When analysing the qualities of natural fibres, complexities arise due to the need to single out the plant species and cultivars from which the fibre is obtained, and the environmental circumstances under which it is cultivated (Nair *et al*. 2013; Bhuvaneshwaran *et al*. 2019a; Selvaraj and Mylsamy 2023). One of the main factors that controls the properties of natural fibres is the respective amount of cellulose, hemicellulose, and lignin into them. Because of their low density, appropriate flexibility, and comparatively high tensile strength, cellulose-based fibres, *e.g.*,

cotton, jute, flax, hemp, and sisal, are well-suited for uses in biocomposites, paper manufacturing, textiles, and other industries. Furthermore, their high levels of stiffness and strength, lignin-containing fibres, such as those found in wood fibres, are well suited for use in composites and construction materials. Natural fibres are used as replacements for synthetic fibres in a broad variety of applications (Arockiam *et al*. 2018; Mylsamy *et al*. 2023; Palanisamy *et al*. 2023). For instance, potential enhancement of cargo capacity in aeroplanes may be attributed to the reduced weight of natural fibre composites (NFCs), in addition to favorable insulating and tribological properties. Boeing and Airbus, prominent entities in the aviation sector, dedicated substantial resources towards investigating the use of natural fibres in the design of aircraft interiors (Mylsamy *et al*. 2020; Aruchamy *et al*. 2023a; Santulli *et al*. 2023; Selvaraj *et al*. 2023a). NFCs refer to composite materials comprising a blend of natural fibres and a matrix material. The use of NFCs is seeing a growing trend due to their sustainability and ecologically conscious nature, serving as a viable alternative to traditional composite materials that mostly depend on synthetic fibres (Elanchezhian *et al*. 2018; Selvaraj *et al*. 2023b; Sumesh *et al*. 2023; Venkatesh *et al*. 2023).

Different materials such as thermosetting resins or thermoplastics, including bio-based polymers, are used for manufacturing NFCs. When compared to traditional composite materials, NFCs have substantial advantages. Beyond their lower density with respect, *e.g.*, to glass fibres, natural fibres may have sufficiently high aspect ratio, are sound proof and biodegradable at end-of-life, while still their mechanical and thermal resistance are significant (Jawaid and Abdul Khalil 2011; Yusriah *et al*. 2014; Sanjay and Yogesha 2017; Balu *et al*. 2020). Fibres that are hydrophilic face a threat in environmental exposure: therefore, the possibility to use them in making composites, does often require a treatment. In most cases, compatibility between the phases can be achieved by the use of a chemical compound for coating (Ali *et al*. 2018; Sepe *et al*. 2018; Mylsamy *et al*. 2019; Aruchamy *et al*. 2023b). Mechanical properties and interfacial bonding are reduced due to factors that include orientation of microfibrils and contact angle. A typical treatment for the aim of surface washing and removal of wood extractives is performed with alkali (normally sodium hydroxide, NaOH). Studies in literature show that treating reinforcement fibres with alkali made a big difference in their mechanical properties, though resulting in inducing a substantial brittleness and a reduction in ultimate strain (Elanchezhian *et al*. 2018; Bhuvaneshwaran *et al*. 2019b; Mylsamy *et al*. 2019, 2020; Nagappan *et al*. 2022; Palanisamy *et al*. 2022a; Venkatesh *et al*. 2023). In some cases, though, tailoring the treatment duration, consistent improvements are found: specifically, an 8 hours alkali treatment of Borassus fruit fibres makes them 41% higher in tensile strength, 69% stronger in modulus, and 40% better in extension (Reddy *et al*. 2013). Substantial improvements were also observed by the application of a 5% NaOH treatment on *Acacia caesia* bark fibre, where the alkali was able to remove amorphous constituents of the fibre, yet exposing it very much to the environmental conditions (Sivasubramanian *et al*. 2021). Improved tensile strength and stiffness properties were particularly obtained in Ijuk (*Arenga pinnata*) fibre when treated with potassium hydroxide (KOH) over other strong basic solutions, such as NaOH and ammonium hydroxide (NH_4OH) (Santhiarsa 2016). The variety of chemical treatments applicable is very wide though, to offer an effective hydrophobic coating to the fibres: among these are bleaching, benzoylation, potassium permanganate, silanization, acetylation, *etc*. (Sathish *et al*. 2021; Palanisamy *et al*. 2022b).

This work focused on ramie (*Boehmeria nivea*), also known as China grass, which belongs to a fibre-yielding plant of the nettle family (Urticaceae), and namely its bast fibre,

native to China. For around 6000 years, ramie fibre has been used for fabric production and is henceforth considered as one of the oldest fibre crops.

Fibres for this study were collected from local markets from Coimbatore, Tamilnadu, India. Ramie has the specific features of a textile fibre, yet not much research has been done on its surface treatment. Here, a relatively light washing process is suggested using sodium lauryl sulphate (SLS), trying to avoid the use of alkali. Treated fibres were characterized and compared with untreated ones. In particular, the tensile strength of the fibre over different length has also been measured. Thermogravimetric analysis (TG-DTA), X-ray diffraction (XRD), Fourier transform infrared spectroscopy (FTIR) and scanning electron microscope studies (SEM) were used to characterize the fibers. Experimental research indicates that ramie fibre that has been handled with SLS has some potential to be used as a prospective and effectively strengthening composite filler, and it is not substantially damaged by the treatment.

EXPERIMENTAL

Treatment of the Ramie Fibre

The ramie fibres were purchased from the local market in Coimbatore. The fibres were treated with SLS solution (5%) for 1 h at room temperature with no agitation. After fibres were treated with surfactant solution they were submerged in distilled water, again for 1 h, to wash away the residues of SLS. At last, the fibres were removed from the solution manually, and dried in open atmosphere for a period of one week, as suggested in a previous study on cotton-kenaf SLS treatment (Karthik *et al*. 2023). This was also done in order to avoid affecting the mechanical tests results by the "slippery" character of SLS.

Physical Properties and Chemical Composition

The percentage of hemicelluloses, lignin, and cellulose content of the ramie fibre were determined as described (Dorée 1933; Pearl 1967). The density of ramie fibre was evaluated by ASTM D 1577-07 (2018). As per common protocol the content of wax was evaluated. Ash content was analysed using ASTM standard E 1755-01 (2020) (Selvaraj and Mylsamy 2023). A universal testing machine was utilised, equipped with a 1 kN load cell, actioned at a crosshead speed of 0.1 mm/mm, for randomly selected samples of ramie fibre with different gauge lengths (10 to 50 mm), testing ten samples per gauge length, according to ASTM D 3822M-14 (2020).

Fourier Transform Infrared (FTIR) Spectroscopy

A Perkin Elmer RXI FTIR analyser was employed to ascertain the presence of free functional groups on ramie fibre. Wave spectral outputs spanning the frequency range of 400 to 4000 cm^{-1} were acquired at a rate of 32 readings per second in transmittance mode.

X-Ray Diffraction Analysis (XRD)

The crystallinity content in ramie fibre was measured by particle X-ray diffraction. An X'PERT PRO diffractometer was used with 30 mA current, 40 kV tension generator with Cu anode substance at 25 °C temperature with 0.05° C resolution (20 step size). The range of the diffraction angle for CuKα radiation was between 5° and 50°, with a wavelength of 0.154 nm. From XRD, CrI (crystalline index) of cellulose and crystallite size (I002) of α-cellulose of the fibres were calculated.

Thermogravimetric Analysis (TGA)

Thermogravimetric analysis was performed with a STA NETZSCH Model 449 Jupiter (Selb, Germany). Nitrogen gas was released into the testing ambient at a rate of 20 mL/min to eliminate the effects of oxidation. Crushing 10 mg of ramie fibre and utilising the thermocouple technique with an alumina crucible ensures accurate temperature readings and improved sample-to-sample coupling. The fibres were subjected to temperatures ranging from 25 °C to 700 °C, heating at a rate of 10 °C/min.

Surface Morphology

Scanning electron microscope (SEM) was used to examine the fibre surface. Before observing the fibre surfaces with SEM, to prevent the release of electrical particles during testing, a small coating of gold was applied to the surface of the ramie fibre specimens.

RESULTS AND DISCUSSION

Physical and Chemical Analysis

SLS treatment of ramie fibres was able to slightly increase their density from 1.2±0.05 to 1.23±0.06 g/cc. The procedure applied was also mainly able to reduce the amount of hemicellulose and loose matter, while it did not produce substantial damage on the fibres. Chemical composition appears in Table 1, compared with a number of other recently investigated and less diffuse ligno-cellulosic fibres. The concept of "less common fibres" has been explored from the review by Sarasini and Fiore and has a specific validity in the sense that it tries to extend the field of natural fibres, and to establish possible comparisons as for applicability between local fibres from different regions (Sarasini and Fiore 2018). In most cases, some of which are reported to Table 1, the principal idea of using "less common fibres" is to demonstrate their potential in natural fibre composites, despite the fact that they do not originate from the restricted number of crops that offer widely available and marketable textiles (*e.g*., flax, hemp, jute, kenaf, pineapple, cotton). For this reason, the question of fibre treatment has been normally postponed to further studies, to be performed after a reasonably facile extraction of the fibres and a sufficient mechanical performance is demonstrated. In the case of ramie, the composition does appear quite typical, with limited amounts of wax and ashes: the objective of a treatment would hence prevalently be the reduction of hemicellulose content.

In Table 2, less diffuse fibres are examined after their treatment, starting from SLS treated ramie. In most cases, such as those reported, less diffuse fibres were treated using an alkali solution based on NaOH, most normally at 5 wt%. The higher cellulose content obtained by SLS treatment (81.3%) is likely to contribute to the enhancement of mechanical properties in fibres, whereas the presence of hemicelluloses (8.8% after SLS treatment) would have a detrimental effect on fibre strength due to the deterioration of microfibrils (Dias *et al*. 2019). The residual presence of lignin (5.5 wt%) maintains some rigidity to the ramie fibre: on its elimination, SLS treatment had a quite reduced effect (Banerjee *et al*. 2015). SLS treatment was also not able to remove wax, which, in the potential manufacturing of composite materials, could reduce the interfacial bonding among the fibre and matrix, though the effect has been found to be more obvious in lignin-rich fibres, such as coir (Brahmakumar *et al*. 2005). The two cases of comparative studies between untreated and treated fibres reported in Table 2, namely for *Coccinia grandis* and *Prosopis juliflora,* suggest that alkali treatment mainly decreased the hemicellulose.

Table 1. Composition of Untreated Ramie Fibre *vs*. Other Untreated Less Diffuse Natural Fibres

Fibre	Cellulose %	Wax %	Ash %	Lignin %	Moisture %	Hemicellulose %	Reference
Ramie	70.7	0.7	1.9	10.7	9	14.3	Present study
Coccinia indica	64.56	0.25	-	12.55	7.27	14.09	Bhuvaneshwaran *et al*. (2021)
Tithonia diversifolia	65.33	0.67	6.78	15.06	9.23	12.25	Selvaraj and Mylsamy (2023)
Ageratina adenophora	65.7	1.3	1.3	12.5	7.4	11.2	Selvaraj *et al*. (2023a)
Ficus carica	63.17	0.42	1.05	13.7	9.07	12.08	Selvaraj *et al*. (2023b)
Hibiscus vitifolius	75.09	0.17	0.94	10.42	11.31	13.34	Manivel *et al*. (2022)
Calotropis procera	54.5	1.5	7.6	14.8	9.3	12.3	Yoganandam *et al*. (2019)
Sida mysorensis	53.36	0.86	3.33	9.46	10.48	15.23	Maran *et al*. (2022)
Sansevieria zeylanica	76.12	-	1.36	4.28	-	9.32	Ponnu Krishnan and Selwin Rajadurai (2017)
Coccinia grandis	62.35	0.79	4.39	15.61	5.6	13.42	Senthamaraikannan and Kathiresan (2018)
Prosopis juliflora	61.65	0.61	9.48	17.11	-	16.14	(Saravanakumar *et al*. 2014)

Table 2. Composition of SLS Treated Ramie Fibre vs. Other Untreated Less Diffuse Natural Fibres

Fibre	Treatment	Cellulose %	Wax %	Ash %	Lignin %	Moisture %	Hemicellulose %	Reference
Ramie	SLS	81.3	0.7	1.9	5.5	8	8.8	Present study
Coccinia grandis	NaOH	68.47	0.56	7.84	11.32	4.8	8.64	Senthamaraikannan and Kathiresan (2018)
Prosopis juliflora	NaOH	72.27	0.1	6.36	12.09	-	4.02	Saravanakumar *et al*. (2014)
Cissus populnea	Acetylation	61.8	1.6	7.6	11.5	3.9	14.7	Azeez *et al*. (2018)
Symphirema involucratum	NaOH	68.69	0.31	13.46	7.54	6.84	7.46	Raju *et al*. (2021)

The tensile strength of the natural fibres was slightly influenced by the treatment. Starting from values of 487 ± 21 MPa for 50 mm gauge length on untreated fibres, the relevant results for SLS treated fibres are reported in Table 3. A limited increase of strength was observed with growing length, which suggests that the influence of defects along the fibres was quite limited and that the treatment was not damaging the fibres. Young's modulus was measured with some approximation, considering the fibres as circular, and taking the diameter measurements over a set of ten fibres each (five measurements per fibre) for untreated and SLS treated ramie. The measurements taken offered values of 51 ± 2.5 µm for untreated fibres, and 46.5 ± 1.9 µm for SLS treated ones, demonstrating that some residual and non-structural matter was removed.

Table 3. Mechanical Properties of SLS Treated Ramie Fibres

Gauge Length (mm)	Tensile Strength (MPa)	Young's Modulus (GPa)	Ultimate Strain (%)
10	486 ± 23	5.6 ± 1.5	5.3 ± 1.3
20	498 ± 22	5.7 ± 1.6	5.4 ± 1.4
30	505 ± 21	5.8 ± 1.4	5.5 ± 1.2
40	512 ± 27	5.9 ± 1.7	5.5 ± 1.4
50	508 ± 29	5.8 ± 1.7	5.6 ± 1.5

XRD Analysis

Figure 1 depicts X-ray diffraction spectrum of untreated and SLS treated ramie fibre. As from Segal's law (Segal *et al*. 1959), the crystallinity index was calculated by Eq. 1, based on the intensity of the (002) peak I_{002}, which is slightly shifted by the treatment, from 23.6° to 23.4°, and of the low intensity peak I_{am}, corresponding to the amorphous cellulose fraction and localized in this study at 18.6°. A recent study on pineapple crown leaf (PCL) fibres also used this method (Johny *et al*. 2023), coming to similar conclusions about the presence of different peaks. In particular, it also included (101) cellulose peak at 14.8° (here at 16.2° for untreated and 15.8° for treated fibres) and a smaller (040) cellulose peak at 35.8° (here at 35.6° for untreated and at 35.4° for treated fibres).

$$CI = [(I_{002}\text{-}I_{AM})/I_{002}]*100 \quad (1)$$

The crystallite size D_{002} was measured from Scherrer's law according to the diffraction peak broadening in the XRD curves, in practice using Eq. 2,

$$D_{002} = k\lambda/(\beta_{002} * \cos\theta) \quad (2)$$

where k is a constant, normally equal to the unity, λ = X ray wavelength used in XRD (in nm), and β_{002} = true peak broadening at full-width half maximum (FWHM) in radians.

Potential limitations of these direct measurements from XRD curves obtained from various lignocellulosic fibres, particularly concentrating on bamboo and relevant viscose, are reported in Rocky and Thompson (2021), which suggests the substantial validity of the method, even in absence of a deconvolution process.

A slight decrease of crystallinity index, still considerably high, and a subsequent increase of crystallite size was revealed by the results reported in Table 4. Always adhering to the concept of less diffuse fibres, some of these, such as *Coccinia indica* (Bhuvaneshwaran *et al*. 2021) and *Ficus carica* (Selvaraj *et al*. 2023b), showed much lower crystallinity indexes, being respectively equal to 53.0 and 41.4%. As regards to crystallite size, value obtained, though very small, would be still higher than is the case for other

recently investigated fibres, such as *Althaea officinalis* (2.4 nm) (Sarikanat *et al*. 2014) and *Ferula communis* (1.6 nm) (Seki *et al*. 2013). It is suggested that the low crystallite size would still minimize water absorption and chemical reactivity in a matrix, despite some increase after SLS treatment (Tamanna *et al*. 2021). Crystallinity index and crystalline size of ramie fibre are listed in Table 4; the values can be compared with those of various other natural fibres, reported in Table 5.

Table 4. Crystallinity Index and Crystallite Size in Untreated and Treated Ramie Fibres

Ramie Fibre	Crystallinity Index (CI) (%)	Crystallite Size (L) (nm)
Untreated	84.4	2.28
5% SLS treated	81.5	2.66

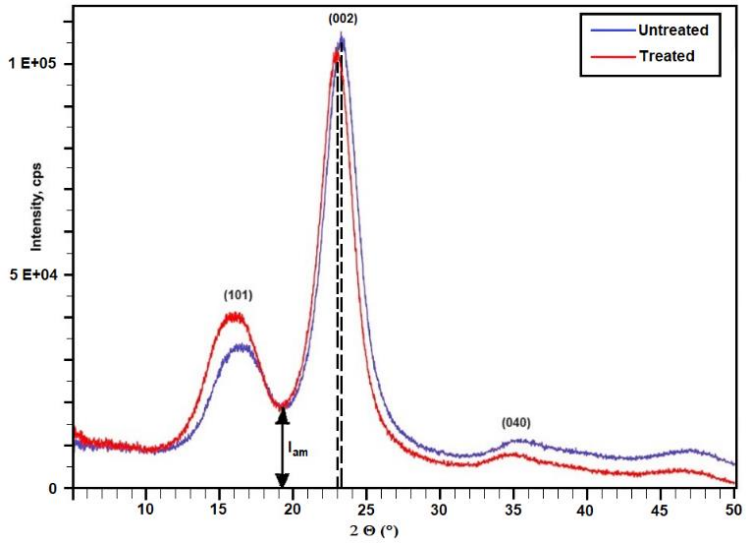

Fig. 1. XRD analysis of untreated and SLS treated ramie fibre

FTIR Analysis

Functional groups present in untreated and SLS treated ramie fibre were found using FTIR analysis, which is depicted in Fig. 2. The differences between spectra of untreated and treated fibres were minimal. As far as the superposition of cellulose, lignin, and hemicellulose signal is concerned, it is noteworthy that, as indicated in Fig. 3, taken from (Raspolli Galletti *et al*. 2015), in the lignin region, centred on the 1246 cm^{-1} peak, indicating the presence of acetyl groups, as also suggested in (Park *et al*. 2010), minor subpeaks had somewhat faded away. In contrast, there was no clear evidence of hemicellulose reduction. If present, possibly such evidence was being concealed by the main cellulose peak at 1030 cm^{-1}, indicating C-O stretching (Nagarajan and Balaji 2016). Another visible effect from FTIR spectra is the reduction of the 1336 cm^{-1}, as the bending in-plane of hydroxyls (-OH), a peak corresponding to amorphous cellulose, which might suggest some "washing effect" (Maceda *et al.* 2022). However, the differences between untreated and treated fibres were really quite limited.

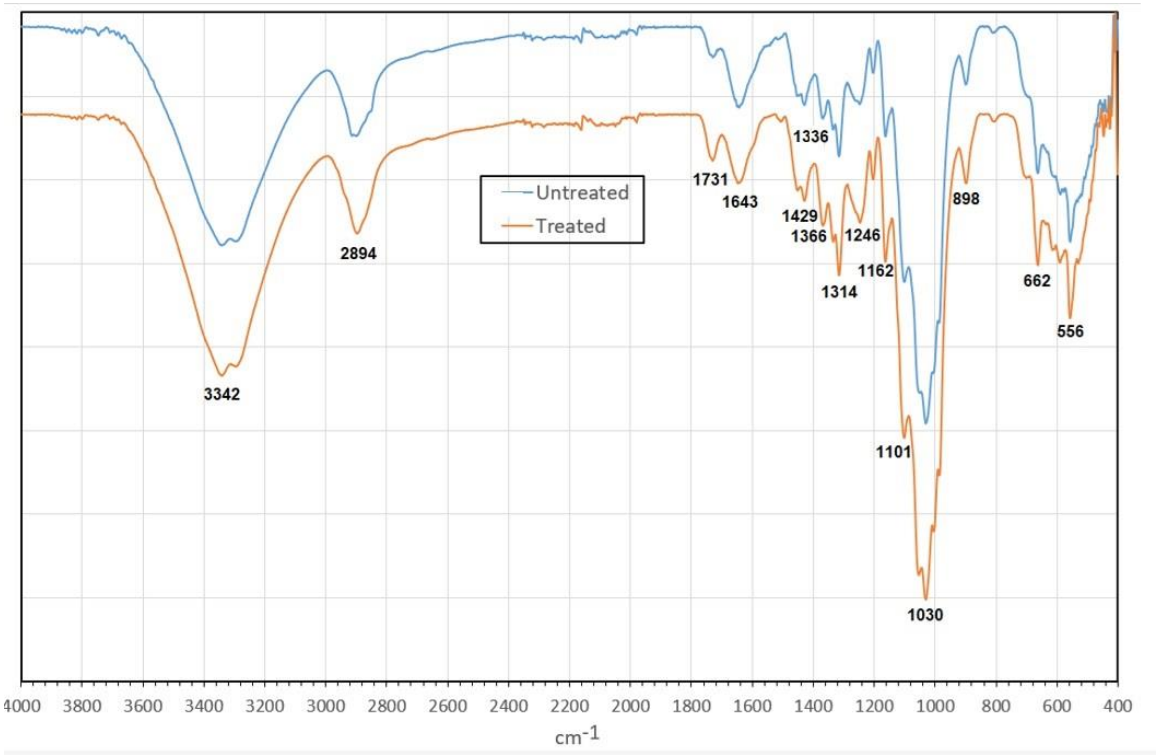

Fig. 2. FTIR analysis of untreated and SLS treated Ramie fibre

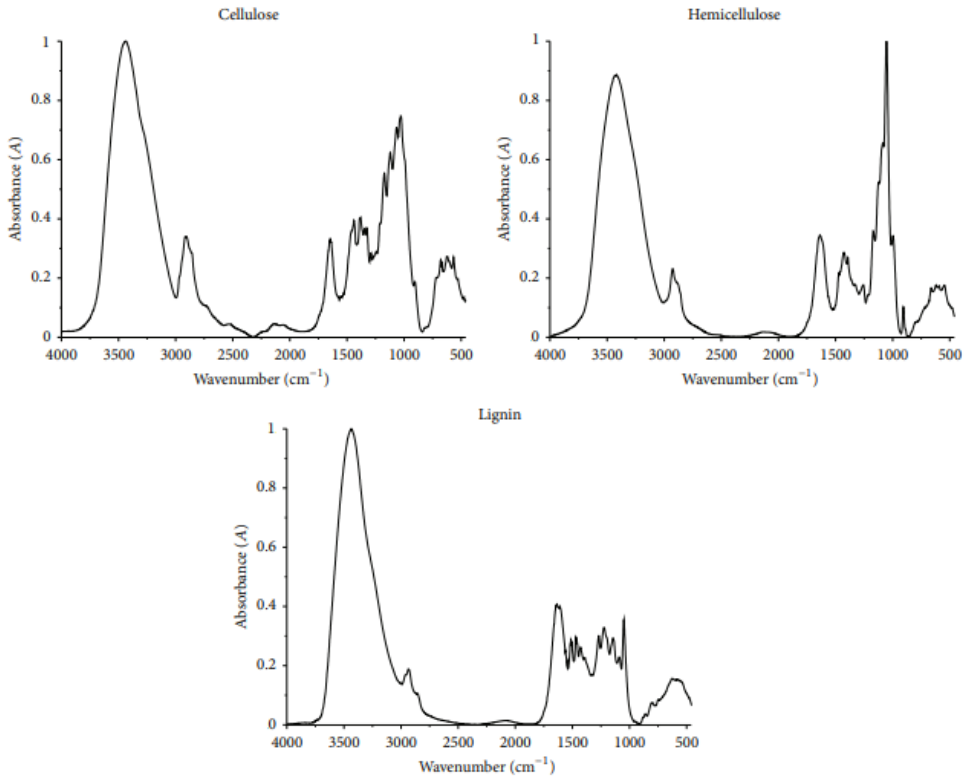

Fig. 3. Separated spectra from cellulose, hemicellulose and lignin from biomass (redrawn from (Tamanna *et al*. 2021), reproduced under Creative Commons Attribution 4.0 International License)

TGA Analysis

The thermal stability of ramie fibre was evaluated using a thermogravimetric method. Treatment delayed the weight loss, which always departs just below 100 °C, due to moisture dissolution, yet showing clearly the lower presence of loose matter, especially related to hemicellulose. Thermal stability started to be lost only around 270 °C in a process, which clearly peaked at a slightly higher temperature for treated fibres: it is then suggested that the thermal depolymerisation of the crystalline cellulose content was completed at 370 °C, and not much difference was indicated by the amount of residue witnessed at 600 °C. In Table 5 the different properties of less diffuse fibres are reported for a comparison with untreated and SLS treated ramie, which almost invariably indicated the superiority of the latter, which on the other hand provided an unusually high content of crystalline cellulose.

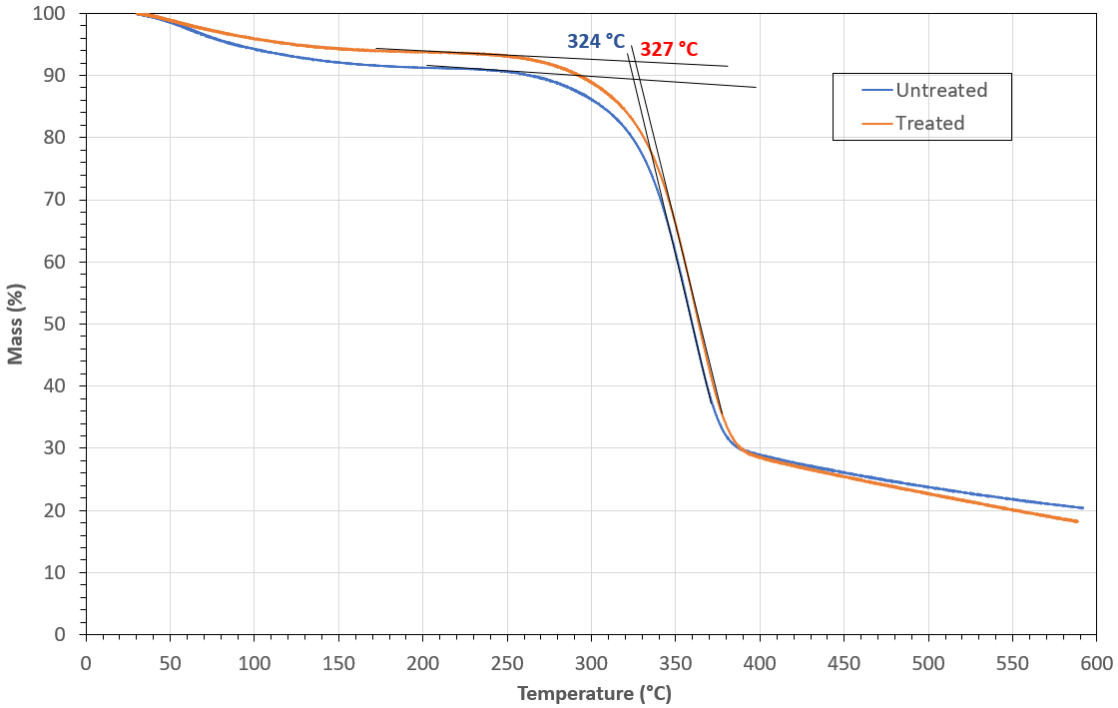

Fig. 4. Thermal analysis of untreated and SLS treated ramie fibre

SEM Analysis of Ramie

To observe the structure of ramie fibre with high resolution, SEM images were produced, in particular concentrating on untreated (Fig. 5) and SLS treated (Fig. 6) fibres. The presence of loose matter is clearly observable in the as-received fibres, as protruding from the surface (Fig. 5a), while fibril separation is already perceivable in Fig. 5b, probably due to the extraction process, which on the other side may results in obvious kinks, which are depicted in Fig. 5c. A study reported that this feature might be due to degumming process, as observed on enzymatically treated ramie fibres (Qi *et al*. 2019).

Table 5. Thermal and Crystal Properties of Other Less Diffuse Natural Fibres

Fibre Name	Thermal Stability (°C)	Tensile Modulus (GPa)	Elongation (%)	Tensile Strength (MPa)	Gauge Length (mm)	Crystal Size (nm)	Crystallinity Index (%)	Reference
Tithonia diversifolia	237	10.54	2.71-4.02	258	50	3	51.8	Selvaraj and Mylsamy (2023)
Coccinia indica	204	-	4.46	645	50	5.81	53.03	Bhuvaneshwaran *et al*. (2021)
Ageratina adenophora	244	7.69 ± 0.94	1.6 ± 0.47	123 ± 9.04	50	16.28 ± 1.55	68.98	Selvaraj *et al*. (2023a)
Ficus carica	225	-	2.72-4.91	482 ± 6	50	5.51	41.42	Selvaraj *et al*. (2023b)
Acacia concinna	280	8.41-69.61	1.38-4.24	317-1608	50	4.17	27.5	Amutha and Senthilkumar (2021)
Albizia saman	306	9.68-42.31	1.65-4.42	381-1092	75	2.85	57.69	Gopinath *et al*. (2022)

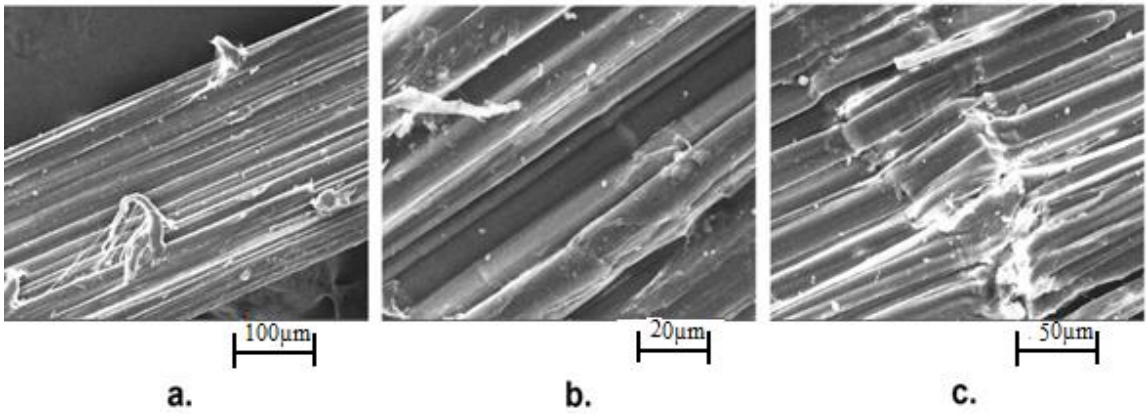

Fig. 5. SEM images of untreated ramie fibres: (a) Loose matter on the surface; (b) Fibrils partial separation; (c) Presence of kinks

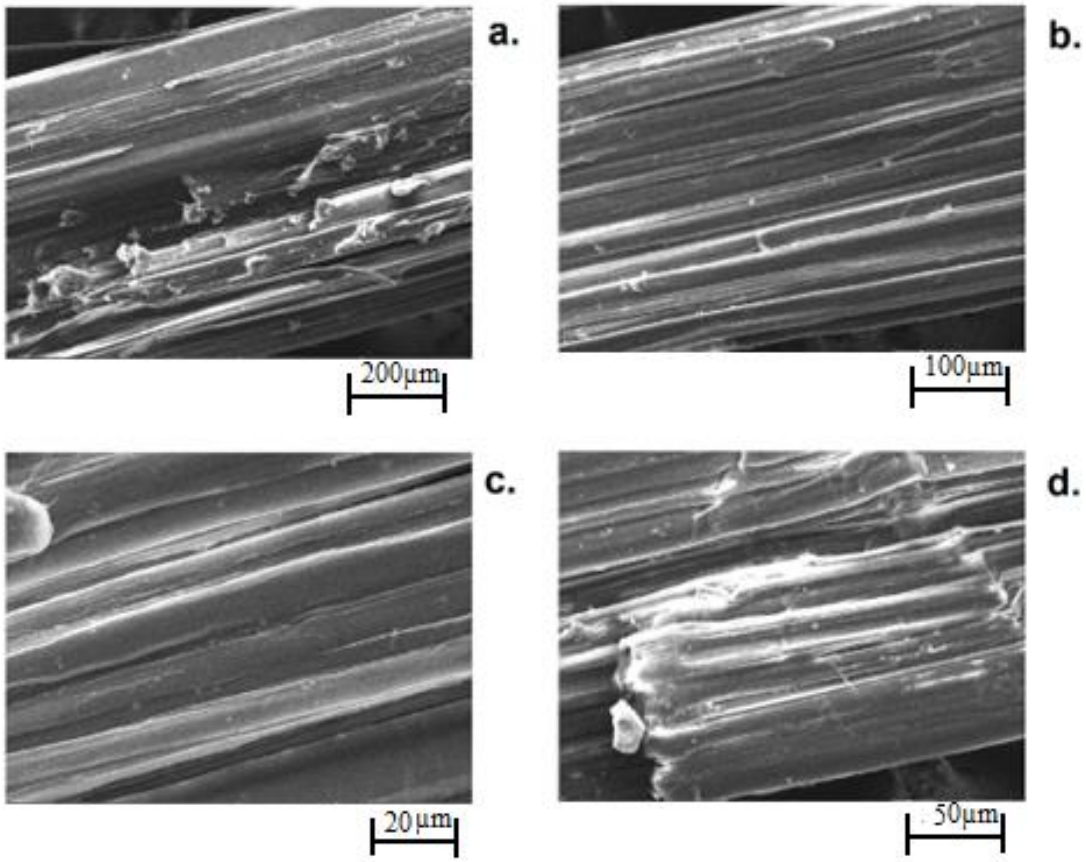

Fig. 6. SEM images of SLS treated Ramie fibres: (a) Presence of cracks; (b) Surface cleanliness; (c) Fibril partition; (d) Multi-fibril fracture

On the other hand, the surface patterns of SLS treated ramie fibres were distinctly different, indicating some presence of cracks (Fig. 6a), yet on the other side the surface appear cleaner, due to the removal of non-structural matter retained after fibre extraction (Fig. 6b), and the fibril partitions (Fig. 6c) appear comparable to what was observed in the untreated fibres. As for the multi-fibril fracture reported in Fig. 6d, it does not appear to be abrupt, therefore suggesting that SLS treatment does not result in a higher brittleness of the fibres.

CONCLUSIONS

1. This study proposed sodium lauryl sulphate (SLS) as a novel and soft treatment for ramie fibres.
2. The results indicated a limited variation in performance, yet the removal of some hemicellulose and lignin, but as a whole the preservation of crystallinity and of the highly manageable crystallite size.
3. The most promising aspects were in the relative softness obtained, with no worsening of fibrils separation. The treatment does make ramie outperform a large number of other fibres that have more recently appeared in the literature, especially in regards the comparatively high temperature degradation.
4. It is not sure whether SLS treatment would be worth pursuing in the future, and possibly more investigations on its effectiveness would be required, although it represents an alternative to the commonly applied alkali treatment with sodium hydroxide, which has become a sort of standard namely with less diffuse fibres.

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Data Availability Statement

Data are available on request from the authors.

Declaration of Conflicting Interests

The authors declared no potential conflicts of interest with respect to the research, authorship, and/or publication of this article.

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