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Arch. Metall. Mater. **68** (2023), 2, 571-578

DOI: https://doi.org/10.24425/amm.2023.142436

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EFFECT OF SURFACE CHEMICAL TREATMENT OF HIMALAYAN NETTLE AND INVESTIGATION OF SURFACE, PHYSICAL AND MECHANICAL CHARACTERISTICS IN TREATED NETTLE FIBRE

The main focus of this work was the effect of chemical alkaline treatment on Himalayan nettle fibre extraction and the characterization analysis of surface-modified nettle fibre. Nettle fibre is an eco-friendly material naturally grown in the Himalayan hills of India, and it is replacing man-made fibres. The fibres are primarily bound to each other and, in turn, to the core of the plant with pectin, lignin, and gums, which begin to break down through fungal, bacterial, enzymes and chemical treatment action. The stem from the nettle plant is fibrous and has a high-quality fibre to develop nettle yarn, which is utilized to make clothes and handicrafts, mostly aimed at generating livelihood opportunities for the rural tribe's people. This method of extraction is an effective chemical treatment for enhancing interfacial adhesion between nettle fibres and the epoxy, which is one of the significant challenges to their usage in textiles. In this paper, nettle fibres treated with chemicals such as 1% sodium hydroxide (NaOH), 0.5% sodium sulphite (Na_2SO_3), 0.05% ethylenediaminetetraacetic acid (EDTA), and 2% acetic acid (CH_3COOH). The impact of bacterial and chemical treatments on nettle fibre and untreated nettle fibre was characterized by Fourier transform infrared spectroscopy (FTIR) analysis, which is used to study the functional elements, Scanning electron microscopy (SEM) images revealed that there is a fibre breaking mechanism and cross-section of yarn twist formation, physical and mechanical characteristics were then determined for fibre tensile strength, fibre length, Young's modulus, elongation break, fineness, and moisture content.

Keywords: Alkaline treatment; Himalayan Nettle; Fibre extraction; Plant fibre; Nettle Yarn

1. Introduction

Natural fibres have been used in various environments in recent years as a result of a transition away from synthetics to natural. Bast fibres produced from plants (nettle, jute, kenaf, hemp, bamboo, roselle, flax) have benefits such as being non-toxic, easy to process, less abrasive, 100% biodegradable, and having pretty good mechanical characteristics [1]. To minimize the CO_2 emissions of materials, they could be recycled. Internal strains, poor mechanical performance of fibre, high resistance to water, and over bonding time are all symptoms of poor interfacial materials. The construction of composite from natural fibre, fibre incompatibility, and lower moisture resistance limit composites potential, and these limitations are the major challenges [2-5]. The nettle fibres are natural, biodegradable, and have renewable resources. Natural fibre requires a small quantity of energy for extraction compared to man-made fibres. As a result, nettle fibres are environmentally friendly and economically beneficial. Furthermore, the amount of water used in fibre production for the textile sector can be reduced by employing nettle fibres. Nettle yarns were employed as alternatives for shortage of cotton yarns during the period of World War I and II in Germany. Because of these advantages, stinging nettle fibres have reappeared in the textile industry, and they have become an area of research [6-8]. The high-quality fibre content of the nettle plant is 17%. The high fibre rate, maximum fibre length is 2-3 meters, it contains 72% cellulose, 11% hemicellulose, 7.5% lignin, and strong strength values make it perfect for textile use. The nettle stem is made up of woody pith and a cortex with bast filaments. The stem of nettle has been demonstrated to have a lignification range from top to bottom (required for mechanical strength at the stem base), as well as varying stages of bast fibre growth [9].

The nettle, also known as stinging nettle or Himalayan nettle, is an annual flowering plant in the Urticaceae family that also goes by the names thebvo, allo, and bichu. It is a high-fibre yielding plant that grows naturally at heights above the Himalayas between 1500 and 3000 meters. Himalayan nettle has one of the world's longest natural fibres, distinguishing it from

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other nettle or bast fibres [10,24]. Also, the nettle roots and leaves are used as traditional medicine. The Himalayan nettle was prepared and used for exquisite clothes and sailcloth. The top section of the nettle stalk has a larger percentage of fibres and a lower percentage of woody core than the bottom. The inner part of the plant's fibre was removed. The bark can be peeled and used in basketry. The inner bark was boiled overnight in a solution of water and wood ash to soften the fibres [11-14]. The fibre is sun-dried and hand-spun using a hand spindle. The tensile strength, impact strength, abrasive wear, hardness, and water absorption of the nettle fibre are good to make fabric. This study discusses the problem that it is the most common and effective method of chemical modification and it has been successfully applied to practically all natural fibres [30-32]. The local NGOs conducted better ways of harvesting, spinning, and weaving process training and income-generating programmes for the local tribes' people [4]. Water retting, dew retting, enzyme retting, and chemical retting are all methods of extrication of the embedded fibre from the nettle stalk. Chemical retting involves placing stem stalks in chemical solutions such as sodium hydroxide treatment, bleaching, acetic acid treatment, silane treatment, polymer coating, benzoyl peroxide treatment, stearic acid treatment, and cellulose powder [26]. Sodium hydroxide extraction is one of the most successful ways of eliminating lignin, pectin, and hemicellulose from nettle fibres. The alkaline treatment can help to improve mechanical interlocking, surface roughness, and fibre cellulose content. Alkaline treatment is also said to improve tensile strength and decrease water absorption in fibrous materials [27,28]. The long natural fibres are used to make the fabric, mat, yarn for textile and composites. The length of a textile fibre can either be filament or staple. Staple fibres are available in lengths of 2 to 46 cm, while filament fibres are limitless upto 2-3 meters.

In this study, fibre extraction and measurement of the properties of Himalayan nettle fibres, sodium hydroxide (NaOH), sodium sulphite (Na_2SO_3), and ethylenediaminetetraacetic acid (EDTA) were used. In comparison to other natural fibres, nettle fibres were chosen for their superior oil absorption, physical and mechanical qualities and abundant availability. Hemicelluloses were removed with an alkali solution, whereas lignin was removed with sodium sulphite and EDTA solutions. After the treatments, the fibres lost over 70% of their original hemicellulose and nearly 65% of their original lignin. Several researchers used a two-stage method to improve their outcomes (sodium hydroxide treatment followed by another chemical treatment). As a result, nettle fibres are first treated with sodium hydroxide, then with sodium sulphite and ethylenediaminetetraacetic acid in this study. Single fibre tensile tests were used to determine the fibres tensile properties. The possibility of using chemically treated cellulose and natural bast nettle fibres to make a yarn was studied. Scanning electron microscopy, Fourier transform infrared spectroscopy (FTIR), and physical and mechanical fibre characteristics are used to investigate the impact of various chemical and water treatment samples (separate and combination) on nettle fibre quality. To determine the chemicals eliminated during the extraction procedure, raw and treated fibre FTIR peaks were compared.

2. Materials and method

2.1. Source of Nettle Plant

The Hill Innovation Lead Organization in Nagaland, India provided the Himalayan nettle fibre filaments, which were 2 to 3 meters long. The fibre lignification happens quickly after flowering, producing a drop in quality, the harvest was done at full flowering in October to November nettle bark across the thickness 2-3mm approx. [15-17].

2.2. Chemicals and substance

The chemicals Sodium hydroxide pellets (low chloride) with examine (NaOH), sodium sulphite (Na_2SO_3), ethylenediaminetetraacetic acid (EDTA), water (H_2O), distilled water, acetic acid (CH_3COOH), thermometers, flasks, and beakers were usually utilised for natural bast fibre chemical alkali treatment [5,34].

2.3. FTIR characterization

Fourier transform infrared (FTIR) test was carried out on a Shimadzu Spectrophotometer used to analyze the chemical functional groups found in nettle fibres. The transmittance FTIR spectra were obtained as a function of wave number [3,18]. Each spectrum was acquired across 20 scans in the spectral range of 4000 to 700 cm^{-1} with a resolution of 4 cm^{-1} using the diamond universal attenuated total reflection attachment in the ATR mode. We investigated nettle fibre samples that had been treated with NaOH, and untreated [19,29].

2.4. Scanning electron microscopy analysis

The fibre breaking mechanism and surface of water and chemical treated nettle fibre was examined using field emission scanning electron microscopy (FESEM) JEOL JSM 6390 microscope resolution 3.0 nm with a magnification ×5 to 3,00,00 [3,40]. The fibre samples were properly placed on aluminium frames using carbon tabs, and then treated in a high-vacuum evaporative coater with a conductive layer of carbon [33].

2.5. Physical and Mechanical properties characterization

The tensile strength and elongation at break values of the nettle samples were determined using an ASTM D-3379 in accordance with the ISO 13934-1 standard. The five nettle fibre samples were prepared and used to measure the average value of fibre length, fineness, fibre diameter, tenacity, Young's modulus and moisture content [34,35]. The average percentage moisture content was computed using five nettle fibre samples. The UniStretch 250 multi- strength tester was used to measure fibre properties. The samples were heated in a 60°C oven for 24 hours. The weight of the fibre was measured as M_0 before the samples were heated. The fibre was weighed again as M_1 after 24 hours in the oven. For this test, 10 g fibres were chosen at random, each with a cut length of 100 mm [14]. We used an optical microscope to measure the nettle fibres diameter. The Shirley waste analyser equipment was used to open and analyse 100 g sample treated nettle fibre. The crosshead speed and gauge length were both kept constant at 25 mm and 2.5 mm/min [36,37].

$$\text{Moisture content}\,(\%) = \frac{M_1 - M_0}{M_0} \times 100$$

Where

M_0 – Intial weight of Nettle fibre,
M_1 – fibre weight after drying in the oven.

2.6. Water degumming nettle fibre extraction

The microbial degumming raw semi-matured nettle plant stems, as well as stems picked after flowering from germination, are dried entirely in the form of light to deep brown in colour, rigid, and connected together with sticky components. Water retting begins in an aquatic environment, with anaerobic, pectinolytic bacteria responsible for the degradation of pectin compounds and consequent fibre release. It was decided to remove the gums in order to obtain high-quality fibres [13,29]. The seeds will have dropped off, and a new plant will sprout in their place the next year. The nettle plant will only be harvested once a year in the Himalayan foothills. Nettle stalks were sun and air dried for two days before being bundled in bundles of nettle bark and stored under cover in a moderate temperature room until retting trials were conducted and processed on a small scale [12,27]. Scutching and hackling will be the next steps in separating fibre from epidermal elements completely. Nettle fibre bundles of these are wrapped and kept. Water retting removes epidermical layers, which can then be readily removed by washing with running tap water. To remove lignin and pectin materials in the nettle to use a method of decay the producing bacteria, a plastic container (50 L) was filled with well water and placed to room temperature. The fibre was separated using a water retting treatment with various time intervals and 1.0 kg of nettle plant ribbons were soaked in 30 litres of water for 7-14 days degradation shown in Fig. 1 [4].

After soaking, nettle bark was boiled with wood ash at a boiling temperature for 3-4 hours for softening and delignification. The nettle fibre ribbons were readily separated by touching and beating the boiled bark with a wooden paddle and washed multiple times with running tap water to eliminate dirt and pollutants at the end of the water degradation. To get good

Fig. 1. Nettle fibre traditional water retting yarn making process [4]

performance, the minimum ratio of nettle bark weight in kg to liquid water volume in litter should be maintained at 1:30. The processed ribbons were then removed from the non-fibrous material and sun dried for 48 hours at 37°C [10]. The Shirley trash analyzer machine was used to open the 100 g water retted fibre twice after air drying. Water retting did not affect the fibre length, allowing it to convey a greater load. SEM micrographs and tensile testing of single fibre were used to investigate the fracture mechanism of nettle plant fibres, and it was discovered that the work of fracture is dependent on the helix angle as well as the cellulose content of the fibre. A significant reduction in fibre length was seen in NaOH retted fibres, enhanced cellulose chain packing gives more crystalline cellulose and thus increased tensile strength [26-28].

2.7. Chemical treated nettle fibre extraction

The retting is a technique for removing non-cellulosic sticky materials from the cellulose portion of plant fibres. Because of their numerous applications in the chemical retting and extraction of plant fibres such as nettle, flax, ramie, hemp, kneaf and jute, they have interested the textile manufacturing industry [23-25]. The chemical (alkali) treatment of nettle fibre extraction was carried out using 1% Sodium hydroxide (NaOH), Sodium sulphite (Na_2SO_3), Ethylenediaminetetraacetic acid (EDTA), and acetic acid solution. Sodium hydroxide (NaOH) can break down lignin more quickly than cellulose and form extracellular hydrolytic enzymes that react on the lignin structure [5,31]. A trial of 50 g of nettle bark was cut into 10-15 cm long pieces and performed by incubating NaOH solution for extraction. Before the fibre chemical processing, each sample was weighed. The nettle fibre filaments were pre-treated in a tank filled with 1500 mL of water and soaked for 12 hours as part of the extraction technique. After that, nettle samples were washed in tap water 4-5 times. The nettle bark was fully dried after the pre-treatment. To get optimum performance, maintain a constant ratio of plant material weight in kg to liquid volume in litter of 1:30 by volume [3-6]. The nettle bark was treated with 1500 mL of 1% (W/V) sodium hydroxide,0.5% (W/V) sodium sulphite, and 0.05% (W/V) ethylenediaminetetraacetic acid, and the PH value was maintained at 10-10.5 in the NaOH solution (10 g sodium hydroxide per 1000 mL of water) [15-16]. The solution was mixed for around 30 seconds before being allowed to rest for 5 minutes. Fibre-containing flasks were treated at 100°C for 60 minutes, the fibre samples stirred with solution for every 10 minutes, the pliers were used to separate the fibre bundles from solution and rinsed for 15 min at 60°C. Finally, fibre bundles were washed with water under pressure for 5-8 minutes after being treated in 2.5% (V/V) CH_3COOH for 15 minutes at 60°C. The acetic acid refractive index (nD) is 1.37, with a purity of 99.90%. The wax and colour were separated by flask in boiling water at 60°C after the retting process, which removes the epidermis shown in Fig. 2. To improve the pre-treatment of nettle sample from the above technique, different experiment sets were developed [17-23]. Hackling is a combing technique that separates long and short strands while also removing any unwanted woody materials.

The fibre was dipped in water and manually soaked ten times before being retrieved and hydro dried to remove the excess water. Drying is required to prevent more fermentation. The equality of fibre quality from the bottom to the top of the plant becomes critical during the chemical treatment method.

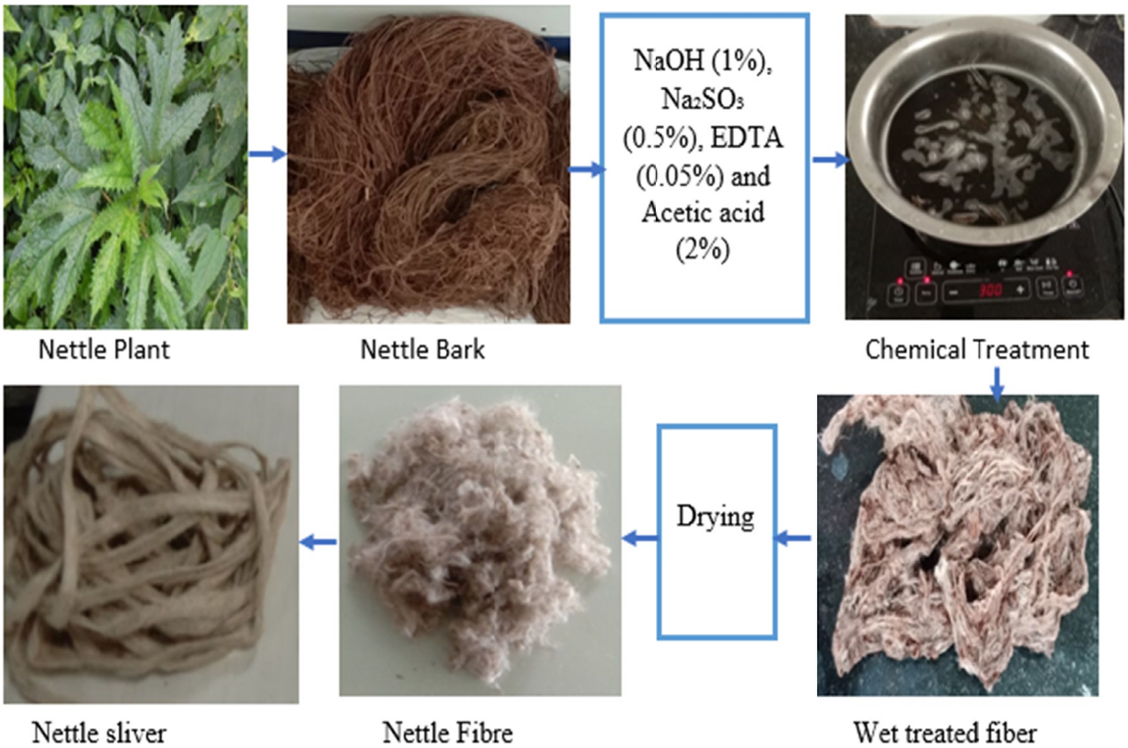

Fig. 2. Chemical treatment process on nettle fibre

As a result, the effects of morphology, thickness, and interfacial incoherency were not examined in comparing mechanical properties in this study, and the effects of NaOH were explored primarily in terms of interface characteristics such as bonding strength and defect formation. Finally, the fibre is immersed in two hot water tanks, after that fibre is air dried at room temperature for 24 hours. These chemically treated nettle fibres are considered to have the strongest fibres among due to their high cellulose content [14-18].

3. Result and discussion

3.1. Physical and Mechanical characteristics of Nettle fibre

Natural fibre hydrophilic nature makes it difficult to get effective adhesion between fibre and matrix and contributes to natural fibre's high moisture absorption, which reduces the composites product in use.

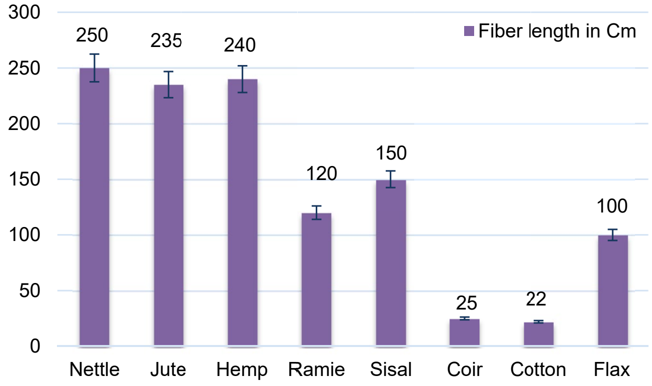

Fig. 3. Comparison nettle fibre length and other natural fibre

This difficulty, however, can be solved by using a surface chemical treatment on natural fibres. The length of the ten sample fibres measured using a simple standard meter scale length of fibre ranged from 200 cm to 300 cm, and the average fibre length is calculated as 250 cm longer than the other natural bast fibres. All natural fibres can be filament or staple fibres, as illustrated in Fig. 3 [5,31]. The average of twenty samples, the outer diameter of the nettle fibre was found to be 77-90 microns, After NaOH treatment under perfect circumstances, a substantial reduction in the 84.33 µm diameter of the nettle fibres was found. It is observed that the treated nettle fibre fineness is 4-7 dtex higher than other natural fibres [24]. Increased cellulose content gives fibres more mechanical strength, making high-strength fibres possible. Natural fibre moisture content is an important factor to consider when selecting natural fibre as a reinforcement material. This is because the moisture content of natural fibre in a composite material impacts its dimensional stability, electrical resistivity, and tensile strength [36,37]. The moisture content of the nettle fibre resulted in a range (3.5-3.7%) that was within standard limit. Tensile strength, elongation break and Young's modulus have been evaluated in water and chemical treated nettle fibre and are listed in TABLE 1. The tensile strength and modulus of nettle fibre are good and comparable to traditional water retting [19-21].

The average tensile strength of nettle fibre was 1596 MPa, whereas the tensile strength of nettle fibre is greater than other bast fibre. The nettle yarn count ranges from 30-37 Tex. This outcome is controlled by cellulose content; with more cellulose structure in the fibre contributing to the high strength qualities and average fibre thickness range (96.4-98.2 µm). This is linked to the crystalline structure of cellulose, which contributed to the plant stem's durability [35]. The good tensile strength and elongation break of nettle make it applicable in the making of composites with other fibres. The chemical treatment was used on water-treated fibres to see if it was possible to improve fineness (4.2-7.1 dtex) without impacting on mechanical properties [20-22]. In terms of fibre properties, chemically treated fibres had a considerable reduction in fibre diameter when compared to fibres obtained via water degumming method, but only when EDTA was applied. The majority of the pectin in the fibre can be destroyed to increase fineness, but only a low percentage (nearly 15%) of hemicelluloses can be eliminated without reducing fibre strength during chemical processing [26-28].

3.2. FTIR spectra Analysis of Chemical treated fibre

The FTIR spectrum results of untreated and chemically treated nettle fibre are shown in Fig. 4 and Fig. 5. The presence of many peaks in the FTIR spectra suggests that nettle fibre may contain a variety of functional groups. The high absorption

TABLE 1

Mechanical and Physical characteristics of nettle Fibre

Parameters	**Symbols**	**Raw Nettle**	**Water treated**	**NaOH Sample1**	**NaOH Sample 2**	**NaOH Sample3**
Fibre length	cm	475	250-280	225	270	300
Fibre Thickness	µm	95.5	85-90.2	86.4	77.0	90.2
Moisture content	%	4.5	3.8	3.6	3.7	3.5
Fibre fineness	dtex	3.8	4.2-6.4	4.2	5.8	7.1
Tenacity	cN/tex	35	40-48	47.6	52.1	58.2
Tensile Strength	MPa	1430	1440-1580	1510	1458	1596
Young's modulus	GPa	55-65	60-78	60	75	90
Elongation break	%	2-4	2.5-6.0	5.0	6.2	6.8

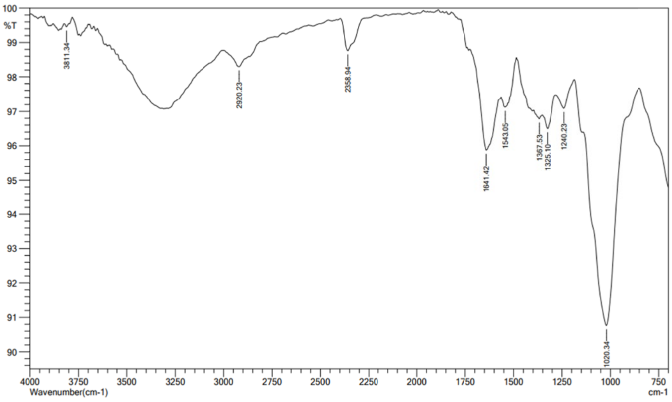

Fig. 4. FTIR spectrum (ATR mode) of untreated nettle fibre

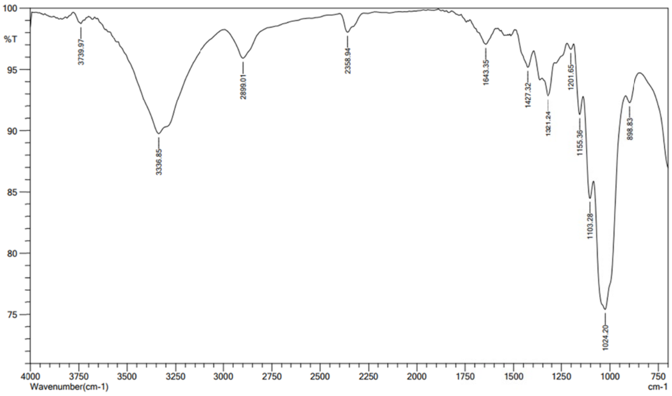

Fig. 5. FTIR spectrum (ATR mode) of Chemical treated nettle fibre

peak at 3336 cm^{-1} in chemically treated nettle fibres is linked to the cellulose molecules O-H bond stretching vibration. The C-H bond stretching groups of cellulose and hemicellulose in methyl and methylene correspond to the peaks at raw fibre 2920 and treated fibre 2899 cm^{-1} [25,40]. The high peaks at 1641 and 1643 cm^{-1} bond corresponds to the CH_2 bond of cellulose, hemicellulose, and lignin, while the peaks at 1543 and 1427 cm^{-1} assigned to the C-O absorbed water in nettle fibres peaks at 1325 and 1321 cm^{-1} corresponds to the bending of C-O bond groups of the aromatic ring in polysaccharides, while the peak at 1155 cm^{-1} represents to the -CO stretching vibration of acetyl group in hemicellulose and carbonyl groups C-O stretching lignin, whereas the peak between long U band 1020 and 1024 cm^{-1} corresponds to C-O-C stretching in cellulose and hemicelluloses [3-5]. Finally, the peak observed between 1000-700 cm^{-1} refers to stretching vibration of C-C bonds or aromatic bonds from lignin were thought to be responsible for this absorption, which indicated that as the length of oxidation extended, more lignin was extracted. As compared to raw and chemical treated nettle fibre peak data values are listed in TABLE 2. The untreated nettle fibre first peak at wave number 3276 cm^{-1} represents the O-H stretching vibration hydroxyl group [27-30].

TABLE 2

FTIR spectrum (ATR Mode) of raw and chemical treated nettle fibres

Wavelength	**Raw Nettle**	**Chemical treated**	**Spectra groups**
3600-3000	3276	3336	O-H stretching of cellulose molecules
3000-2700	2920	2899	C-H stretching of cellulose and hemicellulose
2700-2000	2358	2358	C=C stretching groups
1800-1550	1641	1643	CH_2 stretching groups in lignin
1550-1450	1543	1427	C-O stretching of the acetyl bending
1450-1300	1325	1321	Aromatic ring in polysaccharides groups C-O stretching
1200-1000	1020	1024	Antisymmetric deformation of C-O-C stretching groups
1000-700	—	898	C-C stretching vibration in cellulose

3.3. SEM analysis of nettle fibre

The Fig. 6 (A-D) illustrates the nettle fibre surface cross sectional morphology, yarn twisting cross section and breaking mechanism of water and chemically treated nettle fibres. The broken surface of a single or elementary nettle fibre that failed in tension at a displacement rate of 0.25 mm/min, as shown in cross section. As the exterior surface (which may provide the primary wall as well as the S1 layer) is peeled away, these bacterial cultures in outer layer micro breaks subsequently 48 hr retting expose some of the complex intricacies of the elementary nettle fibre [1,29]. The protrusions present in treated nettle fibres contribute to the tensile characteristics by enhancing the mechanical interlocking between the fibre and the matrix during tensile loading. Furthermore, the treated nettle fibre appears to have a rough surface, which is conducive to effective bonding at the fibre-matrix interface in composite products. The breaking surface of a single nettle fibre is drawn at a displacement rate of 1.8 mm/min [22]. The edge of fibre represents the possible breaking initiation spots, while the arrow represents the breaking propagation direction (nettle fibres can be treated with NaOH). Chemical treated fibre hemicellulose and pectin are anisotropic in their molecular structure, the surface cuticle scales on nettle fibres are quite big.

These results can be explained in terms of the internal structure of the fibre such as cell structure, microfibrillar angle and defects of fibre breaking mechanism [34]. As a result, the matrix of a flax fibre becomes extremely anisotropic, consisting of hemicellulose, lignin, and pectin (all of which have different characteristics). The existence of the chevron in the radial direction indicates that the anisotropy of the matrix may have transferred a twisting action on the fibre in roving and spinning machines [3,38]. As the crack spreads, the velocity increases, making the surface rougher. By eliminating the gum from the fibre, retting and bleaching resulted in personalization of the fibre entity and smoothening of the surface. The nettle fibres twisted the yarn in the z-direction, and as L_{hp} grew, the average twist angle dropped. The thickness of nettle fibre yarns grew as the

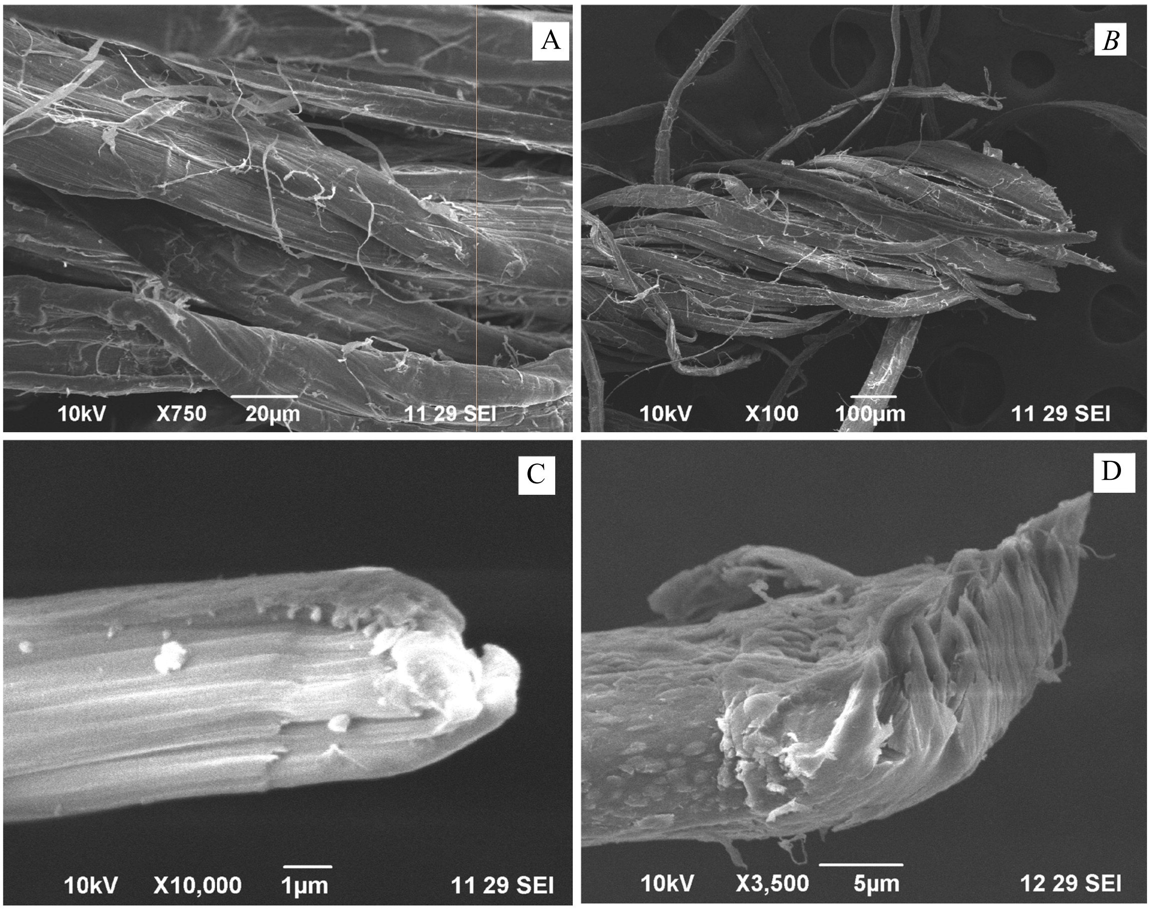

Fig. 6. SEM micrographs A) Fibre cross sectional surface analysis, B) Yarn twisting mechanism, C) NaOH treated fibre breaking mechanism D) Raw fibre breaking mechanism

distance between the ring and the disc grew because the greater the twist angle of the yarn with the same twist, the greater the twist of the fibre, resulting in a thicker yarn [24,39].

4. Conclusion

In this research work, the chemical treatment was carried out for the extraction of textile grade bast fibre from the nettle plant. To increase the fibre strength of nettle fibre and the reduction of fibre diameter, the chemicals like NaOH, Na_2SO_3, EDTA, and CH_3COOH were used. The presence of cellulose, hemicellulose, lignin, and hydroxyl (-OH) groups has been enhanced through the FTIR spectrum analysis, and the results of raw and treated nettle fibre are compared. Tensile strength, Young's modulus and elongation percentage at break of treated nettle fibres were increased by an average of 1521 Mpa, 76.66 Gpa, and 6.0%, respectively. Therefore, it is significant that fibre retting factors have a big impact on the tensile properties of fibre. The SEM results in the changes of fibre breaking mechanism, interfacial bonding characteristics, and surface area of raw fibre and treated nettle fibre. The more work done in the future related to mass production of the treated nettle fibre, to be used in carding, roving, and spinning machines to yield yarn at low cost in less time. The nettle yarn is suitable for the production of textile applications like fabric, handicrafts, bags, ropes, cushion covers, and composite materials.

Acknowledgment

This research was financially supported by the Department of Science and Technology (DST)-SEED division, India, Grant no: SEED/TITE/2018/44(G). We also thank Nagaland Science and Technology Council (NASTEC) and Hill Innovation lead organization (HILO) Nagaland, India for providing nettle plant material. The authors would like to thank BIT – Chemistry Department for providing the facility to carry out the FTIR experiment.

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