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Review on extraction, characterization, surface treatment and thermal degradation analysis of new cellulosic fibers as sustainable reinforcement in polymer composites

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ABSTRACT

Nowadays, as environmental awareness is key issue among researchers, scientific community is looking for natural materials as they are biodegradable, low cost, eco-friendly and also safe for health. Researchers and academicians have found many natural fibers and studied their properties for their sustainable applications in various possible sectors, and studies are also going on. So, in that context several natural fiber like jute, sisal, banana, pineapple, flax, hemp, kenaf, bamboo, cornstalk waste, coir, etc. have been successfully utilized as a reinforcing material in polymer composites by replacing man made synthetic fiber. Apart from traditional natural fibers, scientific community is also looking for locally available natural fibers across the globe in different geographical locations for successful reinforcement in polymer matrix. This will not only decrease burden on traditional fibers and but also at the same time it would be helpful to enrich the rural economy. Natural fiber based composites can be used in different areas such as auto motive industry, construction industry, sports industry and food industry. This study is related with extraction, characterization, surface treatment thermal analysis and activation energy of different uncommon natural fibers available at different geographical locations worldwide. The purpose of this study is to provide a comprehensive knowledge on extraction techniques, treatment methodologies, and properties of these uncommon natural fibers so that these novel materials can be utilized efficiently as a reinforcing material in different polymer matrix. Discussions on traditional natural fibers like Bagasse, Wheat straw, Coir, Pineapple, Banana etc. have been compiled extensively in various review papers but compilation on these new uncommon natural fibers is rare. Thermal analysis along with activation energy evaluation is another aspect which has been given emphasis in discussion because this is also a very important examination to evaluate the thermal stability of these natural fibers.

1. Introduction

Natural fibers are important for humans as well as for environment that is why natural fibers are used since ages because of its specific properties and applications [25]. In present time, natural fibers are replacing man-made fibers because man made synthetic fibers have high cost and environmental issues. On the other hand, natural fibers have many advantages as they are biodegradable, low in cost and eco-friendly [71,97]. Due to increasing demands and sustainable properties of natural fibers, curiosity for developing eco-friendly materials has been increased between researchers and academicians [28,94]. The availability of natural fibres is very vast because natural fibers can be simply extracted from plants that around us in the form of leaves, steams, roots and from their fruits. Some traditional fibers like jute, hemp, banana, bamboo, sisal, pineapple, coir etc., has been studied [4] and some recent studied fibers are Date palm tree, Root of banyan tree, Root of Ficus Religiosa tree, Coccinia Grandis, Tridax Procumbens, Acacia Tortills Bark, Furcracea Foetida, and many more. Due to its availability, cost effectiveness and environment friendly quality, researchers explored many domains so that natural fiber based composites can efficiently be used in many areas like aerospace industry, automotive industry [87] railway coaches, food packaging application [2] construction industry, sports, consumer

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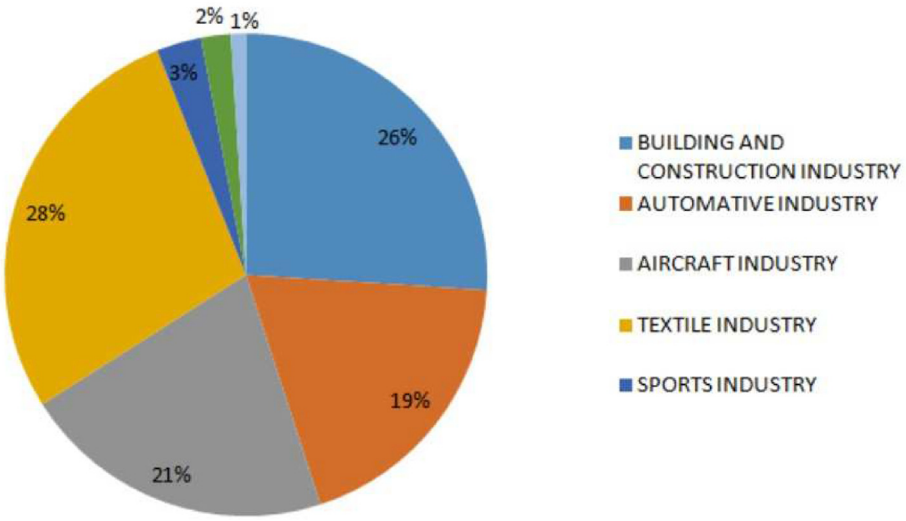

Fig. 1. Applications of natural fibers [93].

products, insulating materials (Shubash et al., 2017) and also in military application [5,38,96,101]. Natural fiber reinforced composites exhibits low cost and provide good environmental benefits [90]. The properties of natural fibers composites are comparably same as that of synthetic fibers composites such as tensile properties [103] water absorption, interlaminar shear strength, impact strength, thermal strength and tribological properties [39,83,86]. Despite of all advantages there are also some disadvantages due to its hydrophobic characteristics, but this drawback can be improved by many chemical treatments and by enhancing interfacial characteristics so that sustainability increases for environment [69]. Many efforts have been done till now for improving mechanical properties, their efforts provide good knowledge and better understanding of extraction, chemical treatment and interfacial properties [9,40,67,84,85,95,111]. So, for utilizing natural fiber in more efficient way it requires to study their characteristics, properties and behaviour. This paper presents a review of recent searched natural fiber their properties and analysing methods like XRD (X-Ray Diffraction), FTIR (Fourier transform-infrared), TGA (Thermogravimetric Analysis), SEM (Scanning electron microscopy), EDX (Energy Dispersive X-Ray), DSC (Differential Scanning Calorimetry), XPS (X-ray photoelectron spectroscopy) and NMR (Nuclear magnetic resonance)which has been used by researchers for characterizing natural fibers. Fig. 1 shows the various applications of natural fibers reinforced polymer composites. Activation energy of degradation has also been discussed with its correlation to thermal stability. To understand the activation energy and its correlation with thermal stability we need to understand the basic model equation

$$d\alpha / \ dt \ = \ k.f(\alpha) \tag{1}$$

where $d\alpha/\ dt$ is the conversion rate at constant temperature, k is rate constant. $f(\alpha)$ is model of reaction and conversion α is defined as as $\alpha = [(m_0\text{-}m_t)/(m_0\text{-}m_f)]$ where m_t, m_0, *and* m_f are weights at time t, initial and final weights of the sample respectively. By Introducing heating rate of TGA analysis $\beta = dT/\ dt$ and rate constant $k = \ \mathrm{Aexp}\left(-\frac{Ea}{RT}\right)$ where Ea is thermal decomposition activation energy which is the minimum energy required to decompose in equation (1) we get

$$dx \Big/ \ dT \ = \frac{\mathrm{A}}{\beta}\exp\left(-\frac{Ea}{RT}\right)f(x) \tag{2}$$

Various models have been used by academicians and researchers to evaluate the thermal decomposition activation energy which signifies the thermal stability.

Enormous applications are there of the natural fiber reinforced polymer composites 12. Applications of natural fibers. Automotive industry is the main sector where these composites are applied for manufacturing of lightweight, economical and biodegradable automotive parts such as interior body parts, door panel etc. Apart from these applications, construction industry also uses natural fibers composites as a a wide range of products. Various natural fiber such as rice husk, Wood flour, bagasse and flax are used for producing low weight structural walls, materials for insulation, frame of windows etc. For making building materials, natural fibers are reinforced with cement. These environment friendly composites also used in sports industries. For developing equipment such as tennis racket, seat posts flax fibers are reinforced in polymer composites. Natural fiber composites are also offering an alternate of traditional synthetic polymers for food packaging and offering excellent properties like low cost, availability, sustainability and environment friendly nature.

The objective of this study is to provide a comprehensive knowledge on various extraction techniques, surface treatment techniques, and state of art characterization techniques of these uncommon and unexplored natural fibers so that these novel materials can be utilized efficiently as a reinforcing material in different polymer matrix.

2. Extraction methods and treatment of natural fibers

Various extraction methods are available like chemical, mechanical, chemical followed by mechanical extraction, alkali, retting process and boiling extraction [11] but one of the most used method is retting extraction method, this method is very easy and also economical [16]and after this process natural fibers undergo for chemical treatment methods. Natural fibers are mainly consist of cellulose, hemicellulose, lignin [21] and wax, because of hydrophilic characteristics they provide poor compatibility to polymer matrix so for reducing hydrophilic and for improving surface roughness, surface treatments are used [27] there are various treatment methods are available like ultrasonic treatment [10], steam explosion [29,31], silane treatment [61,76], alkali treatment, isocyanate treatment, benzoylation, mercerization [22], acetylation, latex coatings, peroxide treatments and enzymatic treatment [49,112]. Chemical treatment helps for increasing adhesion between polymer matrix and natural fiber, and for enhancing mechanical strength [99]. In the above mention methods, alkali treatment method is one of the most preferable method because it is simple and economical. Extraction of Acacia Nilotica L. was done by soaking fiber in water for 20 days then washed and dried in sunlight for 7 days [42]. Sesbania Rostrata plants are taken from the turmeric field, for characterization natural fibers extracted manually from the steam of Sesbania Rostrata plants, chemical

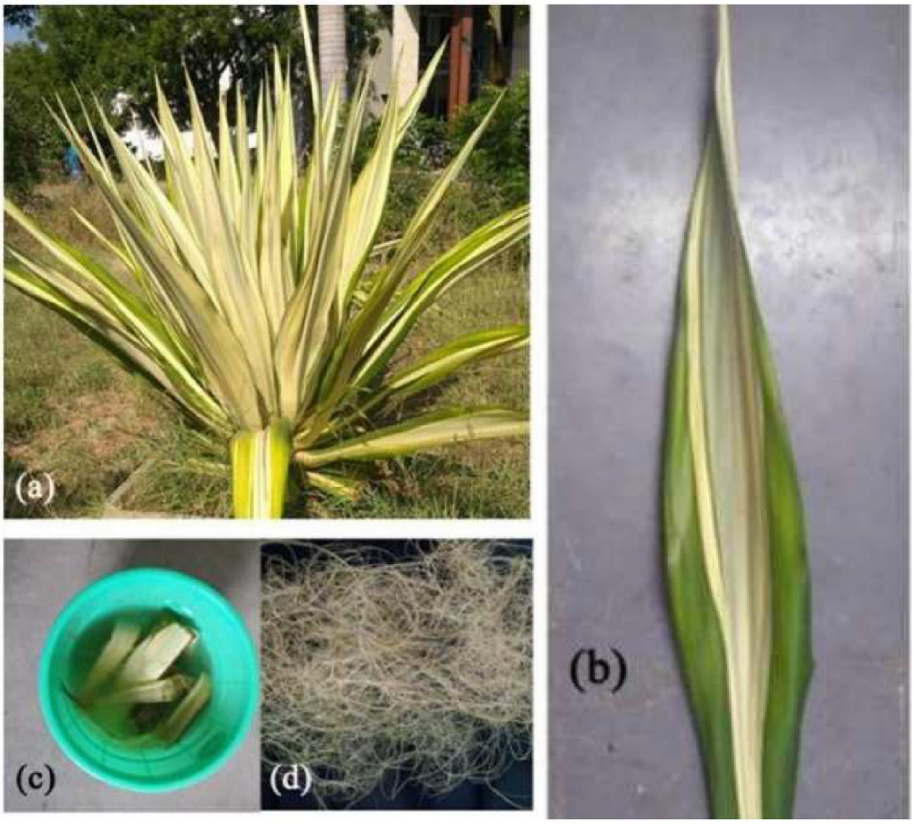

Fig. 2. Extraction process and treatment of Furcraea Foetida fiber [55].

treatment is done by soaking for 1, 3 and 5 days at room temperature in 10% sodium bicarbonate ($NaHCO_3$) and after treatment fiber is washed for removing excess $NaHCO_3$ from the surface of fiber, then washed fibers are dried at 40 °C for 1 day in the oven [78]. Yankee pineapple leaf extracted mechanically and leaf were dried in sun light for 2 days for removing water content from the fiber [65].

Extraction of leucasaspera is done by retting process manually, washed and dried in sunlight, then Leucas Aspera fibers were soaked in a silane solution of 10% concentration (98% pure Triethoxyvinylsilane'$H_2C = CHSi\text{-}(OC_2H_5)_3$'. The ratio of silane solution was taken 20:80 (v/v%) ethanol and water (v/v%) ethanol and water. After that the treated fibers were washed for removing extra silane that present on surface and oven dried at50 °C temperature for 3 h in a hot air oven [108]. Stems of Cortaderia Selloana grass collected and extraction of fiber is done by retting process and then washed and dried in day light for 72 h [33]. Extraction of Adansonia Digitata L. (Baobab) is done manually by retting process of fibers which is obtain by the stem of baobab tree. Next, fiber were washed and dried in shade [15]. Shwetark stem fiber is extracted by cutting manually after that washed and dried [77]. Fiber extraction of Red banana peduncle was done by manually then soak in solution of 5% for 1 h, then washed and dried in oven for 4 h at 80 °C [75]. Tridax fibers were also extracted manually by retting process and then washed and dried in hot air oven at the temperature of 80 °C for 2 days [107]. Saccharum Bengalense is extracted by using retting process and dried in sun light for 1 day [107]. Pigeon pea fibers were extracted by mechanical process and then fibers were washed and dried for 48 h in sunlight [43]. Microbial degradation is used for extraction of Ficus Religiosa tree fiber for 15 days, then extracted fibers were washed and sunlight was used for drying for the duration of 7 days [58]. Eleusine Indica grass and Elettaria card amomum is extracted by retting process then washed and dried for 3 days in sunlight [6,34]. Water retting process is used for extraction of Eichhornia Crassipes fiber, fibers were cleaned and dried in direct sunlight for 4 days [72]. Extraction of Ampelodesmos Mauritanicus (Diss fiber) is done by retting process and for surface treatment, soaked in 5% NaOH solution for 5 h and then washed and dried in oven for 3 h at temperature of 60 °C [70]. Extraction of Cereus Hildmannianus and Albizia Lebbeck Bark is done by water retting process the washed and dried in sun light for 7 days [52,100]. Date palm fruit fiber extracted by water retting method for 5 days and then dried in oven for 3 days at 70°-80 °C temperature [7]. Extraction of Celosia Argentea fiber is performed by manually and dried at room temperature [54]. Extraction of JaravaIchu and Stipa obtuse leaf fibers were done manually and immersed in NaOH solution for chemical treatment [63]. Rattan fiber was extracted by soaking fibers in water for 25 days and for drying fibers were put in sunlight for whole day, then 5% NaOH solution was used for treating fiber [82]. Extraction of Pongmia Pinnata l. fiber was performed by water retting process for 10 days followed mechanical processing with the help of metal comb [104]. Extraction of corn stalks was done by putting fibers in direct sunlight followed by washing with water and processed with the help of skin separator and the carried out for silane treatment by soaking fibers in Aminopropyltriethoxysilane for increasing roughness of fibers [45]. Coconut tree primary flower leaf stalk fiber extracted by cutting manually from coconut tree followed by retting process for 21 days and then fibers were extracted by hammering with wooden mallet and then washed properly and sunlight was used for drying, drying process continues to 3 days [17]. Extraction of Cardiospermum Halicababum was done by retting process, washed and dried in sunlight for 3 days [110]. Extraction of aerial root of banyan tree fibers were done by stripping the skin with the help of mechanical decorticator and then water retting process was followed, fibers the washed and dried in sunlight and treated with 5% NaOH solution for 45 min [18]. Extraction of Acacia Tortills bark fiber was done by manually followed by retting process and washed and dried and soak for 180 min in 10% and 20% NaOH solution [12]. Microbial degradation method used for extraction of Coccinia Grandis fiber then washed and dried for removing excess wetness [26]. Water retting

Table 1
Compositional analysis and densities of uncommon natural fibers.

S.No.	Fibres	Cellulose (%)	Lignin (%)	Hemicellulose (%)	Moisture Content (%)	Density (g/cm^3)	References
1	Bark of Vachellia	38.3	9.2	12.1	11	1.270	[106]
2	Bauhinia Vahlii	61–72	10–13	11–14	–	–	[73]
3	Vernonia Elaealgnifolia	–	–	–	9.18	1.22-1.43	[91]
4	Cyrtostachys renda	21.48	23.63	18.41	–	0.90	[46]
5	*Ptychosperma macarthurii*	32.62	13.13	21.64	–	0.94	[46]
6	Alkali treated Cyrtostachysrenda	45.42	18.97	20.70	–	1.06	[62]
7	5% alkali treated Calotropis gigantea	69.93	13.56	6.72	6.12	0.469	[66]
8	Acacia nilotica L.	56.46	8.33	14.14	–	1.165	[42]
9	Kigelia Africana plant	55.1 ± 0.2	11.7 ± 0.2	9.34 ± 0.08	–	–	[24]
10	Kigelia Africana fruit	61.5	20.94	12.42	10.12	1.316	[98]
11	Sesbania rostrate	72.75	15.91	8.01	4.58	1.482	[78]
12	Yankee pineapple leaf	47.74	2.44	15.98	–	–	[65]
13	Leucas aspera	58.3	4.5	8.9	4.2	1.268 ± 0.0218	[108]
14	Cortaderia selloana	53.7	10.32	14.43	7.6	1.261	[33]
15	Adansonia digitata L. (Baobab)	60.72	5.91	21.98	13	1.1041	[15]
16	Shwetark stem	69.65	16.82	0.2	8.8	1.364	[77]
17	Alkali treated red banana peduncle	79.13	12.3	–	7.51	0.85	[75]
18	Tridax procumbens	45	2.1	3.6	–	1.35 ± 0.16	[107]
19	Saccharum bengalense	53.45	1.7	31.45	2.1	1.165	[109]
20	Cajanuscajan pod fiber	59.15	20.59	10.43	5.29	1.375	[43]
21	Ficus religiosa tree	55.58	10.13	13.86	9.33	1.246	[58]
22	Eleusien indica grass	61.3	11.12	14.7	5.6	1.143	[34]
23	Elettaria cardamomum	63.12	16.5	13.7	10.93	1.470	[6]
24	*Eichhornia crassipes*	59.86	13.19	9.65	13.97	1.350	[72]
25	Ampelodesmos mauritanics (Diss fibre)	–	20.6	–	–	0.93	[70]
26	Cereus Hildmannianus	58.40	10.36	17.14	8.86	1.364	[100]
27	Date palm fruit bunch stalk	44	11.45	26	9.6	–	[7]
28	Albizia Lebbeck Bark	72.59	10.08	9.69	8.56	0.905	[52]
29	Celosia argentea	63.34	8.99	12.61	8.66	0.843	[54]
30	Jaravaichustipa obtuse	42.81 38.67	12.99 15.56	28.71 26.52	8.6 9.33	1.40 1.30	[63]
31	Alkali treated Rattan fibre	–	–	–	–	0.75	[82]
32	Pongamia pinnata	62.34	12.54	14.57	12.31	1.345	[104]
33	Treated Coconut Tree Primary Flower Leaf Stalk Fibre	71.7	11.57	6.03	9.03	1.114	[17]
34	Cardiospermum halicababum	59.82	9.3	16.75	1.9	1.141	[110]
35	Treated aerial root of banyan tree fibre	70.4	12.7	10.74	9.91	1.269	[18]
36	Acacia tortills bark fibre	61.89	21.26	–	6.47	0.906	[12]
37	Coccinia grandis	63.22	24.42	–	9.14	1.5175	[26]
38	Treated ichu	63.62	6.91	19.09	–	1.466 ± 0.0069	[102]
39	Cabuya fibre	82.84	3.50	0.94	–	1.5542 ± 0.0005	[102]
40	Areca palm leaf stalk fibre	57.49	7.26	18.34	9.35	1.09	[92]
41	Albizia Amara	65.54	15.61	14.32	9.34	1.043	[89]
42	Onion skin	41.1 ± 1.1	38.9 ± 1.3	16.2 ± 0.6	–	–	[79]
43	Onion stalk	45.5 ± 0.9	26.2 ± 1.7	25.5 ± 0.3	–	–	[79]
44	Garlic skin	41.7 ± 2.1	34.6 ± 2.4	20.8 ± 1.6	–	–	[79]
45	Garlic stalk	49.8 ± 1.8	28.6 ± 0.7	17.5 ± 0.9	–	–	[79]
46	Furcracea foetida	68.35	12.32	11.46	5.43	0.778	[52]
47	Phaseolus vulgaris	62.17	9.13	7.04	6.1	0.852	[8]
48	Pigeon pea	55.03	18.32	–	8.13	1.738	[37]
49	Napier grass	46.62	21.13	30.77	9.63	–	[3]
50	Pithecellobium dulce fibre	75.15 ± 0.26	12.14 ± 1.56	10.23	6.24 ± 1.26	0.865	[57]
51	Ethiopian kusha plant fibre	65.2–74.0	16.6–19.1	3.9-7.6	–	–	(Ravindra D [30].
52	Ceiba pentandra bark field	60.90	23.5	17.53	7.46	0.682	[41]
53	Sida cordifolia	69.52 ± 1.2	18.02 ± 4.34	17.63 ± 3.35	8.51 ± 1.2	1.33 ± 0.02	[53]

process was used for extracting kans grass and FurcraceaFoetida fiber (Fig. 2) [13,55]. Extraction of both Ichuand Cabuya fiber was done by mechanically (skin removal of fiber) and then treatment was done by NaOH, after immersing in alkali fibers were washed and dried in oven [102]. Extraction of Areca palm leaf stalk fiber was done by manually separating leaf followed by retting process then washed and dried [92]. Extraction of Albizia Amara fiber was done retting process then washed and dried [89]. Extraction of Calotropis gigantea was done manually then washed and dried in sunlight for further characterization [19]. By peeling skin, extraction of Phaseolus vulgaris was done and then washed and dried for 7 days [8]. In general we can summarize that retting is the common process used for the extraction of these fibers and a suitable treatment method (most commonly alkali)is used for inducing roughness on the surface of fibers by removing hemicellulose and lignin which ultimately helps in improved adhesion between natural fibers and polymer matrix.

3. Chemical analysis

Main purpose of chemical analysis is to evaluate the composition of cellulose, hemicellulose and lignin, etc. of these fibers. There are various methods available (TAPPI method, NREL method and many others chemical methods) for chemical analysis, Different plant's chemical compositions is evaluated by using conventional methods of chemical analysis. Some other chemical methods have also been suggested for determining chemical composition of fiber. Acacia nilotica L. and

Sesbania Rostrata fiber consists mainly cellulose (56.46%, 72.75%), hemicellulos (14.14%, 8.01%), and lignin (8.33%, 15.91%), determined by using standard TAPPI methods [42,78]. Chemical analysis of Yankee pineapple leaf is done by TAPPI method, for determining cellulose and hemicellulose TAPPI standard T20305 OS-74 is applied and for lignin T222 OS-83 was applied, corresponding compositions were 47.74% cellulose, 15.98% hemicellulose and 2.44% lignin [65]. Kushner and Hoffer methods were used for determining cellulose content (58.3%) of treated and untreated Leucasaspera fiber, fibers were crushed and 150 g of these crushed fibers were treated with 95% HNO_3 and ethanol. For measuring hemicellulose (8.9), the NFT 120–008 method was used. Klason method according to APPITA P11s-78 was used to measure lignin (4.5%) according to APPITA P11s-78 [108]. Cellulose (53.7%), hemicellulose (14.43%) and lignin (10.32%) contents that present in the Cortaderia Selloana grass were determined with the help of Kushner and Hoffer method. The Mettler Toledo xsz05 and Sartorius, model MA45 was used for measuring wax substance and moisture content (4.2%) of the Cortaderia selloana respectively, ash content of the Cortaderia selloana grass was estimated according to ASTM E1755-01 [33]. Neutral Detergent Fiber (NDF) and Acid Detergent Fiber (ADF) method is used for chemical analysis of baobab fiber, cellulose (60.72), hemicellulose (21.98%) and lignin (5.91%) was calculated [15]. Chemical composition (cellulose 69.65%, hemicellulose and lignin 16.32%) of Shwetark stem fiber is determined with the help of standard testing method. Wax, ash content, and wetness is found by Conrad techniques, ASTM E 1755-01 standards and mass loss techniques respectively [77]. Chemical analysis of Red banana peduncle, Tridax procumbens, Pigeon pea determined by Kushner& Hoffer's method and Klason method for cellulose and lignin respectively [43,75,107], moisture content analyse by electronic moisture analyser, wax is estimated by Conrad method and using ASTME1755-61 standards, ash content was evaluated [75]. Conrad method, Sartorius, model MA45, is used for determining cellulose content (53.45%) and moisture content (2.1%) of Saccharum Bengalense [107]. Kushner and Hoffer method is used for determining cellulose content (55.58%) of Ficus religiosa fiber, neutral detergent fiber method is used evaluating composition of hemicellulose (13.86%), APPITA P11s-78 approach and Conrad method is used for estimating lignin (10.13%) and wax content [58]. The chemical analysis of Eleusine Indica grass was done with the help of Kushner and Hoffer method for estimating cellulose, hemicellulose, and lignin content 55.58%, 14.7% and 11.12%, respectively, wax and ash content was determined by ASTM E1755-01 method and Conrad method respectively, The moisture content (5.6%) of Eleusine Indica grass was estimated by using electronic moisture analyser [34]. Chemical composition of Elettaria Cardamomum and Cereus Hildmannianus were analysed by Kushner and Hoffer, Klason for cellulose and hemicellulose, lignin respectively [6,100]. Cellulose (59.86%) content of Eichhornia Crassipes is analysed by Kushner and Hoffer, APPITA (P11s-78) and Neutral detergent fiber method was used to determine the lignin (13.19%) and hemicellulose content (9.65), ash content was decided by TAPPI method [72]. Acetyl bromide method was used for determining lignin (20.6%) content in Ampelodesmos Mauritanicus (Diss fiber) [70]. For analysing chemical composition (cellulose 44%, hemicellulose 26% and lignin 11.45%) of Date palm fruit bunch fiber, Ichu (63.62%, 19.09% and 6.91%) and cabuya fiber (82.84%, 0.94% and 3.50%) were determined by TAPPI method [7,102]Kurschner and Hoffer's method used for determining cellulose (72.59% and 63.34%) and hemicellulose content (9.69% and 12.61%) of Albizia Lebbeck Bark fibers and Celosia Argentea fibers were estimated by Neutral detergent fibre method, with the help of APPITA P 11s method percentage of lignin (10.08% and 8.99%) was evaluated, TAPPI and Conrad method was applied for estimating ash and wax content [52,54]. TAPPI method used for analysing chemical composition of Jarava Ichu, Stipa obtuse and Pongamia Pinnata L. fibers [63,104]. Kalson lignin method and Soxhlet apparatus was used for determining chemical composition of treated and treated Coconut tree primary flower leaf stalk fiber [17]. With the help of Kushner and Hoffer method chemical compositions like cellulose hemicellulose and lignin of Cardiospermum Halicababum, aerial root of banyan tree fiber, Acacia Tortills bark fiber were determined [12,18,110]. Chemical characterization of Coccinia Grandis fiber and areca palm leaf stalk fiber was done by Kushner, and Hoffer and Klason method for cellulose (63.22% and 57.49%) and lignin (24.42% and 7.26%) respectively [26,92]. Chemical composition of pigeon pea was evaluated by different testing methods including balance and Conrad method and corresponding composition were cellulose 55.03% and lignin 18.32% [37]. Many others natural fibers are also studied and their compositional analysis is listed in Table 1. The purpose of this analysis is to make familiar of different standard methods available for the lignocellulosic estimation of these natural fibers. This analysis will be helpful in selection of fibers to be reinforced in polymer composites across the world because this composition plays a very important role in properties of these fibers.

4. Physical analysis

For Efficient applications of natural fibers in composites it is essential to determine physical properties of these fibers too. Diameter of Sesbania Rostrata fiber(SRF) is determined by using the optical microscope at four different locations and density by Mettler Toledo XSZ05 balance method, it was reported that mean diameter of treated SRFs is decreased by 6%, 12%, and 16% with respect to increase the treatment duration for 1 day, 3 days and 5 days. And by chemical treatment void are filled and resulting in an increase in density of the treated SRFs (3.9% for 1 day, 7.9% for 3 days and 8.3% for 5 days) as compare to untreated SRFs. By Pycnometer (toluene as immersing liquid) having 0.001 g/cc accuracy (Model: Aczet/224C) density (1.268 g/cm^3) of Leucas Aspera was measured and the measurement of diameter was done with optical images (Model: Metso/VERTIMET) [108]. For measuring density of Cortaderia Selloana fiber (1.261 g/cm^3), densitometer was used which is followed by Archimedes principle [33]. Image J 1.42 q software and Origin-Pro 8.1 software was used to measure diameter in micrometer (μm), average fiber diameter of baobab fiber respectively [15]. Archimedes method with Ethanol was used for estimating density of Baobab fiber (1.1041 g/cm^3). By using standard pycnometer, density of Shwetark fiber (1.1364 g/cm^3) was estimated by ASTM D 2320-98(2003), as immersing liquid water had been used [77]. Density (0.85 g/cm^3) and diameter of Alkali treated red banana peduncle is measured by using Mettler Toledo balance method, air wedge micrometer respectively [75]. Carl Zeiss optical microscope is used for measuring diameter of Tridax Procumbens fibers [107]. For estimating density of Tridax Procumbens, Weibull analyser through Minitab 17 software is used and density was reported 1.35 g/cm^3 [107]. For measuring density of Saccharum Bengalense (1.165 g/cm^3), Mettler Toledo xsz05 was used [107]. Gas Pycnometer is used for estimating density of Cajanus Cajan, Ichu and Cabuya fiber, and observed that ichu and cabuya fiber has high density than Cajanus Cajan [43,102]. For measuring density of Ficus religiosa fiber (1.246 g/cm^3) pycnometer is used, optical microscope is used for diameter measurement [58]; Sarvankumar et al., 2019). Densitometer was used for measuring density of Eleusine Indica (1.143 g/cm^3) grass which was followed by Archimedes principle [34]. Carl Zeiss optical polarizing microscope was used for evaluating diameter of Elettaria Cardamomum, with the help of pycnometer density of Elettaria Cardamomum, Eichhornia Crassipes was measured, it was seen that Elettaria Cardamomum has more density g/cm^3 (1.470) [6,72]. With the help of pycnometer it was seen that density of Albizia Lebbeck Bark (untreated Diss fiber) and Acacia Tortills bark fiber exhibits almost equal density (approx. 0.91 g/cm^3) and aerial root of banyan tree has density 1.269 g/cm^3, Coccinia Grandis (1.5175 g/cm^3), Areca palm leaf stalk fiber (1.09 g/cm^3) and Furcracea Foetida has lowest density 0.78 g/cm^3 [12,18,26,52,88,92]. Pycnometer method was used for determining density of Cereus Hildmannianus (1.364 g/cm^3) as per ASTM D578-89 standards, immersing liquid was toluene [100]. Ultrapyc 1200-e pycnometer calculated density of JaravaIchu (1.40) which is higher than Stipa obtuse

Table 2
Mechanical and thermal properties of recently studied natural fibers.

S. No.	Fiber	Tensile strength (MPa)	Young modulus (GPa)	Elongation at break (%)	Crystallinity index (%)	References
	Barks of vachellia	33.075 ± 1.3	–	–	13	[106]
	Bauhinia Vahlii	38.96-91.28	3.08-6.9	–	48.27–61.12	[73]
	Vernonia Elaealgnifolia	259.62	37.25	6.96	–	[91]
	Cyrtostachysrenda Ptychospermamacarthurii	51.82	0.69 ± 0.18	–	44.72	[46]
		42.00	–	–	42.00	
	Alkali treated CyrtostachysRenda	–	–	–	53	[62]
	5% alkali treated Calotropis gigantea	–	–	–	39.8	[66]
	Acacia nilotica L.	–	–	–	44.82	[42]
	Kigelia Africana plant	379.28	15.68 ± 2.92	2.16 ± 0.74	59	[24]
	Kigelia Africana fruit	50.31 ± 24.7	2.17 ± 0.7	–	57.38	[98]
	Sesbania rostrate	439 ± 26.31	42.83 ± 6.92	1.65 ± 8.61	69.71	[78]
	Yankee pineapple leaf	420.3	–	–	55.22	[65]
	Leucas aspera		–	–	22.04	[108]
	Cortaderia selloana	20 ± 1.0	8.88		22	[33]
	Adansonia digitata L. (Baobab)	–	–	–	48.01	[15]
	Shwetark stem	110–533		.58-1.58	72.81	[77]
	Alkali treated red banana peduncle	650.12	–	2.90	64.57	[75]
	Tridax procumbens	25 ± 2.45	.94 ± .09	–	40.85	[107]
	Saccharum Bengalense	33 ± 1.54	–	–	44.02	[109]
	Pigeon Pea	–	–	–	20.7	[43]
	Ficus Religiosa	433.32 ± 44	5.42 ± 2.6		42.92	[58]
	Eleusine Indica	22 ± 1.0	10.75 ± 0.5		45	[34]
	Elettaria Cardamomum	294 ± 1.62	7.63 ± 2.1		36.84	[6]
	Eichhornia crassipes	–	–	–	44.32	[72]
	Ampelodesmos mauritanicus (Diss fiber)	273 ± 36	11.46 ± 2.2	–	–	[70]
	Cereus Hildmannianus	2897.47 ± 23	2.98 ± .2		40.19	[100]
	Date palm fruit bunch stalk	–	–	–	78.6	[7]
	Albizia Lebbeck Bark	270	67.45	–	52.99	[52]
	Celosia argentea	–	–	–	52.54	[54]
	Jaravaichu	–	–	–	64.31	[63]
	Stipa obtuse				43.45	
	Alkali Treated Rattan fiber	225.45	–	–	70.11	[82]
	Pongamia Pinnata l.	322	9.67	2.09	45.31	[104]
	Treated corn stalk waste	223.33 ± 41.22	7.05 ± 1.07	–	69.7	[45]
	Treated Coconut Tree Primary Flower Leaf Stalk Fiber	2008–2653	1.52–2.75	–	44.12	[17]
	Cardiospermum Halicababum	20.7 ± 1.0	–	–	32.21	[110]
	Treated aerial root of banyan tree fiber	20.45 ± 12.20	0.82 ± 0.32	1.6 ± 0.50	76.35	[18]
	Acacia tortills bark fiber	71.63	4.21	1.33	–	[12]
	Coccinia grandis fiber	424–775	26.51–123.51	–	46.09	[26]
	Treated Kans Grass	430 ± 16	9.88 ± .21	–	76	[13]
	Areca palm leaf stalk	334.66 ± 21.46	7.64 ± 1.13	–	–	[92]
	Albizia Amara	640.66 ± 213.4	–	–	63.78	[89]
	African teff straw	280–326	9.2–10.7	–	54	(G. L. Devnani and Sinha, 2018)
	Onion skin	–	–	–	–	[79]
	Onion stalk					
	Garlic skin					
	Garlic stalk					
	Calotropis gigantea	–	–	–	56.08	[19]
	Furcraea foetida	623.52 ± 45	6.52 ± 1.9	–	52.6	[55]
	Phaseolus vulgaris	–	–	–	43.01	[20]
	Pigeon pea	131	2.1	2.31	65.89	[37]
	Pithecellobium dulce fiber	654.28 ± 36	6.18 ± 1.7	–	49.2 ± 2.45	[57]
	Ethiopian Kusha Plant	805–820	–	5.5–6.1	65.18	[30]
	Ceiba pentandra bark Fiber	673 ± 14	–	–	57.94	[41]

fiber (1.30 g/cm^3) [63]. The density of treated Rattan fibers (0.75 g/cm^3) was reported more than untreated (0.62 g/cm^3) [82]. ASTM D3800 standards used for estimating density of Pongamia PinnataL. fiber (1.345 g/cm^3) [104]. [17]stated that untreated Coconut tree primary flower leaf stalk fiber (CPLSF) has more density (1.235 g/cm^3) than 2% NaOH (1.195 g/cm^3), 4% NaOH (1.153 g/cm^3) and 6% NaOH treated CPLSF (1.114 g/cm^3). Densitometer which is followed by Archimedes principle was used for determining density of Cardiospermum Halicababum fiber (1.141 g/cm^3) [110]. Using toluene as immersing method with the help of pycnometer density was evaluated of Acacia nilotica L (1.165 g/cm^3) has more density than Phaseolus vulgaris (0.852 g/cm^3) [8,42]. For estimating density of Kans grass, canola oil was used as immersing liquid and for African teff straw benzene was used as immersing liquid, observed that Kans grass has higher density 1.272 g/cm^3 than teff straw 1.15 g/cm^3. Density of Albizia Amara was determined by Mettler Toledo x 5205 balanced method has density 1.043 g/cm^3 [89]which is less than density of pigeon pea (1.738 g/cm^3) [37]. A common observations has been noticed that density and diameter are changed after chemical treatment because of different densities of cellulose, hemicelluloses and lignin and change in compositional analysis takes place.

5. Mechanical properties

Mechanical properties are an important characteristic of fibers [74]. It has been observed that treatments improve mechanical properties of

fibers. Tensile strength (33.075 ± 1.3 MPa) of bark of Vachellia fiber was determined by using universal testing machine at a 50 mm gauge length with ASTMD3522 [106]. For determining tensile strength of Yankee pineapple leaf is done by single-fiber test with crosshead speed 1 mm/min had been prepared according to ASTM D3822 and maximum load of 5 N, it was noted that 40 mm gauge length of fiber has tensile strength of 420.3 MPa [65]. With the help of INSTRON universal testing machine 5500R according to ASTM D 3822 standards, gauge length of 50 mm and 5 mm/min crosshead speed tensile properties of Shwetark fibers and alkali treated red banana peduncle are estimated [75,77]. INSTRON universal testing machine (5500R) as per ASTM D3822-07 with gauge length of 50 mm and loading rate of 0.1 mm/min was used for determining tensile properties of Saccharum Bengalense [109]. With D3379 standard method, Instron universal testing machine is used for determining tensile strength of Eleusine Indica grass where gauge length is 50 mm at crosshead speed of 1 mm/min and 10 N load shell is used [34]. Tensile testing of Elettaria Cardamomum is tested by INSTRON 5500R model universal testing machine with 2.5 kN load cell weight with crossing head speed of 0.1 mm/min along with 50 mm gauge length [6]. The tensile test of Ampelodesmos Mauritanicus (Diss fiber) were determined by an Instron universal tensile machine (Instron model 3366) load cells of 5 N was supplied, with gauge length 10 mm and moving with cross head speed of 1 mm/min [70]. Tensile testing of Cereus Hildmannianus fiber is done by Zwick/Roell testing machine with ASTM D 3822–01 standard and contain load shell of 2.5 kN for 50 mm gauge length running with crosshead speed of 2.5 mm/min ASTM D 3822–01 standard [100]. Tensile test of rattan fiber was estimated by INSTRON 3382 machine according to ASTM D3379–75 standards with crosshead speed of 0.2 mm/min and load of 1 N, gauge length was taken 50 mm [82]. With the help of INSTRON tensile machine, tensile properties of Pongamia Pinnata l, Cardiospermum Halicababum, Furcracea Foetida and Coccinia Grandis fiber were determined [26,55,104,110]. Mechanical characteristics of treated and untreated corn stalks fibers were determined with the help of MTS Criterion 40 universal testing machine [45]. Mechanical properties of coconut tree primary flower leaf stalk fiber was determined by Zwick/Roell machine with load of 2.5 kN [17]. Tensile properties of aerial root of banyan tree Zwick/Roell equipment with loadhead of 50 kN [18]. Tensile properties of Acacia tortills bark fiber was done by single fiber test followed by ASTMD2256, areca palm leaf stalk fiber followed by INSTRON 5500 R-60211 standard and Albizia Amara followed by ASTM D3379 standards [12,89,92]. Mechanical property of the fiber is the key parameter which makes it suitable or unsuitable for reinforcement in polymer composites. High tensile strength is desirable for a composite having good strength. It was observed in general that chemical treatment improves the mechanical strength of natural fibers because of reduction in hemicelulose and lignin content and increased cellulose content Tensile properties of all above mentioned and other natural fibers are listed in Table 2. These data can be helpful in selection of new natural fibers for the researchers who are looking for sustainable reinforcing materials for future.

6. Fourier transform-infrared (FT-IR) analysis

Different chemical treatments have been tried by researchers to improve the cellulose content, mechanical properties, surface roughness etc. and the chemical changes occurring in the functional groups can be identified by this method Fourier Transform-infrared (FT-IR) analysis of bark of Vachellia fiber was recorded by using PerkinElmer Spectrum and corresponding wave position were 3317 cm^{-1} confirming the presence of cellulose and lignin with OH stretching, CH stretching and CH_2 was represented by peaks 2922 cm^{-1} and 2853 cm^{-1}showing significance of cellulose and hemicellulose, at peak 1599 cm^{-1}aromatic rings was confirmed with C - C stretch, because of C - OH stretching presence of lignin was confirmed at 1051 cm^{-1} [106]. FTIR of Acacia nilotica L. was analysed by FT-IR spectrometer (SHIMADZU FTIR-8400S) and peaks were 3297 cm^{-1} due to O-H stretching of cellulose and hemi-cellulose, 2927 cm^{-1} due to C ≡ C stretching of wax, 1607 cm^{-1} due to C-O stretching of lignin, 1444 cm^{-1} due to CH_2 stretching cellulose, 1323 cm^{-1} due to C-O stretching of polysaccharides and 1022 cm^{-1} due to CO stretching of lignin [42]. For recording FTIR of Coccinia Grandis PerkinElmer spectrum RXI FTIR spectrometer was used and corresponding peaks were 3400 cm^{-1} and 760 cm^{-1} due to cellulose, 1740 cm^{-1}, 1455 cm^{-1} and 1510 cm^{-1}due to lignin, 2919.15 $cm^{-1}$and 2850.79 cm^{-1}due to CH vibration, 1423 $cm^{-1}$to 1368.42 cm^{-1}due to CH bond, 1236.21 cm^{-1} corresponds to C-O stretching showing presence of acetyl group because of lignin and 770.55 cm^{-1} due to saline [26]. FTIR of kans grass was recorded by Nicolet 6700 series FTIR spectrophotometer for untreated, 3%, 5% and 7% alkali treated fiber, in 5% alkali treatment, lignin and hemicellulose removal was observed and corresponding peaks are 1735 cm^{-1} and 1228 cm^{-1} and in 7% treatment degradation was observed. FTIR for treated and untreated ichu and cabuya fiber was analysed by Shimadzu IRTracer-100 FTIR spectrometer and various differences observed for both fiber on alkali treatment, for ichu fiber on alkali treatment peaks of lignin (C=C and C=O stretching of hemicellulose was removed and for cabuya fiber C-H and C=O vibration removed but C=C bonds remains same [102]. Chemical composition of areca palm leaf stalk fiber was confirmed by Perkin- Elmer 2000 FT spectrometer and peaks for untreated areca palm leaf stalk fiber was recorded at 3872.34 cm^{-1} due to OH stretching, 3384.49 cm^{-1}due to OH and CH stretching vibration of cellulose, 2954.63 cm^{-1} due to CH and CH_2 stretching because of hemicellulose and cellulose, 1634.78 cm^{-1} corresponded to C=O stretching of carbonyl in lignin, 1239 cm^{-1} belong to CO linkage vibration and 519.17 cm^{-1}due to C-OH vibration and also observed that on alkali treatment, different impurities like hemicellulose, lignin and wax removed [92]. FTIR of Albiazia Amara was recorded by Shimadzu spectrometer and peaks obtain at 3348 cm^{-1}due to OH stretching, 2918 cm^{-1}corresponded to CH stretching of cellulose, 2361 cm^{-1} attributed to wax constituent due to CH_2 symmetrical stretching, 1647 cm^{-1} due to C=O vibration obtained, 1385 cm^{-1} assigned to t- butyl stretch of polysaccharides and in last 1023 cm^{-1} due to C-OH stretch was observed [89]. For recording FTIR of Africanteff straw same spectrometer was used as that for Kans grass (Fig. 3) and peaks are observed for untreated straw was 3328 cm^{-1} due to OH stretching showing presence of cellulose and hemicellulose, 1738 cm^{-1} and 1226 cm^{-1} due to hemicellulose and lignin, 1010 cm^{-1} bands obtain due to ether group of cellulose and in 5% and 10% alkali treated fiber there is not much more difference in peaks as compared to untreated, removal of 1738 cm^{-1} and 1226 cm^{-1}correspond to lignin and hemicellulose was observed in 5% alkali treated straw and in 10% increment observed near peak 3328 cm^{-1} due to OH stretching vibration [13].

Calotropis gigantea was analysed by SHIMADZU FTIR spectrometer and peaks recorded at 3410 cm^{-1}due to hydroxyl group stretching, 2924 $cm^{-1}$and 2852 cm^{-1}attribute to C-H stretching of aliphatic and aromatic, 2133 $cm^{-1}$was due to wax composition present in fiber, 1635 cm^{-1}due to C=O stretch vibration, 1462 cm^{-1}confirming wavenumber for lignin, 1377 $cm^{-1}$and 1319 cm^{-1}correspond to C-H bending vibration of lignin, 1024 cm^{-1}because of C-O stretching vibration, 900 $cm^{-1}$and 650 cm^{-1}showing presence of glucose bonds [19]. FTIR of Furcraea Foetida recorded at peaks correspond to 3800-3100 cm^{-1}due to OH stretching, 2935 cm^{-1}due to CH stretching, 2844 cm^{-1} due to CH_2 vibration of hemicellulose, 1647 cm^{-1}due to OH bending vibration of hemicellulose, 1426 cm^{-1}due to CH_2 vibration of cellulose, 1023 cm^{-1}due to CO and OH stretching vibration (P. Manimaran et al., 2018). FTIR peaks of Phaseolus vulgaris fiber were 3748 cm^{-1}due to OH stretching of cellulose, 2922 cm^{-1}due to C≡C stretch of wax, 1745 cm^{-1}due to C=O stretch of hemicellulose, 1517 cm^{-1}due to stretching vibration of methyl groups, 843 cm^{-1}due to cellulose (β-glycosidic linkage) [8]. For confirming chemical composition of Pigeon pea fiber wave positions were obtain at 3417 cm^{-1}due to OH stretching of hydroxyl group, 2916 $cm^{-1}$and 2846 cm^{-1}attribute for CH stretching of cellulose content and hemicellulose content, 1704 cm^{-1}due to presence of carbonyl region, 1627 cm^{-1}due CO stretching of acetyl group, 1434 $cm^{-1}$and 1373 cm^{-1}because of CH and CO

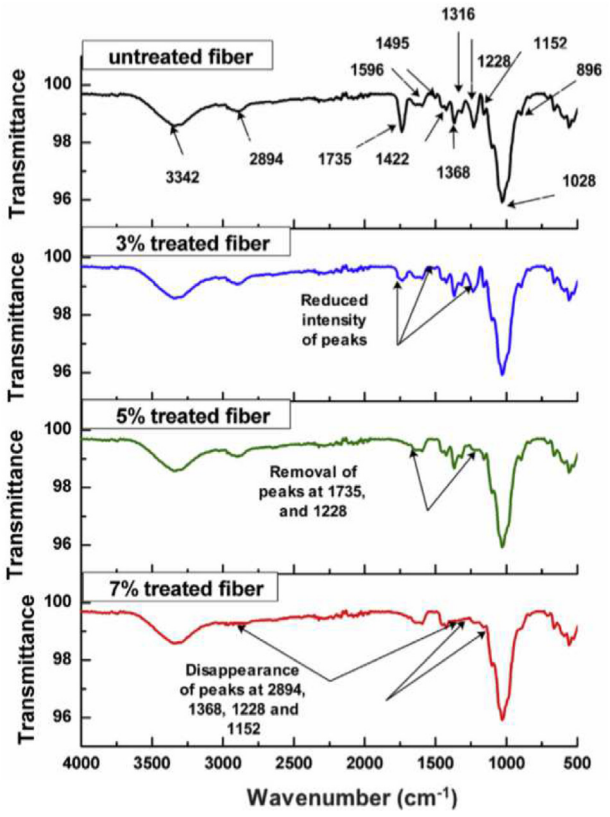

Fig. 3. FTIR spectra of untreated and treated Kans grass fiber [13].

bending vibration, 1056 cm^{-1} assigned to CO bending of alkoxy, and 570 cm^{-1} due to C-OH vibration [37]. In general we can summarize that this analysis helps to check the effectiveness of chemical treatment and the changes brought by them in the functional groups of natural fibers. Vanishing of characteristics peaks of hemicelluloses and lignin can be in generally observed after chemical treatment.

7. Morphological analysis

7.1. Scanning electron microscope (SEM)

Scanning electron microscope (SEM) is an extremely good technology for studying microstructure and surface properties of treated and untreated fibers, it is also beneficial for determining diameter and layers of fibers. With the help of SEM, removal of non-cellulosic component like wax lignin hemicelluloses can be observed [35,50]. Using SEM, information of reinforcing suitability in applications of polymer composites also studied [48]. With the help of SEM, surface morphologies was studied of Barks of Vachellia fiber was studied, author reported surface was rough of Barks of Vachellia fiber and contain some cracks [106]. SEM analysis of Sesbenia Rostrata results revels that the impurities that present on surface fiber removed after chemical treatment with $NaHCO_3$, roughness and pores also observed on treated fiber and it was concluded that Increased roughness and pores on fibers surface was giving advantage for better compatibility between polymer matrix and fiber [78]. Sem images of Malaysian Yankee fiber was taken at the magnification of 300 and 650, and it was observed that fiber contain rough and non uniform surface [65]. SEM analysis showed that untreated leucas aspera had smooth surface and reduction in smoothness of surface seen on chemical treatment with silane because of removal of undesired component like lignin, hemicelluloses and wax components [108]. With the help of Tungsten electron gun scanning microscope (W-SEM) surface characterization of Cortaderia Selloana, unwanted particles and dust was seen on fiber surface, and also concluded that surface roughness of grass will give better interfacial bonding between matrix and fiber [33]. The morphological analysis of Shwetark stem was done by CARL ZEISS MODEL V18 SEM, for analysis of fiber 5.3 mm working distance at 3 kV was maintained and to avoid charge accumulation gold sputtering was also done. It was observed that fiber consists of well packed bundles of fibers and rough surface was seen [77]. For investigating morphological behaviour of Tridax fiber Teskan, VEGA 3LMU OF Czech Republic used, it was seen that untreated had asymmetric smooth surface, and on treatment with alkali roughness enhanced and also grooves were noted [109]. For snapping high resolution images of Saccharum Bengalense grass and Eleusine Indica Grass, Teskan VEGA 3LMU OF Czech Republic was used, study of Saccharum Bengalense grass reveals that fibrils bound (because of non-cellulosic components), microcracks and porous cell was present on fiber surface. And on studying images of Eleusine Indica Grass, undesired waste materials and joints of fibrils was seen [34,107]. For investigating dimensions of Cajanus Cajan pod fibers SEM was used at magnification of 55× and 400x, diameter of 100 μm was evaluated and roughness and non-cellulosic component presence was confirmed [43]. Morphological characteristics of Ficus religiosa fiber was investigated by SEM VEGA TESKON and for avoiding undesired electron beam effects during test gold was done at 20 kV. At 5x magnification in the form of white layer, hemicelluloses can be observed on the fiber surface and requirement of medication was mentioned because of low surface roughness [58]. Elettaria Cardamomum showed closely packed structures on analysing with SEM, presence of undesired materials observed and suggested for surface treatment for removing impure particles in order to make better reinforcement of fibers in polymer matrix [6]. Better interfacial bonding of Eichhornia Crassipes fiber surface was analysed by Field emission SEM, white layer was observed, and medication of surface mentioned because of smooth surface of fiber [72]. To study the morphological characteristics of Diss fiber, fiber was treated with sodium hydroxide, acetic acid and silane. Untreated fibers showed impurities and NaOH treated sample showed roughness on the surface and clean surface was observed in case of acetic acid and silane treated sample [70]. In the SEM images of Cereus Hildmannianus fiber bundles microfibrils, rough, impurities and smooth surfaces was observed, and in enlarged transverse view of fiber, voids with primary and secondary walls that contained lignin was investigated [100]. Morpho graph of date palm fruit bunch stalk fiber showed (Fig. 4) more cracked surface and more symmetrical pattern of particle as compared to other date palm stalk fiber and mean diameter was measured 294.56 μm [7]. On capturing SEM images of Albizia Lebbeck, fiber cluster was observed and diameter was measured between 5 and 25 μm and central lumen was observed with closely packed structure [52]. In Celosia Argenteafew waxy particles were observed on capturing SEM images and at 50 μm magnification roughness was also displaced on fiber surface [54]. As per SEM images of Stipa obtuse and Jarava Ichu, dense and rough surface was observed. On alkali treatment of fibers, difference between untreated and treated morpho graph of these fibers was observed [63]. Surface morphology of rattan fiber was examined by HITACHI SU3500 SEM followed 10 nm gold coating for avoiding charge accumulation, untreated Rattan fiber were observed with smooth texture because of lignin and wax particles and removal of these undesired materials was observed in case of alkali treated rattan fibers [82]. Surface characteristics of untreated and at 1%, 5% 9%, 13% silane treated Corn stalk fibers were examined by using SEM, untreated surface contained with cellulosic and non-cellulosic material which was clearly shown in SEM images, and after silane treatment, presence of non-uniform surfaces and roughness were observed [45]. The surface characterization of CPLFSF was investigated by using SEM for both treated (2%, 4%, and 6%) and untreated fibers, on the surface of untreated fibers cracks, impurities and flaky cells were observed as shown in, removal of these undesired materials was shown in treated surfaces. In 2% and 4% NaOH concentration cells were present and on increasing concentration at 6%, provide cleaner and smooth surface [17].

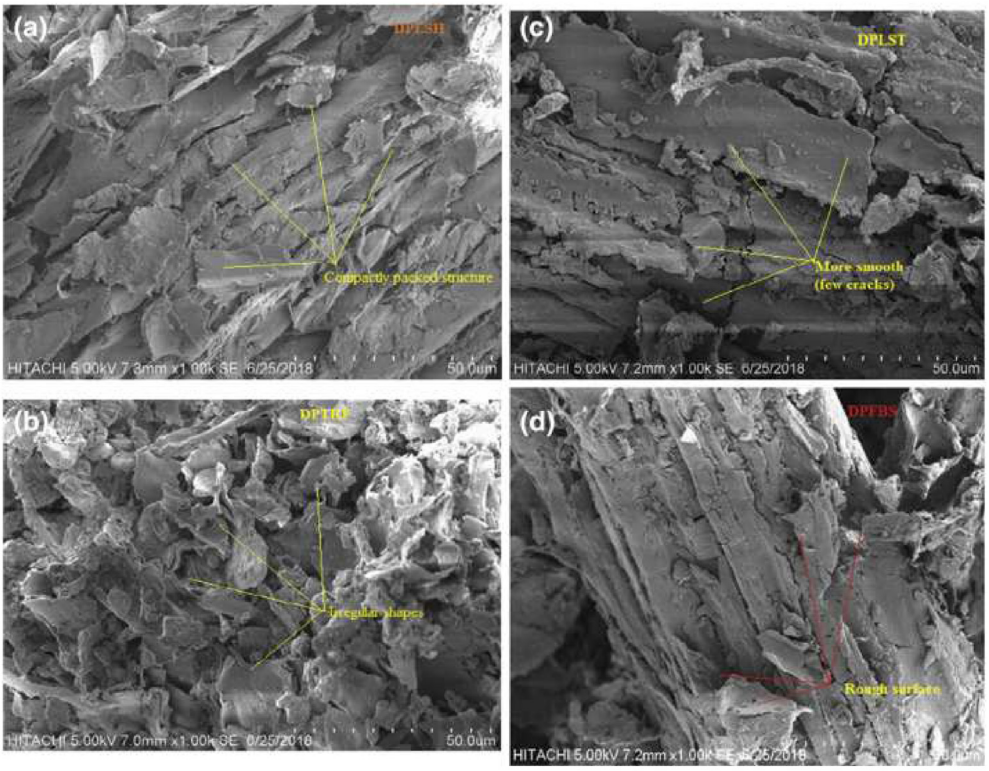

Fig. 4. SEM images of Date Palm tree various parts [7].

Impurities and roughness were noticed on Cardiospermum Halicababum fiber surface by using SEM [110]. For analyzing surface of aerial root banyan tree SEM was used. Voids, fibrils, lignin, wax, and cracks were present on untreated aerial root of banyan tree, on alkali treatment lignin, wax and impurities were removed and roughness was also improved [18]. In scanning electron microscopy of Coccinia Grandis fiber in longitudinal view, small and slanted slit type holes were identified, length of holes were 30–40 μm. and also mentioned that due to these rough surfaces, interface adhesion will be increased between fiber and matrix for composites applications [26]. Surface Morphology of Kans Grass fiber was studied by using SEM. It was seen that untreated Kans grass was packed with fiber bundles due to presence of non-cellulosic materials on the fiber surface, when images of 3% alkali treated fibers were studied it was found that roughness of surface was increased and at 5% alkali treatment, rough and fibrilled surface was identified, it was reported that 7% alkali treatment was leaded to degradation of fiber and surface was damaged due to high concentration of NaOH [13]. Optimum conditions of Ichu fiber were observed by using SEM, impurities and phytoliths were shown in untreated fibers, suggested for use in cement composite because of its better compatibility. Before alkali treatment on cabuya fibers non uniformities observed, and surface changes include clean surface and roughness were observed in both Ichu and cabuya fibers [102]. With the help of SEM, changes were observed between untreated and treated Areca palm leaf stalk fiber. Impurities was observed on untreated surface and in case of treated fiber surface, rough and porous surface was identified [92]. By using SEM, morphological characteristics was studied of African teff straw fiber and before analysis gold coating was done on samples, few impurities was seen on the untreated surface, on other hand rough and cleaner surface was identified in 5% NaOH treated fiber and in case of 10% NaOH treated fiber, collapsed surfaces and depletion of fiber was observed(G.L [14]. With 57x and 1200x magnification, SEM images were captured of Calotropisgigantea fiber and it was reported that the outer part of the surface was contained, and fibrils were detected in high magnified images [19]. These observations can elaborate that in the morphology examination of these natural fibers, scanning electron microscopy is the best analytical tool because it clearly gives the idea about induced surface roughness in these fibers after chemical treatment and surface roughness is very desired property for the fabrication of superior quality polymer composites. Higher the surface roughness, better would be adhesion between natural fibers and polymers and ultimately leads to improved composite quality.

7.2. Energy Dispersive X-Ray (EDX)

Energy Dispersive X- Ray 'EDX' is a method which is based on X-Ray and known for recognition of elemental components of material with the help of SEM [60,64]. Elemental composition analysis of Malaysian Yankee pineapple was performed by using Energy Dispersive X-Ray (EDX). On weight basis carbon was 43.84%, oxygen 46.80%, and potassium 9.36% was confirmed [65]. With the help of EDX elemental composition of Ficus Religiosa fiber was determined such as carbon 67.48%, oxygen 25.32%, chlorine 3.11%, sodium 2.71% and potassium was 1.37% [58]. By using EDX elemental analysis of Aerial Root of Banyan Tree was done and carbon 52.28%, oxygen 47.25%, calcium 0.23% and silicon was 0.04% reported [18]. In EDX spectra of Dracaena reflexa (Fig. 5) existing peaks of carbon (C), chlorine (Cl) and Oxygen (O) was determined ([56]). By using EDX establishment of elemental amount of Furcraea Foetida was done, on weight basis carbon 66.43% and oxygen was 33.7% [55]. EDX analysis basically verifies the findings of compositional analysis as this gives elemental analysis. It also helps to confirm the presence of some new element because of chemical treatment.

8. X-Ray Diffraction (XRD)

Crystallinity index and crystal size use to be analysed by using XPERT-PRO diffraction with monochromatic radiation of CuKα, corresponding wavelength was 0.154 nm and carrying current of 20 mA at 40 kV voltage. Segal empirical method and Scherrer's equation was used for determining crystallinity index (C.I.) and crystalline size [77].

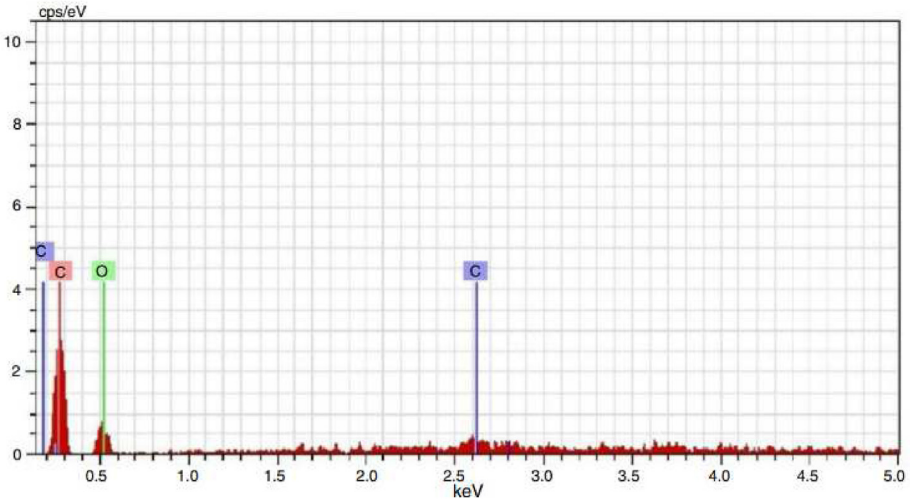

Fig. 5. EDX spectrum of Dracaena reflexa [56].

$$C.I. = (1 - I_{am}/I_{200}) \times 100$$

Where I_{200} is crystalline peak intensity and I_{am} is amorphous fraction.

And for crystalline size,

$$L = (K\lambda/\beta \cos \theta)$$

where, K is Scherrer's constant which value is 0.89, λ is radiation wavelength and β correspond to full width peak. According to XRD pattern of Shwetark stem fiber, one primary and two secondary peaks was reported, a sharp peak was found at 23.15°., which was corresponding to maximum intensity because of cellulose content in fiber and the secondary peak were noticed at 15.13° and 17.01°, attributing to amorphous content. Crystallinity index 72.81% and crystal size 13 nm was evaluated by using empirical correlation of respectively [77].

By using XRD on barks of Vachellia fiber crystallinity index was calculated 13% [106]. XRD analysis explained that C.I. value was better at 3 days' sodium hydroxide treatment and increasing treatment duration for 7 days, C.I. value was decreased by 3.7%, corresponding CI values were 69.11% and 58.41% for 3 and 5 days respectively [77,78]. By XRD, effect of chemical treatment was determined for Leucas aspera fiber, untreated CI values was 20.23%, and 22.81% reported for silane treated fiber and stated that increment in CI value was caused by chemical treatment [108]. With the help of XRD it was determined that crystalline index of Red banana peduncle was 64.57% [75]. XRD reveals that at peaks $2\theta = 21.01°$ crystalline nature was observed of Cortaderia selloana and crystalline index was estimated 22% [33]. XRD explained that untreated Tridax fiber has CI value 34.86% and after alkali treatment CI value was increased to 40.85% due to elimination of lignin and hemicellulose [109]. XRD displayed two significant peaks of Saccharum Bengalense (SB) Grass fiber, one at 16.54° ascribed to existence of lignin and pectin, and at 21.66° presence of cellulose-I and α cellulose was represented. CI value and crystal size of SB fiber was 44.02% and 58.78, respectively [107]. Cellulosic fiber from kans grass shows (Fig. 6) high value of crystallinity index 76% at 5% alkali treatment while untreated fiber has the value 53 and 3% treated fiber has the value 66% this improvement in CI value can be explained because of increased cellulose content and reduced hemicellulose and lignin [13]. Crystallinity index of various natural fibers are shown in Table 2. XRD analysis is very helpful to get the crystallinity index of these novel materials high crystallinity in natural fibers is a desired property when used as a reinforcing material in polymer matrix. Chemical treatment increase the crystalline behavior by reducing hemicelluloses. Cellulose is responsible for crystallinity of natural fibers which increases after treatment. Even if we have to select between two different untreated natural fibers for reinforcement in polymer matrix in terms of crystallinity we will select the natural fiber which has high crystalline percentage and XRD analysis facilitates this decision.

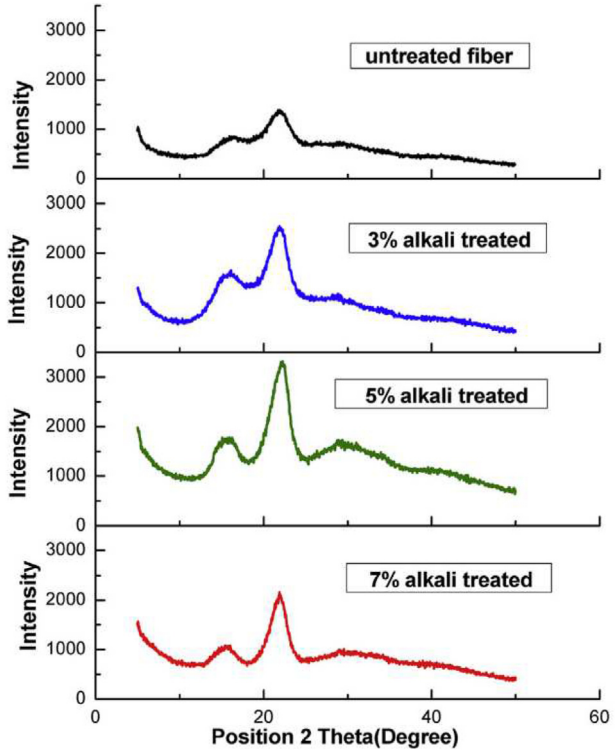

Fig. 6. X ray Diffractogram of Kans grass [13].

9. Thermal analysis

9.1. Thermogravimetric analysis (TGA) and derivative thermogravimetry (DTG)

Thermogravimetric analysis(TGA) is an important analysis to examine thermal stability and kinetic parameters [51]. With the help of TGA information we can evaluate the thermal stability of material and this understanding can help the implementation of reinforcing material in suitable application [44]. Suitable treatment not only improves the

Table 3
Thermal behaviour of various natural fiber where UT- Untreated and T- Treated.

Fiber	First stage temperature (°C)	wt%	Second stage temperature (°C)	wt %	Third stage temperature (°C)	wt%	Thermal stability (°C)	Thermal degradation (°C)	Residual weight %	Reference
Sesbania Rostrata	22–90	UT 6.96 T 5.07	214–293	UT 11.7 T 8.97	294–401	UT -71.96 T -63.72	–	UT 361 T 375	–	[78]
Malaysian yankeen pineapple	19–100	–	150–240	–	–		–	340	–	[65]
Luffa Cylinder	100	4.1	290	–	361	–	244	361	–	[74]
Leucas aspera	30–100	UT 5.1 T 2-7	203–251	UT 13 T 9	UT 325 T 387	–	–	UT 325 T 387	UT 14 T 39	[108]
Kiglia Africana	80–90	5	UT 207-256 T 109-300		300–406	–	300	–	–	[24]
Kigelia Africana fruit	40–115	9.23	212–365	70	365–620	79.64	212	340	1.127	[98]
Cortaderia selloana	100	–	165–240	–	260–375	–	–	–	–	[33]
baobab	250	8.5	250–350	48.16	350–440	37.74	250	–	5.6	[15]
Shwetark fiber	87	–	200–280	–	–	–	225	350	–	[77]
Red banana peduncle	UT 120 T 130	–	150–400	–	–	–	–	UT 320 T 333	–	[75]

mechanical strength and morphology of the natural fiber but also increase the degradation temperature and ultimately the thermal stability which can also be examined by this analysis. At the same time peak degradation temperature (temperature at which degradation is maximum) can be analysed by differential thermogravimetry. In several studies it was discussed that the leading requirement of TGA is, to identify thermal stability of the material or it can be used for estimating drying temperature [80]. By using TGA it was determined that moringa olifiera natural fiber reinforced composite is thermally more stable in comparison to PET matrix [68]. Some recently studied natural fibers's thermal stability are shown in Table 3. By using TGA thermal degradation of Sesbania Rostrata is observed in three stages, mass degradation of 5.07% and 6.96% for treated and untreated respectively was observed in temperature range of 22–90 °C, in temperature range of 214°–293 °C degradation of 8.97% and 11.7% was observed for untreated and treated respectively, and in final stage mass degradation of 63.72 for treated and 71.96% was observed in temperature range of 294°–501 °C, in DTG maximum degradability was observed at 361 °C and 375 °C for untreated and treated respectively [78]. Fig. 7 is showing detailed thermal degradation of Vernonia elaeagnifolia, various phase of decomposition and thermal stability can be clearly seen in figure [91]. With the help of Jupiter simultaneous thermal analyzer TGA of Furcraea Foetida was done, fiber sample was put in furnace and heated from 10° to 600 °C at rate of change of heat 10 °C/min with constant supplying of N_2 gas at flow rate of 20 ml/min, first phase degradation of temperature was seen between room temperature to 140 °C with weight loss of 1.81%, in second phase degradation was observed between 140 °C and 270 °C at a weight loss of 11.56%, which is attributing the removal of hemicellulose, complete decomposition of fiber was observed between 270 °C and 355 °C at a weight loss of 40.28%, a higher peak at 320 °C was seen in DTG curve [55]. TGA of Acacia Nilotica L. Plant fiber revels first range of decomposition of 30°–210 °C with 15% weight loss and the second range was 210°–355 °C due to removal of lignin and wax, in DTG curve maximum degradation was seen at 329°c the third phase of degradation was reported between 355° and 550 °C [42].

Thermal stability of Celosia Argentea plant fiber was determined using TGA, second range of decomposition was observed between 250° and 375 °C attributing hemicellulose polymerization of fiber, at 324 °C with 39.65% degradation of mass was observed [54]. TGA examined first decomposition temperature range between room temperature to 100 °C due to evaporation of moisture content, second range was observed at 230.02 °C and weight loss of 13.71% was observed at 250 °C which is

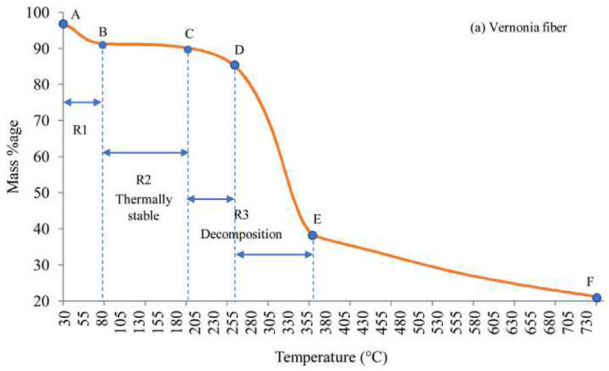

Fig. 7. TGA of Vernonia elaeagnifolia [91].

indicating hemicellulose polymerization. By using TGA second phase of degradation of Eichhornia Crassipes, Phaseolus vulgarius (250°–350 °C) and Sida Cordifolia (250°–400 °C) was investigated [8,53,72]. With the help of TGA, thermal degradation of treated Aerial root of banyan tree fiber and untreated was observed in four stage from 40 to 150 °C, 150° to 300 °C, 300° to 370 °C and final range was 370°–550 °C [18]. These compilations on TGA data and thermal stability of these uncommon natural fibers are the selection or rejection criterion for these fibers when application of high temperature is required.

9.2. DSC (differential scanning calorimetry)

Differential scanning calorimetry (DSC) is a method where "difference in heat flow rate into reference material and substance can be estimated as an element of temperature while the substance and reference are exposed to a temperature program which is well manageable" [59]. With the help of DSC technique transition temperature, polymorphic transformation and glass transition temperature can be obtained [81], The differential scanning calorimetry (DSC) analysis of Eichhornia Crassipes fiber (ECF) was done by a thermal analyser NETZSCH STA 449F3. Mass loss can be clearly seen (Fig. 8) in DSC curve of Phoenix pusilla leaves fiber [47]. By positioning 4 mg of EC fiber in aluminum crucible with 5 °C/min heating rate, fiber was heated from 30 °C to 550 °C. Fours peaks was observed in DSC curve of EC fiber curve such as 76.3 °C, 154.75 °C, 242.75 °C and 330.8 °C corresponding to removal of

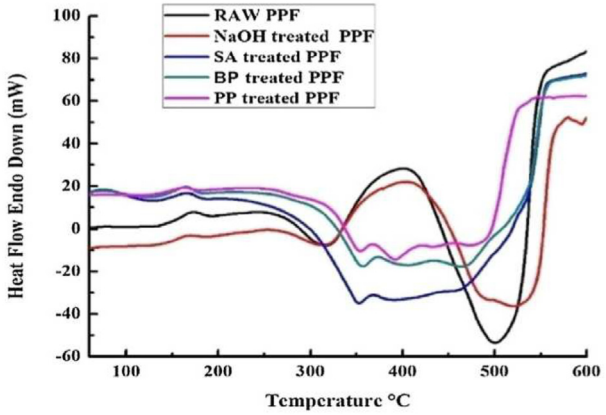

Fig. 8. DSC curve of raw and treated Phoenix pusilla leaves [47].

water, glass transition temperature, hemicellulose degradation and degradation of cellulose respectively [72]. By using DSC analyser for Acacia Niloticaa peak was observed at 104 °C which confirming the removal of water from Acacia Nilotica fiber. At 251 °C small peak was seen signifying the lignin degradation. At 364 °C visible peak was demonstrated for the removal cellulose from the fiber [42]. DSC of Ficus Religiosa fiber was done by PerkinElmer Pyrris 6 and two peaks were seen at 329 °C–76.2 °C [58]. In DSC curve of Aerial Root of Banyan Tree, two endothermic peaks were seen at 85.9 °C–364.4 °C [18]. With the help of DSC analysis the glass transition temperature and crystallinity can be evaluated and good crystallinity is directly related to improved mechanical properties of these natural fibers.

9.3. Activation energy

Thermal decomposition activation energy is another quantitative parameter which can tell about the thermal stability of natural fibers. It is minimum energy to decompose the fiber. Higher the activation energy better is the thermal stability of fiber. Activation energy can be determined by methods such as (1) Model free methods in which popular methods are Kissinger-Akahira-Sunose (KAS), Friedman and Flynn-Wall-Ozawa (FWO) [32] and (2) Graphical methods like Coats and Redfern method [80] and Broido method. In many papers Broido methods was used also in some papers KAS and FWO method were also used.

Flynn-Wall-Ozawa (FWO) is-

$$\ln \beta = c - 1.052\frac{E_a}{RT}$$

Kissinger-Akahira-Sunose (KAS) is –

$$\ln(\beta / T^2) = \ln\left(\frac{AR}{E_a \ x \ g(x)}\right) - \frac{E_a}{RT}$$

Friedman method is-

$$\ln(dx/dt) = \ln[Af(x)] - \frac{E_a}{RT}$$

Broido's equation is,

$$\ln\left[\ln\left(\frac{1}{Y}\right)\right] = \left(-\frac{Ea}{R}\right)\left[\left(\frac{1}{T}\right) + K\right]$$

where, R is universal gas constant (8.32 J/mol K), T is temperature in Kelvin and y is normalized weight.

Activation energy of Acacia nilotica L. Plant fiber and Furcracea Foetida fiber was determined with the help of Broido's equation, corresponding activation energy was 69.73 kJ/mol and 66.64 kJ/mol [42,55]. which is comparatively less. In the temperature range of 280°–375 °C activation energy of Celosia Argentea 61.393 kJ/mol was evaluated by

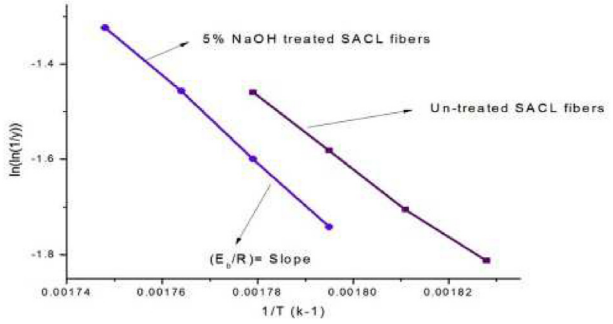

Fig. 9. Broido's plot of Saharan aloe vera cactus leaves ((A.N. and K.J., 2017).

using Broido's method [54]. Activation energy of Albizia Lebbeck bark was calculated 89 kJ/mol, between temperature 200° to 360 °C [52]. With the help of Broido's plot activation energy of Sida Cordifolia and Eichhornia Crapssipes was determined, it was observed that Sida Cordifolia (73.1 kJ/mol) has more activation energy than Eichhornia Crapssipes (66.32 kJ/mol) [53,72]. By using Broido's plot activation energy (shown in Fig. 9) of Phaseolus was calculated 64.38 kJ/mol [8]. It was reported that treated Aerial root of banyan tree fiber had more activation energy 72.45 kJ/mol than untreated 72.65 kJ/mol [18]. Similarly, activation energy of some other fibers like Cardiospermum Halicababum, Coccinia Grandis stem (82.3 kJ/mol) was evaluated [26] which is better as compared to other fibers. Similarly activation energies of untreated and chemically treated Saharan Aloevera was evaluated and it was found that alkali treatment improves the activation energy [1]. For better thermal analysis of thermal degradation of Kans grass fiber, activation energy was calculated by Flynn-Wall-Ozawa (FWO), Kissinger-Akahira-Sunose (KAS) and Friedman method, by using these methods activation energy of 5% alkali treated fiber kans grass (Fig. 10) was reported between 159 and 244 kJ/mol [13], which was significantly high as compared to untreated fiber. Similarly, activation energy of African teff straw was determined using above mentioned methods and rise in activation energy of treated teff straw was observed [14] because of removal of hemicelluloses which are thermally lesser stable as compared to cellulose.

10. ^{13}C (CP-MAS) NMR spectroscopy

With the help of ^{13}C (CP- NMR) Spectroscopy structural characteristics of Ficus religiosa fiber was recognized. Peak obtain at 63 ppm speaks to the formless idea of the cellulose substance (C6). Peaks close to the 70–80 ppm region attributing presence of C2, C3 and C5 carbon in cellulose substance. The nearness of crystalline area is guaranteed by the peak shown from 80 to 85 ppm (C4). Peaks close the 102.5 ppm range is related with presence of C1 (anomeric carbon) substance in fiber. The presence of hemicellulose contents was confirmed by peak close to 174 ppm range which is credited to COOH and CH3COO substance. Little and authorized spectrum between 108 and 170 ppm was happened to confirm the aromatic carbon component that is corresponding towards lignin content [58]. In another study, where identifying the structural feature of Furcraea foetida fiber was done by ^{13} C (CP- NMR) Spectroscopy, cellulose C1 was confirmed at peak (106.324 ppm). Excellently arranged carbons of cellulose were seen at 89.745-84.020 ppm (C4) and 66.625 ppm (C6). Peaks among 77.5, 73.1 and 73.765 ppm were apportioned to the C2, C3, and C5 cellulose carbons. The top at 22.929 ppm allotted to acetyl gatherings of hemicelluloses. The little peaks that arranged somewhere in the range of 128 and 143 ppm, that was attributing towards lignin peaks [57].

For checking structural phenomenon of Albizia Lebbeck fiber, ^{13}C (CP-MAS) NMR Spectroscopy was used and reveals that amorphous polysaccharide of Albizia Lebbeck fiber was distributed at 62.86 ppm (C6). The reverberation peaks acquired somewhere in the range of 73.64

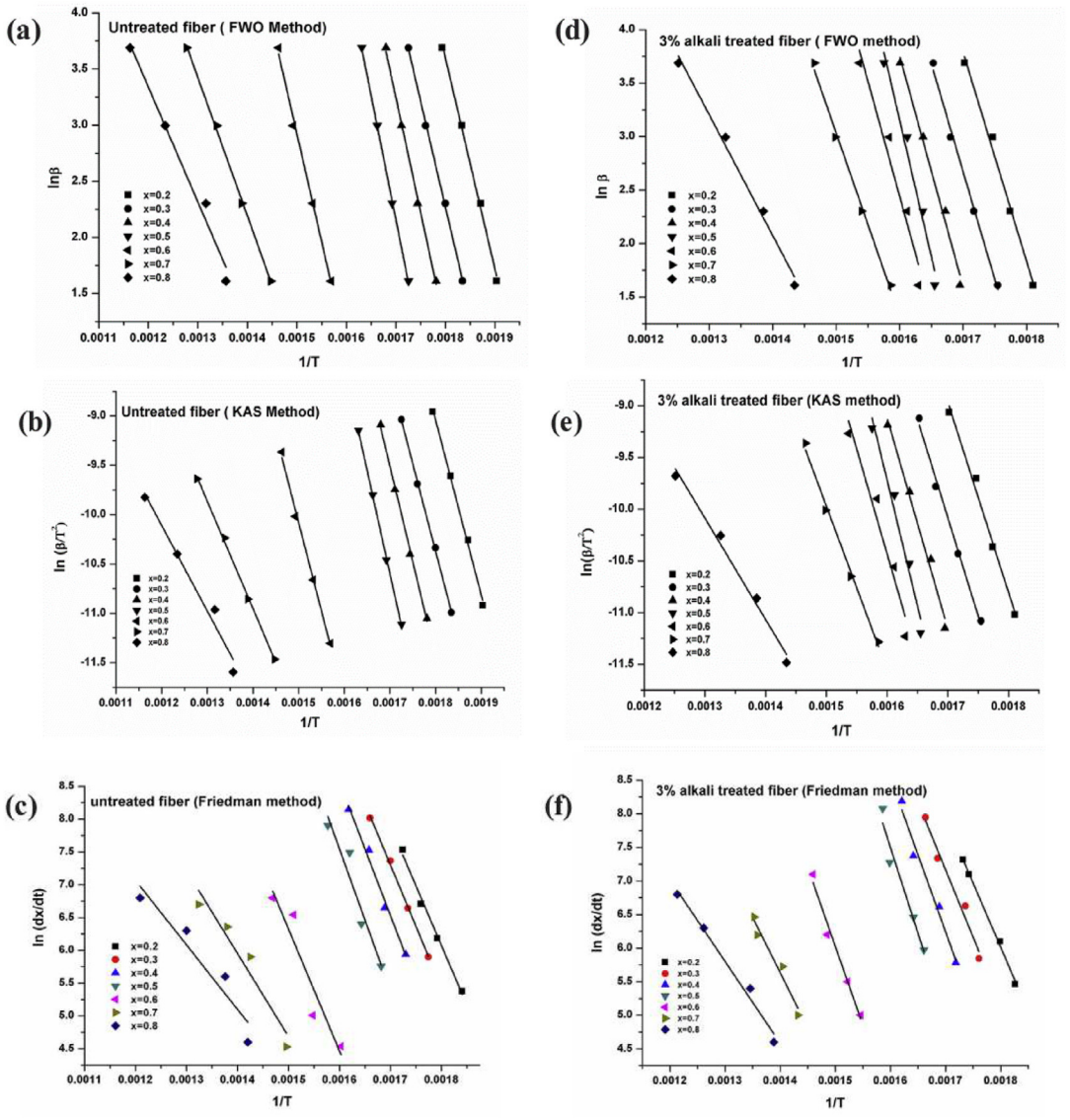

Fig. 10. Activation energies of untreated and treated Kans grass fiber using FWO method and Friedman method [13].

and 77.49 ppm were distinguished to C2, C3, and C5 cellulose carbons ring. Presence of carbon C 4 in crystalline area was approved at two accessible peaks 84.84 and 87.14 ppm. Peaks identified at 29.71 ppm doled out to acetyl gatherings of hemicelluloses. The peaks observed at 77.49 and 73.64 ppm are the most intensifying over different peaks that directed towards a large quantity of cellulose content that present in Albizia Lebbeck fiber [52]. NMR spectra of C4 regions of Tencel, Viscose and cotton are shown in Fig. 11 [23] This analysis also confirms the lignocellulosic structure of the natural fibers and verifies the chemical changes in structure after surface treatment of fibers.

11. X-ray photoelectron spectroscopy (XPS)

With the help of X-ray photoelectron spectroscopy (XPS) carbon (42.47 ± 0.14), oxygen (5.78 ± 0.19) and nitrogen (2.30 ± 1.62) was observed in chocolate brown cotton sample [105]. By using XPS elemental composition of Lotus Peduncle fiber was determined, higher peaks for carbon (80.6) and oxygen (16.3) and weaker peaks for nitrogen (1.1), calcium, iron and magnesium was observed. With the help of X-ray photoelectron spectroscopy (XPS) carbon (42.47 ± 0.14), oxygen (5.78 ± 0.19) and nitrogen (2.30 ± 1.62) was observed in chocolate brown cotton sample [105]. O/C ratio can also be attributed by this analysis

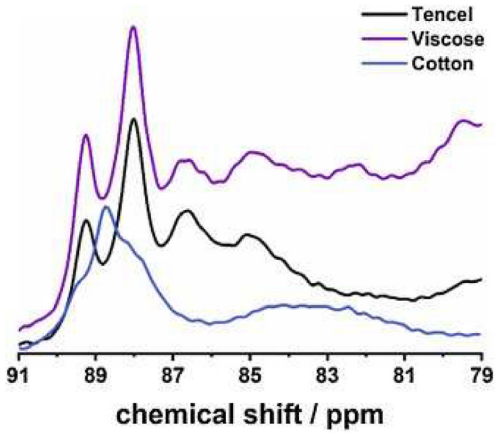

Fig. 11. NMR spectra of the C4 regions of Tencel, Viscose and cotton [23].

which characterizes the hydrophobic and hydrophilic behavior of natural fibers [36] corresponding spectra of C1s and O1s envelope for Conium maculatum fiber is shown in Fig. 12.

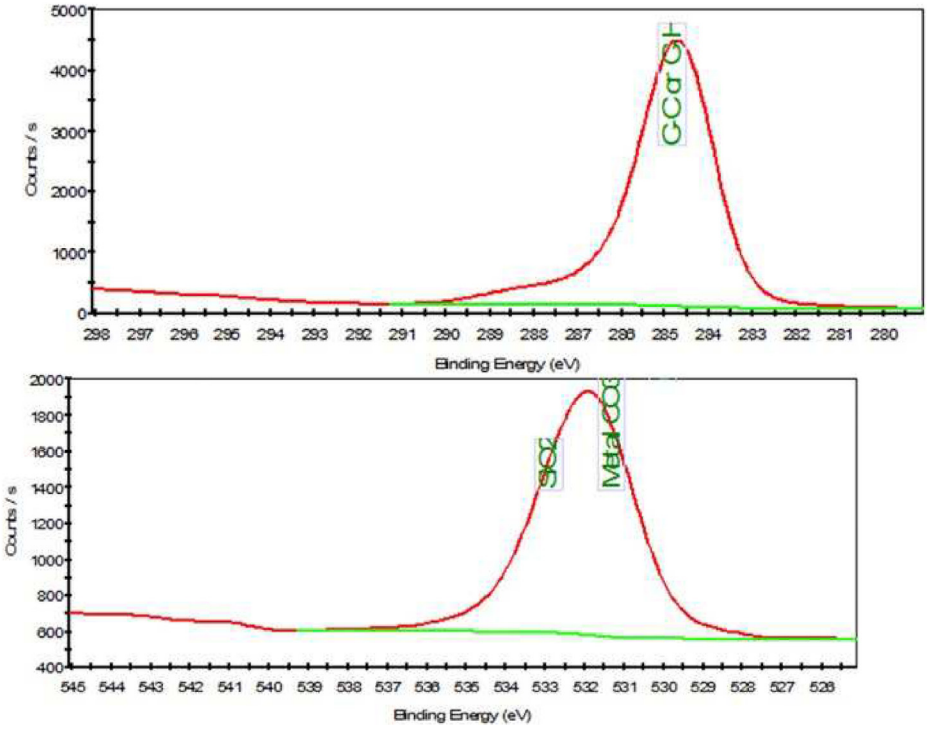

Fig. 12. XPS spectra showing the C1s envelope and O1s envelope for Conium maculatum fibers [36].

12. Conclusions

Several works have been reported on new and uncommon natural fibers, their extraction methods, treatment methods along with discussion of their properties and findings related to various characterization technique. It was observed that mainly retting process are used for extractions of natural fiber and some by mechanically or by manually, and chemical treatments are used for enhancing surface properties and mechanical properties. Chemical analysis helps to calculate cellulosic and non-cellulosic component. Density is an important property of fibers, in various literature for density measurement pycnometer was used and tensile properties also studies so that application potential of fibers can be explored in better way. Surface behaviour and morphological characteristics along with thermal characteristics were determined with the help of TGA, XRD, FTIR and SEM. FTIR analysis confirmed that most of the undesired component like lignin and hemicellulose were removed in case of treated fiber. SEM reveals that treated fiber has rougher cleaner surface. XRD indicated that treated fiber shows more crystallinity index because of high cellulosic content as compared to untreated fiber. TGA examination revealed that thermal stability of chemically treated fiber improved after surface modification with the help of chemicals. Higher Activation energies were observed in case of chemically treated fiber which also indicates better thermal stability, and in most of the literature broido's plot along with FWO and KAS methods were used for determining activation energy. Advance integral methods can also be used for determination of thermal stabilities by precise calculations of decomposition activation energies. The whole compilation in this study explores the unitized and unexplored new natural fibers, their extraction, their physical, chemical, morphological and thermal characterizations so that people working in the field of composites can utilize these new natural fibers as a reinforcement in different polymer composites. The mechanism of property improvement due to chemical treatments was also discussed with the help of state of art analysis procedures. Fabrication and characterization of polymer composites reinforced by these novel fibers can open a new direction in diverse application and commercialization of composites.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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