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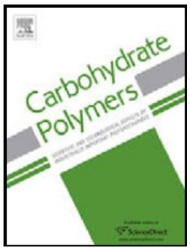

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A Comprehensive Review of Techniques for Natural Fibers as Reinforcement in Composites: Preparation, Processing and Characterization

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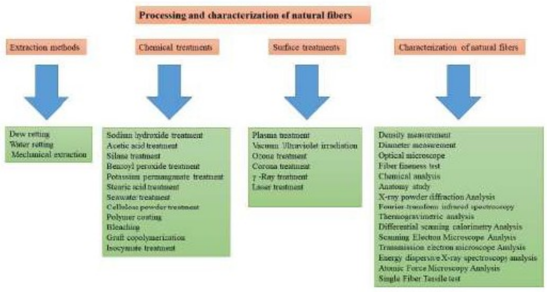

Highlights:

- Environmental concerns have fortified scientists to produce novel composites
- This review presents the commonly used processing techniques of natural fibers
- Provide a robust data base for further development of polymer composites materials
- Create a novel sustainable composite material dedicated for industrial applications

Abstract:

Designing environmentally friendly materials from natural resources represents a great challenge in the last decade. However, the lack of fundamental knowledge in the processing of the raw materials to fabricate the composites structure is still a major challenge for potential applications. Natural fibers extracted from plants are receiving more attention from researchers, scientists and academics due to their use in polymer composites and also their environmentally friendly nature and sustainability. The natural fiber features depend on the preparation and processing of the fibers.

Natural plant fibers are extracted either by mechanical retting, dew retting and/or water retting processes. The natural fibers characteristics could be improved by suitable chemicals and surface treatments. This survey proposes a detailed review of the different types of retting processes, chemical and surface treatments and characterization techniques for natural fibers. We summarize major findings from the literature and the treatment effects on the properties of the natural fibers are being highlighted.

Keywords: Natural Fiber, Extraction method, Chemical Treatment, Surface Treatment, Characterization.

1. Introduction

The high demand for environmentally friendly new materials makes the scientist develop materials from nature itself (Al-Oqla et al., 2014; Alves et al., 2010). Composite materials based on environmentally friendly and renewable materials are increasingly used, to replace conventional materials formed from composites of synthetic materials that allows reducing the greenhouse gas emissions effect (Sanjay et al., 2018; Väisänen et al., 2017). Natural fibers are environmentally friendly materials employed as reinforcement for making biocomposites, suitable for many industrial applications (Neelamana et al., 2013; Gowda et al., 2018; Siengchin, 2017). The plants from which the natural fibers are produced might be characterized as primary and secondary subject to their application (Sanjay et al., 2016). The primary plants such as jute, hemp etc., are grown only for their fibers, while the secondary plants such as banana, pineapple etc., are cultivated for their fruits, and the fibers are produced from these plants as byproducts. The commonly available natural fibers are jute, flax, kenaf, hemp, ramie (extracted from bast), sisal, pineapple, palf (extracted from leaf) cotton, kapok (extracted from seed), coir (extracted from fruit), bamboo, elephant grass, (extracted from stalk) and etc. (John and Thomas, 2008; Thomas et al., 2015, Madhu et al., 2018).

Fig. 1 presents a general configuration of a natural fiber structure and its microstructural organization covering the three main structural components, i.e. the cellulose, hemicellulose and lignin (Kabir et al., 2012; Rong et al., 2001). The plant fiber consists of a primary cell at the peripheral and three secondary walls at the interior and a lumen in the centre (Akil Rong et al., 2011; Mohanty et al., 2005). The main cell wall consists of cellulose crystalline microfibril networks arranged in a disordered manner. In the secondary walls, the cellulose crystalline microfibrils are arranged helically, with the main direction of the fiber (Krassig, 1993).

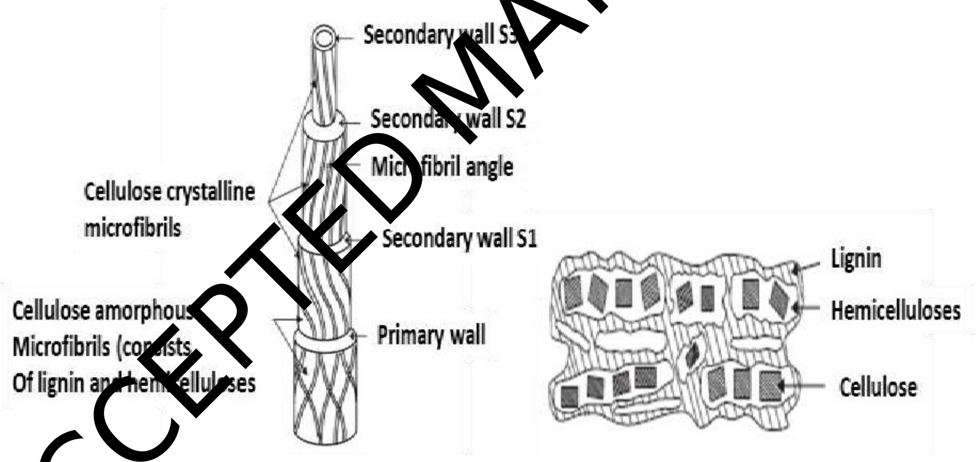

Fig. 1. Natural fiber structure and microstructural organization of the main three constituents of a natural fiber (Kabir et al., 2012).

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The main conditions that affect the fibers quality are (i) growth of the plant (plant species, location of the crop and local climatic conditions), (ii) harvest phase (age of the fibers, fiber thickness and adhesion between fibers etc), and (iii) supply phase (method of transportation, storage time and conditions) (Dittenber and GangaRao, 2012; Thakur and Thakur, 2014). Therefore, to obtain the best fibers quality, the above-mentioned parameters should be optimized for each type of different fibers. There are several advantages of using natural fibers over other synthetic fibers such as glass fibers or carbon fibers. Some natural fibers advantages are as follows : cost-effective, abundantly available, low specific weight, high specific resistance, high rigidity, renewable resource, biodegradability, smaller energy consumption for production thus low CO_2 emission, simple and environmentally friendly processing methods, excellent electrical resistance, good thermomechanical or/and relative high acoustic insulating features (Bledzki and Gassan, 1999; Jawaid and Khalil, 2011). Traditionally, natural fibers were employed in the manufacture of ropes, threads, fabrics, carpets, and cords (Reddy and Yang, 2005; Sanjay et al., 2016; Sanjay and Siengchin, 2018). Recently, these fibers were used in automotive sectors, goods packaging, low-cost housing, other civil structures and paper industries (Holbery and Houston, 2006; Siengchin, 2017). Table 1 shows some of the advantages and disadvantages of natural fibers and the potential applications of natural fibers in various sectors are summarized in Table 2. However, they are still very less exploited. To introduce natural fibers in novel applications it is essential to recognize well its preparation and processing methods. Therefore, in this review, our effort was dedicated to investigating the methods of extraction of the fibers, chemical treatments, surface treatments and characterization techniques.

Table 2. Advantages and disadvantages of natural fibers (Jawaid and Khalil, 2011; Saravana Bavan and Mohan Kumar, 2010; Sanjay et al., 2016; Sanjay and Siengchin, 2018)

Advantages	Disadvantages
Low specific weight results in a higher specific strength and stiffness than glass	Lower strength, especially impact strength
Renewable resources, production require little energy and low CO_2 emission	Variable quality, influence by weather
Production with low investment at low cost	Poor moisture resistant which causes swelling of the fibers
Friendly processing, no wear of tools and no skin irritation	Restricted maximum processing temperature
High electrical resistant	Lower durability
Good thermal and acoustic insulating properties	Poor fire resistant
Biodegradable	Poor fiber/matrix adhesion
Thermal recycling is possible	Price fluctuation by harvest results or agricultural politics

Table 2. Potential applications of natural fiberin various sectors (Ahmed et al., 2018; Puglia et al., 2005; Puttegowda et al., 2018; Sanjay et al., 2016; Sanjay and Siengchin, 2018)

Sector	**Application**
Aerospace	Tails, wings, propellers, helicopter fan blades
Automotive	Door frames, door shutters, window frame, mirror casing

Marine	Boat hulls, fishing rods
Building and Construction	Roofing sheets, bricks, furniture panels, storage tanks, pipelines
Sports & leisure goods	Ice skating boards, bicycle frames, baseball bats, tennis racket, fork, helmet, post-boxes
Electronics Appliances	Laptop and mobile cases, chip boards, projector and voltage stabilizer cover
Others	Pipes carrying coal dust, construction of weapons, textiles, industrial fans, paper and packaging

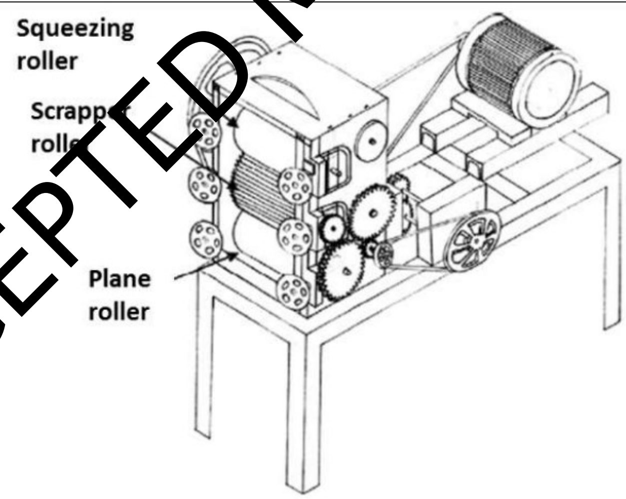
2. Extraction methods

The appropriate natural fibers extraction represents a major test faced during the processing of plant fibers. The most common methods to separate the plant fibers are dew retting and water retting process. Depending on the fiber category, these methods require approximately 14 to 28 days for the degradation of waxes, pectin, hemicellulose and lignin. To reduce long processing time, alternative methods such as mechanical extraction and chemical treatments have been introduced. In retting process, the existence of the bacteria and moisture in the plants allows to break down large parts from cellular tissues and its adhesive substances that surrounds the fibers, enabling the separation of individual fibers from the plant (Gurukarthik et al., 2018; Hyness et al., 2018). The reaction time must be carefully evaluated when using dew or water retting because excessive retting can cause difficulties for the separation of individual fibers or may weaken the fiber strength (Manimaran et al., 2018a; Paridah et al., 2011).

In the dew retting method, the stems of the plants were cut and evenly distributed in the fields, where the presence of bacteria, sunlight, atmospheric air and dew causes break down of its cellular tissues and adhesives substances that surrounds the fibers (Ahmed and Akhter, 2001; Antonov et al., 2007). Dew retting is preferred in locations having heavy night dew and warm day. This process is economical and is widely used for the industrial production of blast fibers. However, the most widely practised method is water retting process, where bunches of stems plant were submerged in water (Bacci et al., 2011; Booth et al., 2004). The water can penetrate into the central part of the stem and swells the internal cells, this results in the bursting of the outer layer of the plants (Jankauskienė et al., 2015; Pickering et al., 2007; Sisti et al., 2018). For natural water retting process, ponds, slow streams and rivers can be also utilized. Note that, the water retting process generates low-quality fibers (Amaducci and Gusovius, 2010; Lampke et al., 2005; Manimaran et al., 2018b & 2018c). Also, water retting is a long-term process and potential of water contamination prove this method to be less attractive for industrial applications (Paridah et al., 2011; Ribeiro et al., 2015; Van der Werf et Turunen, 2008).

On the other hand, the mechanical extraction process of fibers produces high-quality fibers with shorter retting time, however in respect to dew or water retting process this technique is more expensive (Paridah et al., 2011). A mechanical decorticator is presented in Fig. 2(a). The mechanical decorticator consists of a series of components (i.e. rollers, beater and etc.). The space between these rollers is 3 to 8 mm and has been maintained for the extraction of the fibers. The outer layers of the fibers such as the gums and the stems skin are eliminated by the continuous feeding of the fibers between the rotating rollers. The decorticated fibers were repeatedly washed with water and dried for 48 h in sunlight eliminating the water content from the fibers (Sathishkumar et al., 2013, Sreenivasan et al., 2011).

Recently, Bezazi et al., (2014), proposed two simple environmentally benign procedures for the extraction of Agave fibers. In the first method, the Agave leaves were buried at a depth around of 30-40 cm in the soil for three months (Fig. 3a). In the second method, the fibers were submerged in a container with water for around 10-13 days (Fig.3b). The authors observed total biodegradation of the leaves from the matrix, allowing to separate the fibers. Table 3 presents a short summary of the extraction methods comparing different process, namely the dew, water retting and mechanical extraction process.



(a)

Fig. 2 (a). Schematic diagram of a mechanical decorticator (Sreenivasan et al., 2011).

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Fig. 3 (a). Extraction of fibers from the ground when the plant is buried (Bezazi et al., 2014). (Reproduced with permission from Elsevier, License Number- 4443450240953)

Fig. 3 (b). leaves from water immersion (Bezazi et al., 2014). (Reproduced with permission from Elsevier, License Number- 4443450240953)

Table 3. Comparison between water retting and mechanical extraction process.

Extraction Methods	Dew Retting	Water Retting	Mechanical Extraction
Description	Gathered samples plant stems are spread evenly on the grassy fields, to receive a combined action of bacteria, sunlight, atmospheric air and dew	Plant stems needs submersed in water (rivers, ponds, or tanks) and checked periodically (microbial retting)	Fibers hammering are separated with a hammer mill or/and decorticator

	that causes break down of its cellular tissues and adhesive substances that surrounds the fibers		
Duration	Two to three weeks, depending on the climatic conditions	7-14 days	Depending on production fibers
Advantages	Common in areas in locations having heavy night dew and warm day and with limited water resources.	The fibers produced are uniform and of higher quality	It produces large quantities of short fibers in a short time
Disadvantages	The obtained fibers are darker in colour and are of poor quality. For dew retting process, the agricultural lands need to be occupied for several weeks, and also the obtained fibers are contaminated with soil and fungi.	High cost, environmental concerns and inferior fibers quality, but better than fibers obtained through dew retting process. Requires high water treatment maintenance	High cost and acceptable quality fibers

Reference	Ahmed and Akhter, 2001; Antonov et al., 2007; Bacci et al., 2011; Booth et al., 2004; Jankauskienė et al., 2015; Paridah et al., 2011; Pickering et al., 2007; Sisti et al., 2018	Amaducci and Gusovius, 2010; Lampke et al., 2005; Manimaran et al., 2018b & 2018c; Paridah et al., 2011; Sisti et al., 2018, Ribeiro et al., 2015; Van der Werf et Turunen, 2008	Paridah et al., 2011, Sathishkumar et al., 2013, Sreenivasan et al., 2011

3. Chemical treatments

The natural fibers hydrophilic features and the polymer matrix hydrophobic characteristics are the main fundamental problems of using natural fibers as reinforcement for the polymer composites. However, by using a chemical treatment on the natural fibers allows reducing its fibers hydrophilic features (Bezazi et al., 2014; Li et al., 2007; Mwaikambo and Ansell., 2002). The most important chemical treatments used in order to reduce the hydrophilic characteristics of a natural fiber are presented as follows:

3.1 Sodium hydroxide (NaOH) treatment

The natural fibers treatment by NaOH is the most common. This covers four different practices, such as 1) a constant concentration of NaOH for a constant period of time (Reddy et al., 2009a; Senthamaraikannan and Kathiresan, 2018; Shanmugasundaram et al., 2018), 2) using different NaOH concentration for a constant period of time (Reddy et al., 2009b), 3) keeping a constant NaOH concentration for different time periods (Arthanarieswaran et al., 2015; Rajkumar et al., 2016; Reddy et al., 2013) and 4) using different NaOH concentrations for different time periods (Saravanakumar et al., 2014a). The second and third practice methods are the most common treatments to determine the optimal conditions for natural fiber modification. At different NaOH concentrations with a constant period of time, the fibers should be treated with 2% to 5% (w / v) NaOH solutions keeping the temperature around 23^0C for a constant time, maintaining a proportion of the liquor 20:1 that permits to eliminate hemicellulose and other fatty materials. After this chemical practice the fibers are neutralized, cleaned and dried (Reddy et al., 2009b; Liu et al., 2018). At constant NaOH concentration with different time periods, the natural fibers are treated in 5% (w / v) NaOH solution (generally 5% is optimal for most natural fibers) varying the time (i.e. 15, 30, 45, 60, 75 and 90 mins) (Arthanarieswaran et al., 2015; Herlina Sari et al., 2018; Rajkumar et al., 2016). Later, the treated natural fibers are washed with deionized water, followed by the addition of few drops of 0.1 N hydrochloric acid to remove the excess impurities (Reddy et al., 2013; Saravanakumar et al., 2014a; Sonnier et al., 2018).

3.2 Acetic acid (CH_3 COOH) treatment

The acetic acid solution (indicated as 5, 10 and 15% (w /v)) is used to treat the fibers for 2 h in ambient temperature (23^0C) to remove hemicellulose and other fatty materials from the surface of the fibers. Later, it is used 0.1% (w / v) NaOH solution to neutralize the fibers, followed by washing with water and then drying at 100 °C for 24 h (Kommula et al., 2016).

3.3 Silane (SiH_4) treatment

Silane is a multifunctional molecule that is used as a coupling agent to modify the fiber surface (Asim et al., 2016 & 2018; Atiqah et al., 2018; Sepe et al., 2018). The vinyltrimethoxysilane and aminopropyl triethoxy silane are the commonly used silanes in order to obtain reliable modification of natural fibers (Singha et al., 2009; Singha and Thakur, 2009 ; Indira et al., 2012). When is used silane treatment, is required some amount of vinyltrimethoxysilane or aminopropyl triethoxy silanes that is mixed with an ethanol water mixture using the ratio (60:40). This solution is kept for 1 h and the pH is maintained at 4, by adding acetic acid. The fibers were immersed in the above solution for 2h, the treated fibers were later dried overnight at 60 °C (Sreekala et al., 1997; Xie et al., 2017; Zegaoui et al., 2018).

3.4 Benzoyl peroxide ($C_{14}H_{10}O_4$) treatment

For benzoyl peroxide treatment the fibers were immersed in 6% benzoyl peroxide in acetone for 30 min. The treated fibers were washed and air dried for 24 h (Paul et al., 1997; Saravanakumar et al., 2014b).

3.5 Potassium permanganate ($KMnO_4$) treatment

For potassium permanganate treatment, the fibers were immersed in 0.5 % potassium permanganate in acetone for 30 min. The treated fibers were washed and air dried for 24 h (Saravanakumar et al., 2014b).

3.6 Stearic acid ($(CH_3(CH_2)_{16}COOH)$) treatment

For stearic acid treatment, a solution of 1% stearic acid in ethyl alcohol was used. This solution is later poured slowly into the natural fibers placed in the glass vessel with continuous stirring. The treated fibers were later dried at 80 °C for 45 min (Paul et al., 1997; Saravanakumar et al., 2014b).

3.7 Seawater treatment

Another simple and economical method to modify natural fibers indicates the use of seawater. Firstly, pH of the seawater and the salinity need to be checked, later the fibers could be immersed in seawater for up to 30 days. Finally, the fibers were washed with water and dried at ambient temperature (Leman, et al., 2008; Rashid et al., 2016; Sreekala et al., 1997).

3.8 Cellulose powder (($C_6H_{10}O_5$)n) treatment

The preparation of natural fibers with cellulose powder assume that the fibers are soaked separately in steel containers containing 2% to 10% of cellulose pulp. It is prepared within hot distilled water, for up to 30 min. Later, the treated fibers were dried at 70 °C for 3 h (Indran et al., 2016).

3.9 Polymer Coating

Solution of polymer made from 12% mixture of A (i.e. 46% acrylic acid, 42% water, 8% styrene, 1.5% itaconic acid, and 2.5% alkyl diphenyl oxide disulfonate (anionic surfactant)), 1% of B mixture (i.e. 7.5% sodium persulphate and 92.5% water), 1% of anionic surfactant and water 86% were mixed at 14,000 rpm, at circa 82 °C for 3 h. Later, the fibers are immersed in the mixed polymer solution for 30 min. In this process, the carboxylic acid from a functional group of itaconic acid allows replacing the hydroxyl groups of natural fibers. Finally, the fibers were filtered and then dried for 24 h at 60 °C (Hajiha et al., 2014).

3.10 Bleaching

For bleaching, natural fibers are being treated with $Ca(ClO)_2$ (calcium hypochlorite) for 45 min. The bleached fibers are then washed with deionized water and then dried for 24h at 80 °C in a vacuum oven (Jayaramudu et al., 2011).

3.11 Graft copolymerization

In the graft copolymerization reaction, an initiator (KPS) in a small amount together with the monomer (MMA) is used once the fiber is immersed in distilled water. The reaction parameters should be controlled in order to obtain the optimum grafting percentage while combining the parameters such as time, temperature, the volume of solvent, initiator and monomer concentration (Bledzki et al., 1996; Malkapuram et al., 2009). The percentage grafting could be calculated using the equation, (Pg) = {(Wg – W)/ W} × 100 where 'W' is the weight of raw Grew (Mishra et al., 2001; Thakur et al., 2013 & 2014).

3.12 Isocyanate treatment

The treatment with isocyanate assumes that the natural fibers are prepared in a bottom flask having a round form that contains a certain amount of carbon tetrachloride (CCl_4) and a small amount of a catalyst (dibutyltin dilaurate). The urethane derivative is added dropwise to natural fibers containing a catalyst with stirring. The reaction will be completed in 1 h, later urethane treated fibers were refluxed during 8h in acetone by a soxhlet apparatus. Distilled water is required in the final step to treat the fibers while washing followed by drying at 80 °C in an oven (Bledzki et al., 1996; Malkapuram et al., 2009; Paul et al., 1997).

Among all chemical treatments, the most common chemical treatment for natural fibers is NaOH, because it is an easy and feasible technique to treat huge quantity of fibers, very little work being published on other chemical treatments. A summary of the chemical treatments and its effects on the natural fibers is presented in Table 4.

Table 4. The chemical treatments and its effects on the natural fibers

Chemical Treatment	Treatment effect	References
NaOH treatment	Remove the amorphous content, hemicellulose and lignin, which leads to fiber surface becoming rough. It is expected that such rough surfaces promote a strong link of interfacial bond when the fibers and the matrix composites are made of fibers as reinforcement	Arthanarieswaran et al., 2015; John and Thomas, 2008; Rajkumar et al., 2016; Reddy et al., 2013; Saravanakumar et al., 2014b;. Valadez-Gonzalez et al., 1999; Wang et al., 2007
Acetic acid treatment	Acetic acid treatment enhances tensile properties and the initial degradation temperature of the fibers. Therefore, the acid surface treatments will improve the performance when the fibers are used as natural reinforcement for composites.	Kabir et al., 2012; Kommula et. al., 2016; Mohanty et al., 2005
Silane treatment	Improves the physicochemical properties of natural fibers	Singha et al., 2009; Singha and Thakur, 2009; Sreekala et al., 1997

Benzoyl peroxide treatment	It allows to enhances the adhesion mechanism between the natural fiber and polymer matrix	Paul et al., 1997; Saravanakumar et al., 2014b
Potassium permanganate treatment	Enhance the physicochemical properties through the removal of wax and other cementing materials	Paul et al., 2008; Rahman et al., 2007; Saravanakumar et al., 2014b
Stearic acid treatment	Provides superior physicochemical properties when compared to all the above chemical treatments	Paul et al., 1997; Saravanakumar et al., 2014b
Seawater treatment	Removes the hemicellulose and generate pectin. This treatment leads to fibrillation of natural fibers similar to alkali treatment.	Ishak et al., 2009; Leman, et al., 2008; Rashid et al., 2016; Sreekala et al., 1997
Cellulose powder treatment	Provides a good wetting chemistry between the natural fibers and the matrix	Indran et al., 2016
Polymer Coating	Helps to improve the compatibility between the natural fibers and its polymer matrix	Hajiha et al., 2014
Bleaching	Permits to better control its thermal stability and/or the tensile properties of the natural fibers	Jayaramudu et al., 2011
Graft copolymerization	Improve the swelling and its thermal properties	Bledzki et al., 1996; Malkapuram et al., 2009;

		Mishra et al., 2001; Thakur et al., 2013 & 2014
Isocyanate treatment	Significantly increases the mechanical properties and water resistance of the natural fibers	Bledzki et al., 1996; Malkapuram et al., 2009; Paul et al., 1997

4. Surface treatments

The natural fibers require some surface treatments in order to improve its surface performance [George et al., 2001; Mohanty et al., 2001]. The chemical treatments are aimed to reduce the hydrophilic nature of the fibers, but the surface treatments may not only modify the fiber surface but also increase the fiber strength which leads to improving the adhesion mechanisms between the fiber surface and the polymer matrix. Despite the importance of surface treatment, there are a limited amount of approaches used to tackle this challenge due to the lack of availability of surface treatment equipment. This section explains the most commonly used surface treatments dedicated to the natural fibers and some beneficial effects on the fibers. Table 5 presents the surface treatments effects on natural fibers.

4.1 Plasma treatment

Plasma treatment has been successfully used to remove the impurities on the surface of the natural fibers (Cruz and Fangueiro, 2016; Shahidi et al., 2013). Oliveira et al., 2012, provided a robust review regarding the modification of the banana surface fibers by treatment with atmospheric dielectric discharge (DBD) plasma. A semi-industrial prototype machine from Softal Electronics GmbH was used to carry out the experiments in the ambient conditions while using atmospheric pressure. A brief representation of experimental DBD is shown in Fig.4. On a paper frame, each fiber was placed parallel to another one, they were fixed on a cotton fabric for a continuous flow treatment. After the plasma treatment, the paper frame is turned upside down for the treatment on the other side.

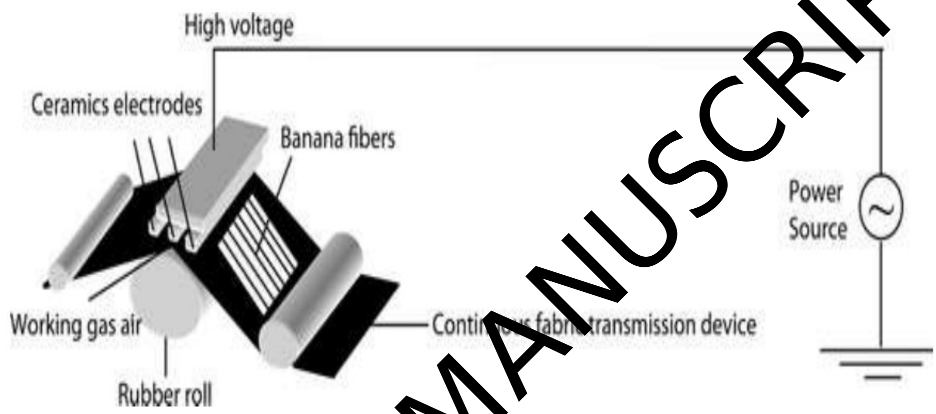

Fig. 4. Schematic DBD Plasma treatment device for surface preparation (Oliveira et al., 2012).

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4.2 Vacuum Ultraviolet Irradiation treatment

Vacuum ultraviolet irradiation (VUV) is considered a relatively new technique, widely accepted for removing impurities from the plant fiber surface. In an interesting work, Kato et al., 1999, addresses the surface oxidation of fibers by UVU treatment. During the treatment, the fibers were placed in a chamber of stainless steel having a length of 140 mm and diameter of 35 mm. High energy radiations below 200 nm were used for irradiation. The experiments are performed at room temperature with an applied pressure of 2.5 Torr. The Xe KsR-2A[8] was used as the source lamp equipped with MgF_2 window, 30 mm away from a sample holder. The photodiode measures an intensity of 3×10^{15} photons/(s cm2) when irradiated with radiations of 147 nm.

4.3 Ozone treatment

Ozone or oxygen-fluorine gas has been used successfully to improve the surface of the natural fiberssurface. In one of the important works by Kato et al., 1999, explained in detail the process that allows generating surface oxidation on the cellulose fibers using an ozone treatment method. This method assumes that the cellulose nonwoven form of fabrics are exposed to ozone gas at 20 °C, while the flow rate is set as 50 L/h. The time of exposure varies from 5 min to 9 h. An ozone generator type O-Z-2 from Nippon Ozone Co. Ltd., Tokyo, Japan was used to generate ozone, the machine was operated at a voltage of 100 V. The fibers that are treated are washed thoroughly using distilled water in order to remove any ozone adsorbed on the fiber surface, later on being vacuum dried for 24 h at 60 °C.

4.4 Corona treatment

Belgacem et al., 1995, conducted some experiments to observe the surface improvements of the cellulose fibers using corona treatment. In the experimental set up was consisted of two flat aluminium electrodes and a quartz plate as the dielectric spacer. About 1 g of cellulose fibres was placed in the corona cell with the cell volume of 5 cm^3 and treated for 1 min with an applied potential of 15 kV and frequency of 60 Hz at 25 °C with 50% relative humidity (Uehara and Sakata, 1990).

4.5 γ -Ray treatment

Toth et al., 2003, explained the treatment of γ -irradiation on cotton-cellulose. In this method, firstly the cotton cellulose was treated with NaOH (1– 6 mol dm^{-3}) this is followed with TMAH (1–3 mol dm^{-3}). The treated fibers were neutralized and dried. In the final stage, the fibers were post-processed with 5 kGy/h dose rate by a Co60 γ source in open air (Földváry et al., 2003; Takacs et al., 1999).

4.6 Laser treatment

Mizoguchi et al., 2003, conducted laser surface treatment on cellulose fibers by excimer laser irradiation system. Three types of excimer lasers (i.e. ArF (193 nm), KrF (248 nm) and XeCl (308 nm) were used for irradiation. The Lambda-Physik LPX210i (of ArF and KrF laser) and Lambda-Physik EMG102MSC (of XeCl laser) were used for fiber treatment at ambient condition. The pulse width consists of 20 ns of ArF, 23 ns of KrF and 14 ns of XeCl depending on the type of the laser, the frequency used is 1 Hz. Ne gas was used as a buffer gas. A concavo-convex lens is used to obtain good focus on the laser beam that permits to adjust the laser fluence. The fluence is measured using a joulemeter and an oscilloscope. The sample is placed in the sample holder between the concave-convex lens and joule meter. The area of irradiation is 0.24–1.20 cm^2. The influence of irradiation fluence (100–500 mJ/cm^2) and the pulses number (0–100) on the fiber surface structure could be analyzed.

Table 5. The surface treatments effects on natural fibers.

Surface Treatment	Treatment effect	References
Plasma treatment	Surface etching improves the surface roughness of the plant fibers, resulting in a better interface with the matrices through mechanical interlocking	Cruz and Fangueiro, 2016; Maissel and Glang, 1970; Oliveira et al., 2012; Shahidi et al., 2013; Sinha and Panigrahi, 2009

Vacuum Ultraviolet Irradiation treatment	Improves the surface properties such as adhesion, wettability, tribiological properties, fouling, barrier, insulation, dyeing, and biocompatibility	Kato et al., 1999
Ozone treatment	Helps to maintain its mechanical properties	Ali et al., 2018; Kato et al., 1999
Corona treatment	Enhance the acidity and the basicity of surface of fibers	Bataille et al., 1994; Belgacem et al., 1995, Uehara and Sakata, 1990
γ -Ray treatment	Increase in strength of natural fiber with gamma radiation dose due to the intercross-linking between the neighbouring cellulose molecules	Földváry et al., 2003; Khan et al., 2006; Takacs et al., 1999; Toth et al., 2003
Laser treatment	Remove lignin content and increases structural properties of fibers	Botaro et al., 2001; Kolar et al., 2000; Mizoguchi et al., 2003

5. Characterization Methods

This section is dedicated to presents the most useful characterization methods for the natural fibers. The characterization methods are very essential in order to select the natural fibers suitable as reinforcement for polymer composites.

5.1 Density measurement

Firstly, for measuring the density of the fibers, the fibers require drying for 48 h in a non-hygroscopic desiccator that contains calcium chloride (Varma et al., 1989). Later, the fiber is impregnated in toluene for 2 h to eliminate the existence of microbubbles from the fibers. The fibers were cut to a length of 5-10 mm and kept into the pycnometer (Beakou et al., 2008; Sathishkumar et al., 2013; Manimaran et al., 2018a). The density of natural fiber (ρ) is calculated by formula; $\rho = \left(\frac{m_2 - m_1}{(m_3 - m_1)(m_4 - m_2)} \right) \rho_T$, where m_1, m_2, m_3, and m_4 are the mass of the empty pycnometer (kg), pycnometer filled with chopped fibers (kg), pycnometer filled with toluene (kg), and pycnometer filled with chopped fibers and toluene solution (kg) respectively (McNulty and Kennedy, 1982; Rao and Rao, 2007; Truong et al., 2009).

5.2 Diameter measurement

In practice, the measurement of the diameter of the natural fibers is made using a digital micrometre or by a microscope (OM and SEM). The use of a digital micrometre (Beakou et al., 2008) permits measurements with an accuracy of 0.001 mm, otherwise, measurements by an air wedge (± 0.001 mm) (Balaji and Nagarajan, 2017) is another alternative technique.

5.2.1 Optical Microscope (OM)

It is well recognized the difficulty of measuring with precision the diameter of the natural fiber, because the fibers have an irregular shape and its thickness can vary. The natural fiber may form as numerous numbers of elements (i.e. fibers) surrounded by lignin and hemicelluloses, therefore their cross section is not circular. Fig. 5 is presented as a common option to measure an individual fiber bundle diameters by an optical microscope image analyser. The fiber (for consistency 5-10 samples were evaluated) is measured on 3-4 locations along its length and the average diameter is considered (Asim et al., 2016; Kabir et al., 2013).

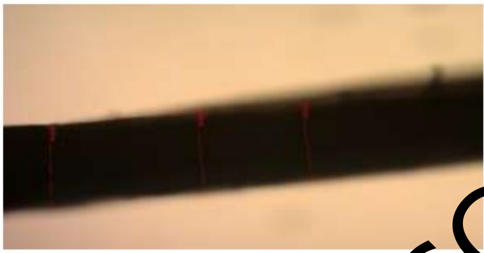


Fig. 5. Measurement of fiber diameter by an optical microscope (Sample Image for reference).

5.3 Fiber Fineness test

By following the guidelines of ASTM D1577 standard it is possible to obtain details of the fibers quality. This practice requires at least 10-20 samples of fiber (length of 200-300 mm) to be evaluated for consistency (Rwawiire and Tomkova, 2015).

5.4 Chemical analysis

Cellulose, hemicellulose, pectins and some lignin are considered as major components that form the natural plant fibers cell walls. As a result, a chemical measurement of a fiber composition is required. Beakou et al. adopted the method of Kurshner and Hoffer to measure the cellulose content. The crushing and extraction of the fiber are made by dichloromethane and later treated with 95% nitric acid solution and a mixture of ethanol. After the fibers treatment, the cellulose is generated in an insoluble fraction (Beakou et al., 2008). Goering in 1970 has proposed to estimate the hemicellulose contents, of the fibers, by a neutral detergent fiber approach. In this method, the fiber is prepared by refluxing the fiber in a solution of 10 ml that contain cold neutral detergent solution plus some sodium sulfite (the percentage depends on the amount of fiber) for 1 h. The mixture is filtered through a sintered glass crucible (G-2), later the residue require washing using hot distilled water and potentially ethanol (Agu et al., 2014). Lignin is usually calculated using the APPITA method P11s-78 (Pulp, 1978; Ververis et al., 2004), the TAPPI method is used to record the ash content (Tappi, 1993; Ververis et al., 2004), while the wax content is determined by the Conrad method (Conrad, 1944; Marsh et al., 1950).

5.5 Anatomy study

Anatomy investigations permits to obtain hierarchical structure details of the natural fibers (such as size, fiber bundles, fiber cells in fiber bundle, data of primary and secondary cell walls, and its cell chemical compositions) through optical microscope or by using SEM measurements (Belouadah et al., 2015; Saravanakumar et al., 2013).

5.6 X-ray powder diffraction (XRD) Analysis

XRD is a non-destructive and rapid analytical technique that is mainly used to identify the crystallographic structure, and chemical composition of natural fibers (Liu and Hu, 2008; Madhu et al., 2018; Marman, 1993). The X-ray diffraction allows the natural fibers (processed) to be scanned in a 2θ range, varying from 10º to 50º. The spectrum acquired from the measurements, corresponding to a given fiber (plotted in Fig. 6a), shows the diffraction peaks of the amorphous and crystalline regions. From the obtained X-ray diffractogram, the crystallinity index (CI) is calculated by the formula; $\mathrm{CI} = \left(1 - \frac{I_{AM}}{I_{000}}\right) \times 100\%$ where I_{00} and I_{AM} is the intensity of crystalline phase, and the amorphous phase respectively (Segal et al., 1959). The crystallite size (CS) could be calculated using the equation; $CS_{000} = \frac{0.89\lambda}{\beta_{000}\cos\theta}$, where β is the full-width at half-maximum of the peak, while θ is the Bragg angle (Seki et al., 2013).

5.7 Fourier Transformation by Infrared Spectroscopy (FTIR) Analysis

FTIR is considered a non-destructive analysis that can provide quantitatively and of course qualitative details of the natural fibers. An infrared absorption spectrum is obtained from chemical compositions of the natural fibers. FTIR spectra obtained from the natural fibers is principally observed in the range of 400-4000 cm^{-1} frequency (Madhu et al., 2018; Manimaran et al., 2018a). Some information gathered from an analysed sample are presented in Fig.6b. Table 6 provides the details of peak positions and its corresponding chemical stretching mode vibrations on the natural fiber.

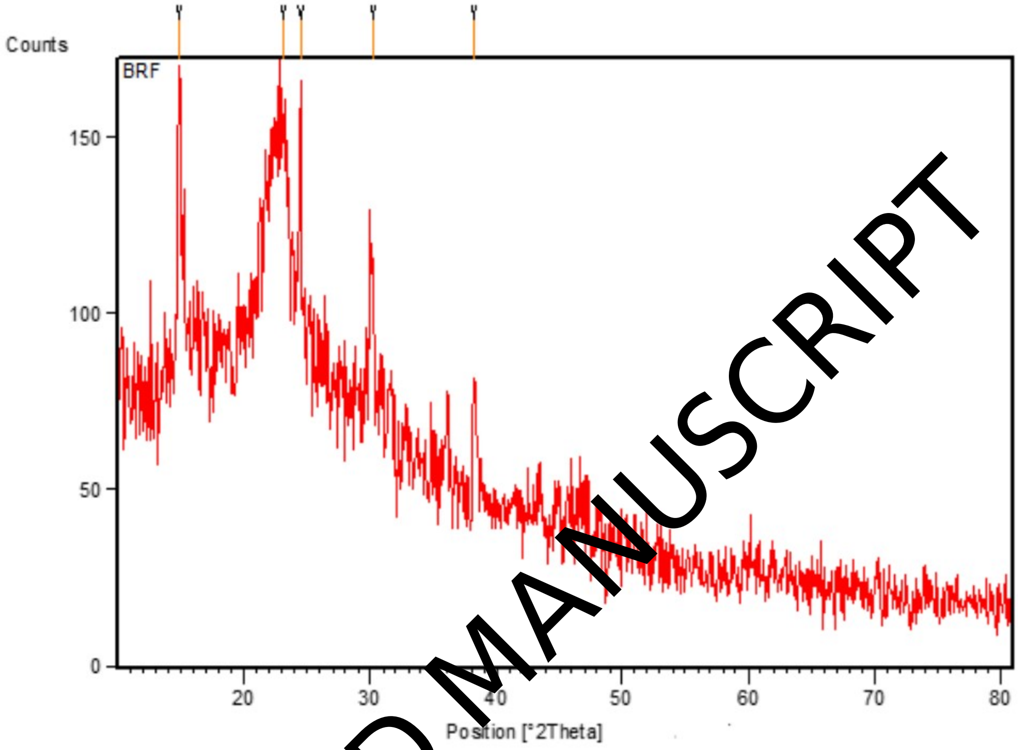


Fig. 6 (a). Natural fiber characterization by XRD (Sample Image for reference).

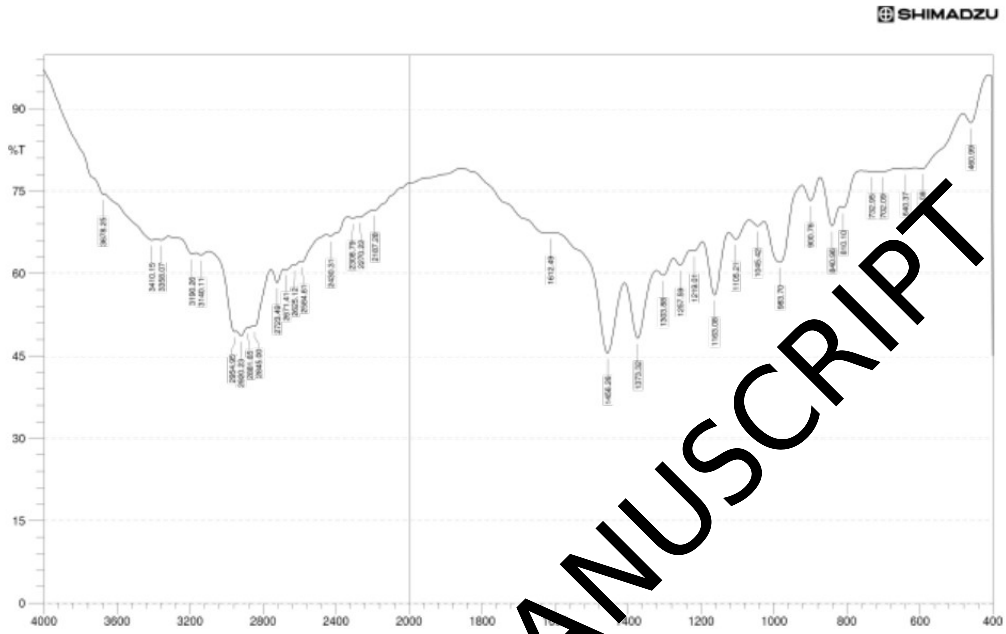


Fig. 6(b). Details obtained when is analysed a natural fiber sample using FTIR (Sample Image for reference).

Table 6. FTIR peak positions and corresponding chemical stretching mode vibrations on the natural fiber (Alawar et al., 2009; Arthanarieswaran et al., 2015; De Rosa et al., 2010; He et al., 2007; Jayaramudu et al., 2010; Le Troedec et al., 2008; Li et al., 2014; Maepa et al., 2015; Pandey, 1999; Tserk et al., 2005)

Wave number (cm^{-1})	**Allocations**
3700 – 3500	O-H stretching of α-cellulose
3500 – 3300	N-H stretching (Amine)
3500	N-H stretching (Amide)

3100 – 3000	C=C-H stretching
2960 – 2850	C-H stretching
2590 – 2540	S-H stretching
1740 – 1720	C=O stretching of hemicelluloses
1685 -1655 & 1600	α, β – unsaturated stretching
1680 – 1650	N=O stretching
1650 – 1630	OH (Absorbed water)
1600	C=O stretching (Amide)
1600,1580,1500,1450	C=C stretching
1450 – 1400	CH_2 Symmetric bending
1567,1380	NO_2 stretching
Above 1500	C=O,NH_2,NH,C=C,C=N functional group stretching
Below 1500	Single bonds and bending vibrations
1395-1385	t-butyl stretching
1365	CH bending (deformation)
1350-1260	C-O stretching
1150 – 1070	C-O-C stretching
1050 – 1020	Symmetric C-OH stretching of lignin
815 – 750	N-O stretching
800 - 600	C-S stretching
770 – 735	C-C deformation
520 – 420	S-S stretching

5.8 Thermogravimetric analysis (TGA)

TGA method permits to measure the thermal performance of the fibers when the weight of material changes. The physical and its chemical properties can be measured as a function of increasing temperature while keeping a constant heating rate. The changes in mass during the measurement is evaluated by a thermal analyser. This method requires that the natural fibers are exposed to nitrogen gas while using a flow rate of ca. 20 ml/min. The natural fiber powder is placed in an alumina pan to measure the temperature of the thermocouple. The increase in the temperature is made by increasing the heating rates in steps from a given value of 10 °C/min over a range of temperature (from room temperature to 1000 °C) (Manimaran et al., 2018b & 2018c; Saravanakumar et al., 2013).

5.9 Differential scanning calorimetry (DSC) Analysis

The analysis is performed by using a DSC machine. Approximately 2–3 g of natural fiber powder is placed and sealed in aluminium pans. An imposed heating rate of 10°C/min is required, conditions that are obtained while heated in an inert N_2 atmosphere that starts from room temperature to the melting peak of the fibers. The melting peak (ΔH) and Tg are determined as standard (Chand and Joshi, 2010; Rwawiire and Tomkova, 2015).

5.10 Scanning Electron Microscope (SEM) Analysis

The morphology of a fracture surface produced from the composites and fibers are analysed by SEM to determine the ability of the fiber to act as a good reinforcement. SEM provides detailed high-resolution images (e. g. in Fig.7) of the fibers when scanning by an electron beam focused across the surface and detecting the secondary or backscattered electronic signal. The sample used on the SEM analysis requires to be covered with some amount of thin gold layer in order to prevent the potential accumulation of the electrical charges once analysis (Manimaran et al., 2018a, 2018b & 2018c; Madhu et al., 2018).

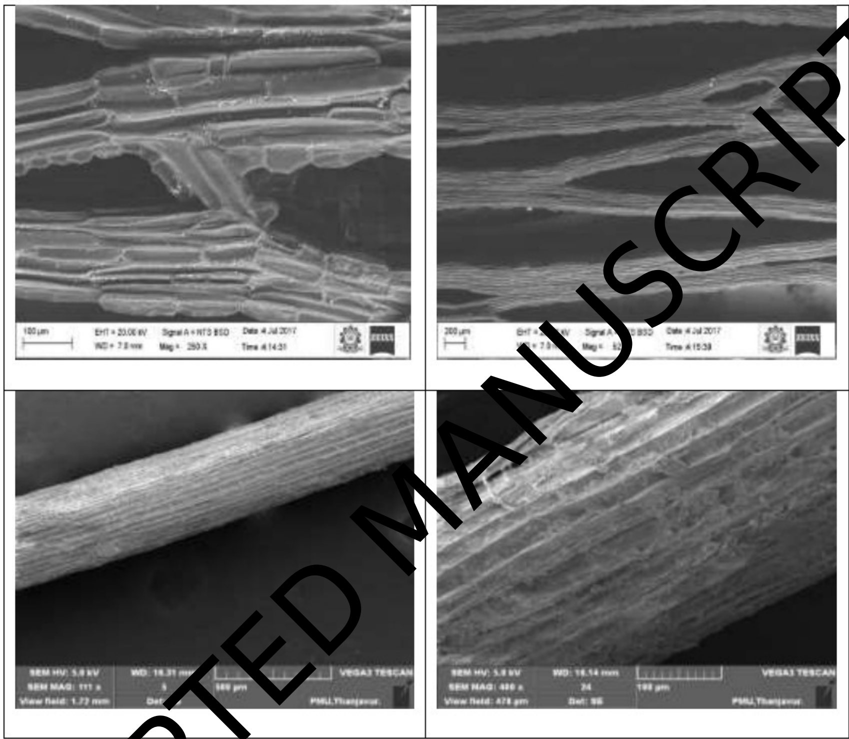


Fig. 7. SEM micrographs of a natural fiber (Sample Images for reference).

5.11 Transmission electron microscope (TEM) Analysis

The TEM micrographs give precisely diameter of the fibers. Even the minute details of the fibers could be analysed by TEM micrographs. The transverse dimensions of various sublayers that forms a cell wall could be analysed by TEM micrographs (Beakou et al., 2008). However, these methods require a stringent process of sample preparation. Initially, the fibers are boiled twice with excessive 1% NaOH solution, for 3 h. The fibers were then treated with 0.05 mol/L HCl solution and washed with water. Later, the fibers are placed in distilled water, over 4h, to produce a sample that permits carefully examination of the fibers structure in TEM (Liu et al., 2009).

5.12 Energy dispersive X-ray spectroscopy analysis (EDX)

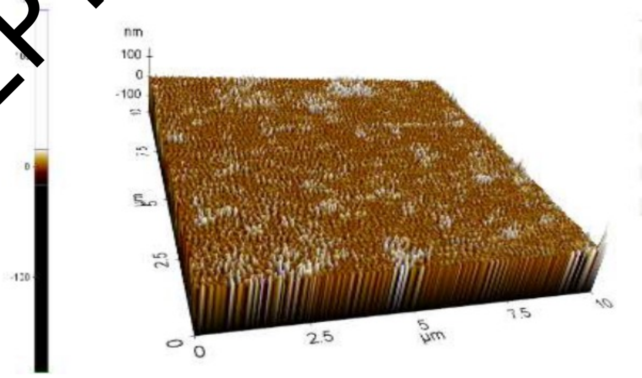
The EDX requires an analytical approach to explore the chemical composition generated by fibers surface. This method allows detecting the major chemical compositions of fiber including C and O along with Na, Al, Si, Mg. However, it cannot detect H, which represents the major constituents of natural fibers (Ali, 2016; Rashid et al., 2016). Fig. 8a. presents a sample image obtained by EDX.

5.13 Atomic Force Microscopy (AFM) Analysis

The AFM technique allows obtaining measurements of the surface profile with a resolution up to the subnanometer level. This method requires only some amount of sample preparation (i.e. cleaning). AFM measures the attractive and/or repulsive forces existing between the tip of a cantilever and the fiber. Therefore, AFM can measure directly the forces that dominate the adhesion phenomena (Balnois et al., 2007; Sghaier et al., 2012). In this method, a sharp tip cantilever requires scanning over the fiber. The repulsive forces produced when the cantilever tip touch the fiber allows to deflect the cantilever. With the help of a laser beam and a photodiode detector, the amount of cantilever deflection can be identified and monitored. The roughness parameters obtained by this measurement are, the average roughness (R_a), the root mean square roughness (R_q) the maximum peak to valley height (R_t) the average absolute height roughness of 10 points (R_z), the asymmetry (R_{sk})and the kurtosis (R_{ku}). The AFM can provide a characterization of the fiber surface on the 3-D dimension (Senthamaraikannan et al., 2016). Fig. 8 (b) & (c). shows a sample image captured by AFM analysis.

Fig. 8 (a). Image sample obtained by EDX of a natural fiber.

(b)



(c)

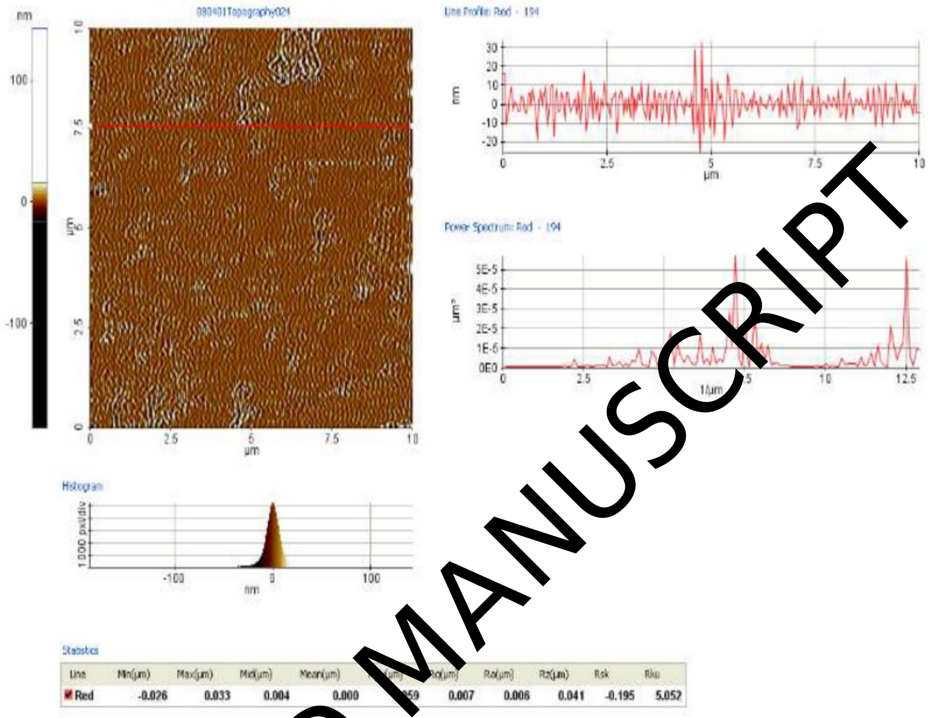


Fig. 8. Detail of a natural fiber image acquired by AFM. b) 3D characterization and c) 2D profile (Sample Images for reference).

5.14 Single Fiber Tensile test

The natural fibers tensile properties are driven by three main factors such as test parameters (conditions), type of plant fiber, and the fiber dimensions (thickness, width, and length) on the cross-section (Bezazi et al., 2014). The fibers should be tested under tensile loading by following the ASTM C1557-03 and/or ASTM D 3822–07 standards using universal test machine at an operating speed of 0.5 mm/min. In order to obtain a higher precision, it is recommended to perform the test with a servo-electric tensile machine with a 5 kN load cell or less. The fiber samples need some preparation before testing. Each fiber edge requires fixing using epoxy resin and then bonded on a classical stiff paper frame. On the testing machine, the samples should be clamped on a mechanical grip. Once the sample is placed on mechanical grips, the edge of the paper frame is carefully cut into two parts (Bourahli, 2017; (Fiore et al., 2011; Maache et al., 2017).

It is well known that very few researchers have also applied other less practical characterization techniques to investigate the natural fibers surface characteristics such as the nuclear magnetic resonance spectroscopy (NMR) analysis (Borchani et al., 2015; Chaulia, 2010; Reddy et al., 2014), inverse gas chromatography(IGC) (Heng et al., 2007), and X-Ray photoelectron spectroscopy (XPS) (Sarikanat et al., 2014; Seki et al., 2013).

Conclusion

Global warming, environmental concerns and the new technology requirements have fortified scientists to produce novel materials such as natural fiber composites. The use of bio-materials from local resources for polymer composites which increases the environmental awareness and reduce the unsustainable consumption of synthetic materials, as well as the cost of natural fiber is very little compared to other synthetic materials. This review was devoted to generating a robust understanding of the preparation and processing methods of natural fibers. The findings presented can be used to create novel polymer composites structure. It was highlighted the importance of selection suitable retting method that plays a crucial role. By applying proper chemical treatments on the natural fibers will be possible to obtain better surface characteristics that allow to reduce the hydrophilic tendency and permits improving the compatibility between the fibers and its material matrix. Among all chemical treatments studied, alkali treatment is an easy, economical and a very effective technique for treatment of a huge quantity of fibers. It was noted as well as that the alkaline solution may react with the OH groups, made of fibers, and helps in increases the hydrophobicity of the fibers, resulting in better fiber reinforcement for polymer composites. Moreover, the alkaline treatment potentially leads to an unexpected mechanism that generates the splitting of the fiber bundles. The surface treatments of the natural fibers represent a very challenge topic for the research community mainly because of the effective use of the natural fibers in polymer composites for various applications. Further, the fibre-reinforced polymer composites performances are largely dependent on the material reinforcement properties and its behaviour. It concluded that chemical and surface treatments can enhance the physicomechanical and thermochemical properties of the natural fibers. An appropriate method of characterization of natural fibers permits to create a novel sustainable composite material dedicated for industrial applications.

Future Prospectives

Composite materials made from particularly natural fibers are perspective materials in which case a reinforcing material is based on natural and renewable resources. Due of its environmentally friendly and sustainable nature, the natural fibers can be used for creating new composites structure that offers new technology and commercial prospects for different sectors, for instance, aerospace, automotive, and electronics industries. These composites can also be used as construction and building materials, which enhances environmental concerns that can also result in the depletion and reduction of forest wood resources. Future research on possible ways of improving quality of natural fibers leading key changes in material properties and their subsequent potential future applications in several composite based industries is also desirable. Overall, the present review article will give a substantial understanding of the processing techniques of commonly used natural fibers, considered as a robust database for sustainable growth towards producing the best fiber composites materials.

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References

Agu, C.V., Njoku, O.U., Chilaka, F.C., Agbiogwu, D., Iloabuchi, K.V., Ukazu, B., 2014. Physicochemical properties of lignocellulosic biofibres from South Eastern Nigeria: Their suitability for biocomposite technology. Afr. J. Biotechnol. 13(20), 2050-2057.

Ahmed, M.J., Balaji, M.S., Saravanakumar, S.S., Sanjay, M.R., Senthamaraikannan, P., 2018. Characterization of Areca javanica fiber–A possible replacement for synthetic acrylic fiber in the disc brake pad. J. Indus. Tex., p.1528083718779446.

Ahmed, Z., Akhter, F., 2001. Jute retting: an overview. Online. J. Biol. Sci. 1(7), 685-688.

Akil, H., Omar, M. F., Mazuki, A.A.M., Safiee, S.Z.A.M., Ishak, Z. M., Bakar, A. A., 2011. Kenaf fiber reinforced composites: A review. Mat. Des. 32(8-9), 4107-4121.

Alawar, A., Hamed, A. M., Al-Kaabi, K., 2009. Characterization of treated date palm tree fiber as composite reinforcement. Compos. Part B: Eng. 40(7), 601-606.

Al-Oqla, F.M. and Sapuan, S.M., 2014. Natural fiber reinforced polymer composites in industrial applications: feasibility of date palm fibers for sustainable automotive industry. J. Clean. Prod. 66, 347-354.

Ali, A., Shaker, K., Nawab, Y., Jabbar, M., Hussain, T., Militky, J., Baheti, V., 2018. Hydrophobic treatment of natural fibers and their composites—a review. J. Ind. Text. 47(8), 2153-2183.

Ali, M., 2016. Microstructure, Thermal Analysis and Acoustic Characteristics of Calotropis procera (Apple of Sodom) Fibers. J. Nat. Fibers. 13(3), 343-352.

Alves, C., Silva, A.J., Reis, L.G., Freitas, M., Rodrigues, L.B. and Alves, D.E., 2010. Ecodesign of automotive components making use of natural jute fiber composites. J. Clean. Prod. 18(4), 313-327.

Antonov, V., Marek, J., Bjelkova, M., Smirous, P., Fischer, H., 2007. Easily available enzymes as natural retting agents. Biotechnol. J. 2(3), 342-346.

Amaducci, S., Gusovius, H.J., 2010. Hemp–cultivation, extraction and processing. Industrial Applications of Natural Fibres Structure, Properties and Technical Applications. John Wiley & Sons, Ltd, Chichester, West Sussex, United Kingdom, 109-134.

Arthanarieswaran, V.P., Kumaravel, A., Saravanakumar, S.S., 2015. Physico-chemical properties of alkali-treated Acacia leucophloea fibers. Int. J. Polym. Anal. Ch. 20(8), 704-713.

Asim, M., Jawaid, M., Abdan, K., Ishak, M.R., 2016. Effect of alkali and silane treatments on mechanical and fibre-matrix bond strength of kenaf and pineapple leaf fibres. J. Bio. Eng. 13(3), 426-435.

Asim, M., Paridah, M.T., Saba, N., Jawaid, M., Alothman, O.Y., Nasir, M., Almutairi, Z., 2018. Thermal, physical properties and flammability of silane treated kenaf/pineapple leaf fibres phenolic hybrid composites. Compos. Struct. 202, 1330-1338.

Atiqah, A., Jawaid, M., Ishak, M. R., Sapuan, S. M., 2018. Effect of Alkali and Silane Treatments on Mechanical and Interfacial Bonding Strength of Sugar Palm Fibers with Thermoplastic Polyurethane. J. Nat. Fibers. 15(2), 251-261.

Bacci, L., Di Lonardo, S., Albanese, L., Mastromei, G., Perito, B., 2011. Effect of different extraction methods on fiber quality of nettle (Urtica dioica L.). Tex. Res.J. 81(8), 827-837.

Balnois, E., Busnel, F., Baley, C., Grohens, Y., 2007. An AFM study of the effect of chemical treatments on the surface microstructure and adhesion properties of flax fibres. Comp. Interf. 14(7-9), 715-731.

Balaji, A.N., Nagarajan, K.J., 2017. Characterization of alkali treated and untreated new cellulosic fiber from Saharan aloe vera cactus leaves. Carbohydr. Polym. 174, 200-208.

Bataille, P., Dufourd, M., Sapieha, S., 1994. Copolymerization of styrene on to cellulose activated by corona. Poly. Inter. 34(4), 387-391.

Beakou, A., Ntenga, R., Lepetit, J., Ateba, J.A., Ayina, L.O., 2008. Physico-chemical and microstructural characterization of "Rhectophyllum camerunense" plant fiber. Compos. Part A: Appl. Sci. Manuf. 39(1), 67-74.

Belgacem, M.N., Czeremuszkin, G., Sapieha, S., Gandini, A., 1995. Surface characterization of cellulose fibres by XPS and inverse gas chromatography. Cellul. 2(3), 145-157.

Belouadah, Z., Ati, A., Rokbi, M., 2015. Characterization of new natural cellulosic fiber from Lygeum spartum L. Carbohydr. Polym. 134, 429-437.

Bezazi, A., Belaadi, A., Bourchak, M., Scarpa, F., Boba, K., 2014. Novel extraction techniques, chemical and mechanical characterisation of Agave americana L. natural fibres. Compos. Part B: Eng. 66, 194-203.

Bledzki, A.K., Reihmane, S., Gassan, J., 1996. Properties and modification methods for vegetable fibers for natural fiber composites. J. Appl. Polym. Sci. 59(8), 1329-1336.

Bledzki, A.K., Gassan, J., 1999. Composites reinforced with cellulose based fibres. Prog. Poly. Sci. 24(2), 221-274.

Botaro, V.R., Dos Santos, C.G., Júnior, G.A., Da Costa, A.R., 2001. Chemical modification of lignocellulosic materials by irradiation with Nd-YAG pulsed laser. Appl. Surf. Sci. 183(1-2), 120-125.

Booth, I., Goodman, A.M., Grishanov, S.A., Harwood, R.J., 2004. A mechanical investigation of the retting process in dew- retted hemp (Cannabis sativa). Ann. Appl. Biol. 145(1), 51-58.

Borchani, K.E., Carrot, C., Jaziri, M., 2015. Untreated and alkali treated fibers from Alfa stem: effect of alkali treatment on structural, morphological and thermal features. Cellul. 22(3), 1577-1589.

Bourahli, M. E. H., 2017. Uni-and bimodal Weibull distribution for analysing the tensile strength of Diss fibers. J. Nat. Fibers. 1-10.

Chand, N., Joshi, R., 2010. Analysis of mechanical, thermal, and dynamic mechanical behaviors of different polymer-coated sisal fibers. J. Nat. Fibers. 7(2), 100-110.

Chadlia, A., 2010. Chemical modification of Posidonia with cyclic anhydrides: effect on thermal stability. Carbohydr. Res. 345(2), 264-269.

Conrad, C.M., 1944. Determination of wax in cotton fiber a new alcohol extraction method. Indus. Eng. Chem. Analy. Edi. 16(12), 745-748.

Cruz, J., Fangueiro, R., 2016. Surface modification of natural fibers: a review. Proc. Eng. 155, 285-288.

De Rosa, I.M., Kenny, J.M., Puglia, D., Santulli, C., Sarasini, F., 2010. Morphological, thermal and mechanical characterization of okra (Abelmoschus esculentus) fibres as potential reinforcement in polymer composites. Compos. Sci. Technol. 70(1), 116-122.

Dittenber, D.B., GangaRao, H.V., 2012. Critical review of recent publications on use of natural composites in infrastructure. Compos. Part A: Appl. Sci. Manuf. 43(8), 1419-1429.

Fiore, V., Valenza, A., Di Bella, G., 2011. Artichoke (Cynara cardunculus L.) fibres as potential reinforcement of composite structures. Comp. Sci. Technol. 71(8), 1138-1144.

Földváry, C.M., Takács, E., Wojnarovits, L., 2003. Effect of high-energy radiation and alkali treatment on the properties of cellulose. Rad. Phy. Chem. 67(3-4), 505-508.

George, J., Sreekala, M.S., Thomas, S., 2001. A review on interface modification and characterization of natural fiber reinforced plastic composites. Polym. Eng. Sci. 41(9), 1471-1485.

Goering, H.K. 1970. Forage Fiber Analysis (Apparatus, reagents, procedures and some applications. Agric Handbook, 379, 20.

Gowda, T.Y., Sanjay, M.R., Bhat, K.S., Madhu, P., Senthamaraikannan, P., Yogesha, B., 2018. Polymer matrix-natural fiber composites: An overview. Cog. Eng. 5(1), 1446667.

Gurukarthik Babu, B., Princewinston, D., SenthamaraiKannan, P., Saravanakumar, S.S., Sanjay, M.R., 2018. Study on characterization and physicochemical properties of new natural fiber from Phaseolus vulgaris. J. Nat. Fibers. 1-8.

Hajiha, H., Sain, M., Mei, L. H., 2014. Modification and characterization of hemp and sisal fibers. J. Nat. Fibers. 11(2), 144-168.

He, J.X., Tang, Y., Wang, S.Y., 2007. Differences in morphological characteristics of bamboo fibres and other natural cellulose fibres: Studies on X-ray diffraction, solid state 13C-CP/MAS NMR, and second derivative FTIR spectroscopy data. Iran. Polym. J. 807-818.

Herlina Sari, N., Wardana, I.N.G., Irawan, Y.S., Siswanto, E., 2018. Characterization of the chemical, physical, and mechanical properties of NaOH-treated natural cellulosic fibers from corn husks. J. Nat. Fibers. 15(4), 545-558.

Heng, J.Y., Pearse, D.F., Thielmann, F., Lampke, T., Bismarck, A., 2007. Methods to determine surface energies of natural fibres: a review. Compos. Interface., 14(7-9), 581-604.

Holbery, J., Houston, D., 2006. Natural-fiber-reinforced polymer composites in automotive applications. Jom, 58(11), 80-86.

Hyness, N.R.J., Vignesh, N.J., Senthamaraikannan, P., Saravanakumar, S.S., Sanjay, M.R., 2018. Characterization of new natural cellulosic fiber from Heteropogon contortus plant. J. Nat. Fibers. 15(1), 146-153.

Indira, K. N., Jyotishkumar, P., and Thomas, S., 2012. Thermal Stability and Degradation of Banana Fibre/PF Composites Fabricated by RTM. Fiber. Polym. 13(10), 1319-1325.

Indran, S., Raj, R.E., Daniel, B.S.S., Saravanakumar, S.S., 2016. Cellulose powder treatment on Cissus quadrangularis stem fiber-reinforcement in unsaturated polyester matrix composites. J. Reinf. Plast. Compos. 35(3), 212-227.

Ishak, M. ., Leman, Z., Sapuan, S.M., Salleh, M.Y., Misri, S. 2009., The effect of sea water treatment on the impact and flexural strength of sugar palm fibre reinforced epoxy composites. Inter. J. Mech. Mater. Eng. 4(3), 316-320.

Jankauskienė, Z., Butkutė, B., Gruzdevienė, E., Cesevičienė, J., Fernando, A. L., 2015. Chemical composition and physical properties of dew-and water-retted hemp fibers. Ind. Crops. Prod. 75, 206-211.

Jawaid, M., H.P.S., Khalil, H.A., 2011., Cellulosic/synthetic fibre reinforced polymer hybrid composites: A review. Carbohydr. Polym. 86(1), 1-18.

Jayaramudu, J., Guduri, B.R., Rajulu, A.V.. 2010. Characterization of new natural cellulosic fabric Grewia tilifolia. Carbohydr. Polym. 79(4), 847-851.

Jayaramudu, J., Maity, A., Sadiku, E.R., Guduri, B.R., Rajulu, A.V., Ramana, C.V., Li, R., 2011. Structure and properties of new natural cellulose fabrics from Cordia dichotoma. Carbohydr. Polym. 86(4), 1623-1629.

John, M.J., Thomas, S., 2008. Biofibres and biocomposites. Carbohydr. Polym. 71(3), 343-364.

Kabir, M.M., Wang, H., Lau, K.T., Cardona, F., 2012. Chemical treatments on plant-based natural fibre reinforced polymer composites: An overview. Compos. Part B: Eng. 43(7), 2883-2892.

Kabir, M.M., Wang, H., Lau, K.T., Cardona, F., 2013. Tensile properties of chemically treated hemp fibres as reinforcement for composites. Compos. Part B: Eng. 53, 362-368.

Kato, K., Vasilets, V. N., Fursa, M. N., Meguro, M., Ikeda, Y., Nakamae, K., 1999. Surface oxidation of cellulose fibers by vacuum ultraviolet irradiation. J. Polym. Sci. Part A: Polym. Chem. 37(3), 357-361.

Khan, M.A., Haque, N., Al-Kafi, A., Alam, M.N., Abedin, M.Z., 2006. Jute reinforced polymer composite by gamma radiation: effect of surface treatment with UV radiation. Polymer. Plast. Tech. Eng. 45(5), 607-613.

Kolar, J., Strlic, M., Müller-Hess, D., Gruber, A., Troschke, K., Pentzien, S., Kautek, W., 2000. Near-UV and visible pulsed laser interaction with paper. J. Cul. Her. 1, S221-S224.

Kommula, V.P., Reddy, K.O., Shukla, M., Marwala, T., Reddy, E.S., Rajulu, A.V., 2016. Extraction, modification, and characterization of natural ligno-cellulosic fiber strands from napier grass. Int. J. Polym. Anal. Ch. 21(1), 18-28.

Krassig, H. A., Cellulose-Structure, A., Reactivity, G., 1993. Breach Science Publisher. New York.

Lampke, T., Mishra, S., Bismarck, A., 2005. Plant Fibers as Reinforcement for Green Composites. In Natural Fibers, Biopolymers, and Biocomposites (pp. 52-128). CRC Press.

Le Troedec, M., Sedan, D., Peyratout, C., Bonnet, J.P., Smith, A., Guinebretiere, R., Krausz, P., 2008. Influence of various chemical treatments on the composition and structure of hemp fibres. Compos. Part A: Appl. Sci. Manuf. 39(3), 514-522.

Leman, Z., Sapuan, S.M., Azwan, M., Ahmad, M.M.H.M., Maleque, M.A., 2008. The effect of environmental treatments on fiber surface properties and tensile strength of sugar palm fiber-reinforced epoxy composites. Polymer. Plast. Tech. Eng. 47(6), 606-612.

Li, X., Tabil, L.G., Panigrahi, S., 2007. Chemical treatments of natural fiber for use in natural fiber-reinforced composites: a review. J. Polym. Environ. 15(1), 25-33.

Li, Y., Li, G., Zou, Y., Zhou, Q., Lian, X., 2014. Preparation and characterization of cellulose nanofibers from partly mercerized cotton by mixed acid hydrolysis. Cellulose. 21(1), 301-309.

Liu, Y., Hu, H., 2008. X-ray diffraction study of bamboo fibers treated with NaOH. Fiber. Polym. 9(6), 735-739.

Liu, D., Han, G., Huang, J., Zhang, Y., 2009. Composition and structure study of natural Nelumbo nucifera fiber. Carbohydr. Polym. 75(1), 39-43.

Liu, Y., Ma, Y., Yu, J., Zhuang, J., Wu, S., Tong, J., 2018. Development and characterization of alkali treated abaca fiber reinforced friction composites. Compos. Interf. 1-16.

Maache, M., Bezazi, A., Amroune, S., Scarpa, F., Dufresne, A., 2017. Characterization of a novel natural cellulosic fiber from Juncus effusus L. Carbohydr. Polym. 171, 163-172.

Maepa, C.E., Jayaramudu, J., Okonkwo, J.O., Ray, S.S., Sadiku, E.R., Ramontja, J., 2015. Extraction and characterization of natural cellulose fibers from maize tassel. Int. J. Polym. Anal. Ch. 20(2), 99-109.

Maissel, L.I., Glang, R., 1970. Handbook of thin film technology. New York: McGraw-Hill, 1970, edited by Maissel, Leon I.; Glang, Reinhard.

Madhu, P., Sanjay, M.R., Senthamaraikannan, P., Pradeep, S., Saravanakumar, S.S., Yogesha, B., 2018. A review on synthesis and characterization of commercially available natural fibers: Part-II. J. Nat. Fibers. 1-13.

Malkapuram, R., Kumar, V., Negi, Y.S., 2009. Recent development in natural fiber reinforced polypropylene composites. J. Reinf. Plast. Compos. 28(10), 1169-1189.

Manimaran, P., Senthamaraikannan, P., Murugananthan, K., Sanjay, M.R., 2018a. Physicochemical properties of new cellulosic fibers from Azadirachta indica plant. J. Nat. Fibers. 15(1), 29-38.

Manimaran, P., Senthamaraikannan, P., Sanjay, M.R., Marichelvam, M.K., Jawaid, M., 2018b. Study on characterization of Furcraea foetida new natural fiber as composite reinforcement for lightweight applications. Carbohydr. Polym. 181, 650-658.

Manimaran, P., Sanjay, M.R., Senthamaraikannan, P., Jawaid, M., Saravanakumar, S.S., George, R., 2018c. Synthesis and characterization of cellulosic fiber from red banana peduncle as reinforcement for potential applications. J. Nat. Fibers. 1-13.

Mannan, K.M., 1993. X-ray diffraction study of jute fibres treated with NaOH and liquid anhydrous ammonia. Poly. 34(12), 2485-2487.

Marsh, P.B., Barker, H.D., Kerr, T., Butler, M. L., 1950. Wax content as related to surface area of cotton fibers. Text. Res. J. 20(5), 288-297.

McNulty, P.B., Kennedy, S., 1982. Density measurements of grass by toluene displacement and air comparison pycnometry. Irish. J. Agri. Res. 75-83.

Mishra, S., Misra, M., Tripathy, S.S., Nayak, S.K., Mohanty, A.K., 2001. Graft copolymerization of acrylonitrile on chemically modified sisal fibers. Macro. Mat. Eng. 286(2), 107-113.

Mizoguchi, K., Ishikawa, M., Ohkubo, S., Yamamoto, A., Ouchi, A., Sakuragi, M., Sugiyama, O., 2000. Laser surface treatment of regenerated cellulose fiber. Compos. Interf., 7(5-6), 497-509.

Mohanty, A.K., Misra, M., Drzal, L.T., 2001. Surface modifications of natural fibers and performance of the resulting biocomposites: an overview. Compos. Interf., 8(5), 313-343.

Mohanty, A.K., Misra, M., Drzal, L.T., Selke, S.E., Harte, B.R., Hinrichsen, G., 2005. Natural Fibers, Biopolymers, and Biocomposites: An Introduction. Natural Fibers, Biopolymers and Biocomposites, Taylor & Francis: Boca Raton.

Mwaikambo, L.Y., Ansell, M.P., 2002. Chemical modification of hemp, sisal, jute, and kapok fibers by alkalization. J. Appl. Poly. Sci. 84(12), 2222-2234.

Neelamana, I. K., Thomas, S., Parameswaranpillai, J., 2013 Characteristics of Banana Fibers and Banana Fiber Reinforced Phenol Formaldehyde Composites-Macroscale to Nanoscale. J. Appl. Polym. Sci. 130, 1239–1246.

Oliveira, F.R., Erkens, L., Fangueiro, R., Souto, A. P., 2012. Surface modification of banana fibers by DBD plasma treatment. Plas. Chem. Plas. Proc. 32(2), 259-273.

Pandey, K.K., 1999. A study of chemical structure of soft and hardwood and wood polymers by FTIR spectroscopy. J. Appl. Poly. Sci. 71(12), 1969-1975.

Paridah, M.T., Basher, A.B., SaifulAzry, S., Ahmed, Z., 2011. Retting process of some bast plant fibres and its effect on fibre quality: A review. BioRes. 6(4), 5260-5281.

Paul, A., Joseph, K., Thomas, S., 1997. Effect of surface treatments on the electrical properties of low-density polyethylene composites reinforced with short sisal fibers. Comp. Sci. Tech., 57(1), 67-79.

Paul, S.A., Boudenne, A., Ibos, L., Candau, Y., Joseph, K., Thomas, S., 2008. Effect of fiber loading and chemical treatments on thermophysical properties of banana fiber/polypropylene commingled composite materials. Compos. Part A: Appl. Sci. Manuf., 39(9), 1582-1588.

Pickering, K.L., Li, Y., Farrell, R.L., Lay, M., 2007. Interfacial modification of hemp fiber reinforced composites using fungal and alkali treatment. J. Bio. Mat. Bio. 1(1), 109-117.

Puglia, D., Biagiotti, J., Kenny, J.M., 2005. A review on natural fibre-based composites—Part II: Application of natural reinforcements in composite materials for automotive industry. J. Nat. Fib. 1(3), 23-65.

Pulp, A., Paper Industry Technical Association. 1978. Klason lignin in wood and pulp. Appita Standard P11s.

Puttegowda, M., Sanjay, M.R., Jawaid, M., Shivanna, P., Basavegowda, Y., Saba, N., 2018. Potential of natural/synthetic hybrid composites for aerospace applications. In Sustainable Composites for Aerospace Applications, 315-351.

Rahman, M.M., Mallik, A.K., Khan, M.A. 2007., Influences of various surface pretreatments on the mechanical and degradable properties of photografted oil palm fibers. J. Appl. Poly. Sci. 105(5), 3077-3086.

Rajkumar, R., Manikandan, A., Saravanakumar, S. S., 2016. Physicochemical properties of alkali-treated new cellulosic fiber from cotton shell. Int. J. Polym. Anal. Ch. 21(4), 359-364.

Rao, K.M.M., Rao, K.M., 2007. Extraction and tensile properties of natural fibers: Vakka, date and bamboo. Compos. Struct. 77(3), 288-295.

Rashid, B., Leman, Z., Jawaid, M., Ghazali, M.J., Ishak, M.R., 2016. Physicochemical and thermal properties of lignocellulosic fiber from sugar palm fibers: Effect of treatment. Cellul. 23(5), 2905-2916.

Reddy, N., Yang, Y., 2005. Properties and potential applications of natural cellulose fibers from cornhusks. Green. Chem. 7(4), 190-195.

Reddy, K.O., Maheswari, C.U., Rajulu, A.V., Guduri, B.R., 2009a. Thermal degradation parameters and tensile properties of Borassus flabellifer fruit fiber reinforcement. J. Reinf. Plast. Compos.28(18), 2297-2301.

Reddy, K.O., Maheswari, C.U., Reddy, D.J.P., Rajulu, A.V., 2009b. Thermal properties of Napier grass fibers. Mat. Let.63(27), 2390-2392.

Reddy, K.O., Maheswari, C.U., Shukla, M., Song, J.I., Rajulu, A.V., 2013. Tensile and structural characterization of alkali treated Borassus fruit fine fibers. Compos. Part B: Eng. 44(1), 433-438.

Reddy, K.O., Ashok, B., Reddy, K.R.N., Feng, Y.E., Zhang, J., Rajulu, A.V., 2014. Extraction and characterization of novel lignocellulosic fibers from Thespesia lampas plant. Int. J. Polym. Anal. Ch. 19(1), 48-61.

Ribeiro, A., Pochart, P., Day, A., Mennuni, S., Bono, P., Baret, J. L., Mangin, I., 2015. Microbial diversity observed during hemp retting. Appl. Micro. Biotech. 99(10), 4471-4484.

Rong, M.Z., Zhang, M.Q., Liu, Y., Yang, G.C., Zeng, H.M., 2001. The effect of fiber treatment on the mechanical properties of unidirectional sisal-reinforced epoxy composites. Comp. Sci. Tech., 61(10), 1437-1447.

Rwawiire, S., Tomkova, B., 2015. Morphological, thermal, and mechanical characterization of Sansevieria trifasciata fibers. J. Nat. Fibers. 12(3), 201-210.

Sathishkumar, T.P., Navaneethakrishnan, P., Shankar, S., Rajasekar, R., 2013. Characterization of new cellulose sansevieria ehrenbergii fibers for polymer composites. Compos. Interf., 20(8), 575-593.

Sanjay, M.R., Arpitha, G.R., Naik, L.L., Gopalakrishna, K., Yogesha, B., 2016. Applications of natural fibers and its composites: An overview. Nat. Res. 7(03), 108.

Sanjay, M.R., Madhu, P., Jawaid, M., Senthamaraikannan, P., Senthil, S., Pradeep, S., 2018. Characterization and properties of natural fiber polymer composites: A comprehensive review. J. Clean. Prod. 172, 566-581.

Sanjay, M.R., Siengchin, S., 2018. Natural Fibers as Perspective Materials. KMUTNB Int. J. Appl. Scie. Tech. 11, 233.

Saravana Bavan, D, Mohan Kumar, G.C., 2010. Potential use of natural fiber composite materials in India. J. Rein. Plas. Compos. 29(24), 3600-3613.

Saravanakumar, S.S., Kumaravel, A., Nagarajan, T., Sudhakar, P., Baskaran, R., 2013. Characterization of a novel natural cellulosic fiber from Prosopis juliflora bark. Carbohydr. Polym. 92(2), 1928-1933.

Saravanakumar, S.S., Kumaravel, A., Nagarajan, T., Moorthy, I.G., 2014a. Investigation of physico-chemical properties of alkali-treated Prosopis juliflora fibers. Int. J. Polym. Anal. Ch. 19(4), 309-317.

Saravanakumar, S.S., Kumaravel, A., Nagarajan, T., Moorthy, I.G., 2014b. Effect of chemical treatments on physicochemical properties of Prosopis juliflora fibers. Int. J. Polym. Anal. Ch. 19(5), 383-390.

Sarikanat, M., Seki, Y., Sever, K., Durmuşkahya, C., 2014. Determination of properties of Althaea officinalis L.(Marshmallow) fibres as a potential plant fibre in polymeric composite materials. Compos. Part B: Eng. 57, 180-186.

Segal, L., G.J.M.A., Creely, J. J., Martin Jr, A. E., Conrad, C.M., 1959. An empirical method for estimating the degree of crystallinity of native cellulose using the X-ray diffractometer. Text. Res. J. 29(10), 786-794.

Seki, Y., Sarikanat, M., Sever, K., Durmuşkahya, C., 2013. Extraction and properties of Ferula communis (chakshir) fibers as novel reinforcement for composites materials. Compos. Part B: Eng. 44(1), 517-523.

Senthamaraikannan, P., Saravanakumar, S.S., Arthanarieswaran, V.P., Sugumaran, P., 2016. Physico-chemical properties of new cellulosic fibers from the bark of Acacia planifrons. Int. J. Polym. Anal. Ch 21(3), 207-213.

Senthamaraikannan, P., Kathiresan, M., 2018. Characterization of raw and alkali treated new natural cellulosic fiber from Coccinia grandis. L. Carbohydr. Polym. 186, 332-343.

Sepe, R., Bollino, F., Boccarusso, L., Caputo, F., 2018. Influence of chemical treatments on mechanical properties of hemp fiber reinforced composites. Compos. Part B: Eng. 133, 210-217.

Sghaier, A.E.O.B., Chaabouni, Y., Msahli, S., Sakli, F., 2012. Morphological and crystalline characterization of NaOH and NaOCl treated Agave americana L. fiber. Ind. Crops. Prod. 36(1), 257-266.

Shahidi, S., Wiener, J., Ghoranneviss, M., 2013. Surface modification methods for improving the dyeability of textile fabrics. In Eco-Friendly Textile Dyeing and Finishing. InTech.

Shanmugasundaram, N., Rajendran, I., Ramkumar, T., 2018. Characterization of untreated and alkali treated new cellulosic fiber from an Areca palm leaf stalk as potential reinforcement in polymer composites. Carbohydr. Polym. 195, 566-575.

Siengchin, S., 2017. Editorial corner–a personal view Potential use of'green'composites in automotive applications. eXPR. Poly. Let. 11(8), 600-600.

Singha, A.S., Thakur, V.K., 2009. Synthesis and characterizations of silane treated Grewia optiva fibers. Int. J. Polym. Anal. Ch. 14(4), 301-321.

Singha, A.S., Thakur, V.K., Mehta, I.K., Shama, A., Khanna, A.J., Rana, R.K., Rana, A.K., 2009. Surface-modified Hibiscus sabdariffa fibers: Physicochemical, thermal, and morphological properties evaluation. Int. J. Polym. Anal. Ch. 14(8), 695-711.

Sisti, L., Totaro, G., Vannini, M., Celli, A., 2018. Retting Process as a Pretreatment of Natural Fibers for the Development of Polymer Composites. In Lignocellulosic Composite Materials (pp. 97-135). Springer, Cham.

Sonnier, R., Taguet, A., Ferry, L., Lopez-Cuesta, J.M., 2018. Flame Retardancy of Natural Fibers Reinforced Composites. In Towards Bio-based Flame Retardant Polymers(pp. 73-98). Springer, Cham.

Sreekala, M.S., Kumaran, M.G., Thomas, S., 1997. Oil palm fibers: Morphology, chemical composition, surface modification, and mechanical properties. J. Appl. Polym. Sci. 66(5), 821-835.

Sreenivasan, V.S., Somasundaram, S., Ravindran, D., Manikandan, V., Narayanasamy, R., 2011. Microstructural, physico-chemical and mechanical characterisation of Sansevieria cylindrica fibres–An exploratory investigation. Mater. Des. 32(1), 453-461.

Takacs, E., Wojnárovits, L., Borsa, J., Földváry, C., Hargittai, P., Zöld, O., 1999. Effect of γ-irradiation on cotton-cellulose. Rad. Phy. Chem. 55(5-6), 663-666.

Thomas, M. G., Abraham, E., Jyotishkumar, P., Maria, H. J., Pothen, L. A. Thomas, S. Nanocelluloses from jute fibers and their nanocomposites with natural rubber: Preparation and characterization. International Journal of Biological Macromolecules 81 (2015) 768–777.

Tamburini, E., León, A. G., Perito, B., Mastromei, G., 2003. Characterization of bacterial pectinolytic strains involved in the water retting process. Envir. Micro. 5(9), 730-736.

Tappi, T., 1993. Ash in wood, pulp, paper and paperboard: combustion at 525 C. TAPPI test methods T, 211.

Tóth, T., Borsa, J., Takács, E., 2003. Effect of preswelling on radiation degradation of cotton cellulose. Rad. Phy. Chem 67(3-4), 513-515.

Truong, M., Zhong, W., Boyko, S., Alcock, M., 2009. A comparative study on natural fibre density measurement. J. Tex. Ins. 100(6), 525-529.

Tserki, V., Zafeiropoulos, N.E., Simon, F., Panayiotou, C., 2005. A study of the effect of acetylation and propionylation surface treatments on natural fibres. Compos. Part A: Appl. Sci. Manuf., 36(8), 1110-1118.

Thakur, V.K., Thakur, M.K., Singha, A.S., 2013. Free radical–induced graft copolymerization onto natural fibers. Int. J. Polym. Anal. Ch. 18(6), 430-438.

Thakur, V.K., Thakur, M.K., 2014. Processing and characterization of natural cellulose fibers/thermoset polymer composites. Carbohydr. Polym. 109, 102-117.

Thakur, V.K., Thakur, M.K., Gupta, R.K., 2014. Graft copolymers of natural fibers for green composites. Carbohydr. Polym. 104, 87-93.

Uehara, T., Sakata, I., 1990. Effect of corona discharge treatment on cellulose prepared from beech wood. J. Appl. Poly. Sci. 41(7- 8), 1695-1706.

Valadez-Gonzalez, A., Cervantes-Uc, J. M., Olayo, R., Herrera-Franco, P.J., 1999. Chemical modification of henequen fibers with an organosilane coupling agent. Compos. Part B: Eng. 30(3), 321-331.

Väisänen, T., Das, O. and Tomppo, L., 2017. A review on new bio-based constituents for natural fiber-polymer composites. J. Clean. Prod. 149, 582-596.

Van der Werf, H. M., Turunen, L., 2008. The environmental impacts of the production of hemp and flax textile yarn. Ind. Crops. Prod. 27(1), 1-10.

Varma, I.K., Krishnan, S.A., Krishnamoorthy, S., 1989. Effect of chemical treatment on density and crystallinity of jute fibers. Text. Res. J. 59(6), 368-370.

Ververis, C., Georghiou, K., Christodoulakis, N., Santas, P., Santas, R., 2004. Fiber dimensions, lignin and cellulose content of various plant materials and their suitability for paper production. Ind. Crops. Prod. 19(3), 245-254.

Wang, B., Panigrahi, S., Tabil, L., Crerar, W., 2007. Pre-treatment of flax fibers for use in rotationally molded biocomposites. J. Reinf. Plast. Compos. 26(5), 447-463.

Xie, Y., Xiao, Z., Militz, H., Hao, X., 2017. Silane Coupling Agents Used in Natural Fiber/Plastic Composites. Handbook of Composites from Renewable Materials, Functionalization, 4, 407

Zegaoui, A., Ma, R., Dayo, A. Q., Derradji, M., Wang, J., Liu, W., Xu, Y., 2018. Morphological, mechanical and thermal properties of cyanate ester/benzoxazine resin composites reinforced by silane treated natural hemp fibers. Chin. J. Chem. Eng. 26(5),1219-1228.