

A New Treatment Method for Cellulose-Rich Fibers Toward High Performance Bio-composite Applications

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Abstract

This article reports a new chemical treatment method to extract rich cellulosic fibers from date palm agro-residues. The technique comprises a multistep process which is quite unique, efficient, sustainable, and capable of producing high-performance natural date palm fibers. In specific, it consists of three consecutive steps: dewaxing, acetylation, and mercerization, which are carried out in a strict sequence. Each step is designated to achieve a specific goal in preparation for the next step. In addition, procedures have been adjusted from its initial state by incorporating input parameters that are configured experimentally to optimize outputs via the design of experiment approach. The method has been applied in two variants with respect to heating mechanism: conventional heat-assisted and microwave heat-assisted sources. An experimental characterization of mechanical, physical, and thermal properties of the treated fibers exhibited a significant enhancement over the untreated counterparts which confirms the potency of the new process.

Introduction

Plenty of agro-residues are produced at various phases of the plant life cycle. Common practice is that such contaminants are destined for landfill or incineration, resulting in far-reaching and long-lasting environmental problems (Navas et al. 2015; Baniyas et al. 2020). Utilization of agro-residues as fillers in the manufacture of bio-based composites is a viable route which contribute to the environment preservation but requires an adoption of appropriate sustainable waste management practices. Although several researchers have explored the potential use of natural fibers as an alternative to replace synthetic fibers as reinforcement agent, less focus is paid to the use of agro-residue-based fillers. Date palm agro-residues are perhaps the most abundant (40 Kg/tree per annum) and least expensive (\$0.1/Kg) agro-residues worldwide, which are still not well-utilized. Natural fibers extracted from date palm agro-residue are characterized by their modest material properties and cost effectiveness relative to other types like coir, hemp and sisal fibers (Rajeshkumar et al. 2020; Nassar et al. 2021). Proper integration of date palm fibers with numerous polymeric materials resulted in improved mechanical, physical, thermal, and acoustic properties of developed bio-composites (Masri et al. 2018; Dixit et al. 2020). Undoubtedly, manufacturing natural filler reinforced composites utilizing date palm waste is a favorable path with high environmental benefits (Bellatrache et al. 2020).

Efforts are still being directed to explore appropriate techniques to resolve the fundamental issue related, in particular, to low mechanical properties of natural fibers. A successful upgrade of their strength will grant natural fibers an opportunity to be utilized as competitive reinforcements for virgin or recycled polymers (Macedo et al. 2019; Rosa et al. 2020). Fibers acquired from different sources of agro-residues are contributing to a wide variety of bio-composites, where each type of bio-composite can be tailored to a specific application depending on required material properties and performance. Conversely, there are inherent drawbacks of natural fibers such as poor thermal resistance, considerably lower dimensional stability, high absorbent of moisture and incompatibility with matrix materials (Gholampour and Ozbakkaloglu 2020; Lin et al. 2020). Therefore, surface treatment of natural fibers has been subjected to

a variety of techniques in order to achieve sustainable and efficient fibers from natural resources in manufacturing bio-composites that is economical when used to design industrial components (Cruz and Fangueiro 2016; Karimi and Taherzadeh 2016). Various chemical treatment methods such as mercerization (alkali treatment), silane, malleated and isocyanates coupling agents, permanganates, acetylation, benzylation and acylation (Tanasă et al. 2020; Alzebdeh and Nassar 2021) are adopted to modify surface of fiber to enhance its characteristics as reinforcing agent in polymers. The modification processes aim for eliminating contaminants and impurities, and increase the crystalline biomass structure of the fiber, mainly cellulose. The crystalline content and rough surface are required characteristics of natural fibers that play a critical role in developing bio-composites (Yusriah et al. 2014).

Most of reported fiber treatment methods in the literature aimed to modify surface of fiber to achieve a better compatibility between fiber and synthetic polymer. While this work includes this objective, it further illustrates the development of a new modification process for strengthening natural fibers extracted from date palm agro-residues and improving its overall characteristics. The developed method is a systematic multi-step chemical treatment technique (process) to achieve improved fiber properties. Processing the fiber using microwave-electrical power accelerates the fiber modification and significantly enhances the overall characteristics and in specific the fiber tensile strength and modulus.

Materials And Methods

Materials

Date palm agro-residues generated from pedicles have been collected from a local farm. Initially, water retting was performed by immersing the pedicels in water for a week after removing its bark. This allows the pedicel bundles to absorb water, making it easier to be separated into individual date palm fiber(s) (DPF). Next, the fibers were extracted manually from the retted pedicels and heated in an oven at 105°C for 24 hours to evaporate moisture.

Multistep Chemical Treatment

In all designated steps of the current treatment method which is filed for international patent application (Nassar et al. 2020), a mixture of solid to liquid (solvents) (*S:L*) ratio (1:20) was used to ensure a sufficient chemical reaction taking place at the fiber's surface. The treatment process comprises three phases: dewaxing, acetylation, and mercerization (alkalization) of the fiber assisted by either conventional heating (CH) or accelerated heating (AH) source. The following subsections expand these steps in more details.

Step 1: Surface Dewaxing

The main purpose is to eliminate impurities and contaminants like wax, pectin, and others residing on the surface of untreated fiber. The untreated fibers were dewaxed with ethanol/water (1:1, v/v) in a Soxhlet apparatus. In case of conventional heating, the procedure was conducted at 75°C for 4 hours. The

dewaxed fibers were then dried at 105°C for at least 24 hours. In the accelerated heating form, a microwave heat source is used. The method of weight loss and FTIR analysis were used to optimize the microwave power and time variables. To achieve this, three samples, each subjected to one of three microwave power levels (low ≈250 W, medium ≈450 W, and high ≈900 W) at a constant temperature of 75 ± 2°C were analyzed. After completion, the average weight loss was recorded after being verified by FTIR at an incremental time step of 10 minutes. Thus, the optimal time and microwave power that achieve the highest wax and impurities removal is determined.

Step 2: Acetylation

This step aims to bleach and sanitize the fibers to further improve its processability and anti-microbial properties. For CH option, fibers were soaked in acidified sodium chloride solution (30% acetic acid, 5 % sodium chloride) in Soxhlet at 105°C for 2 hours. For AH option, microwave power and time were optimized against the acetylation degree as shown in Fig. 1. Acetylation degree (r) is defined as the ratio of peak intensity (A) at 1724 cm^{-1} (acetyl group) to the peak intensity (B) at 1026 cm^{-1} (cellulose backbone). Measurements were taken every 5 minutes and the acetylation degree was recorded, based on the average of three replications.

Step 3: Mercerization (alkalization)

In case of mercerization using conventional heating, an alkali solution of 5% NaOH was prepared, and the acetylated fibers were soaked in the boiling solution at 100°C for 45 minutes. In case of microwave assisted mercerization, processing parameters were optimized using Design of Experiment (DoE). The selected output in the design is crystallinity degree of fibers (Cr.%) and the independent variables are NaOH concentration (wt. %), microwave power (W), and treatment time (min) at constant temperature (80°C) for all experimental runs. The analysis was carried out using Full Spherical Central Composite Design option within Stat-Ease Design-Expert® software (v 12.0.3.0) including six replicates of central levels to evaluate the reliability and repeatability of outputs. Since microwave accelerates the elimination of amorphous biomass from the fiber, preliminary trials were performed to evaluate the process factors to avoid disruption of fiber and retain its integrity. Otherwise, over extraction of hemicellulose and lignin would weaken the structural integrity of fiber and generate a pulp that is rich in cellulose. Consequently, the levels of the design parameters were selected as shown in Table 1. After each experimental run, treated fibers were washed using distilled water several times until pH neutral was attained.

Table 1
Range of levels for parameters used in microwave assisted mercerization.

Process Factors	Factors Range (Coded & Actual levels)		
	Lower (-1)	Center (0)	Higher (+ 1)
NaOH Concentration (wt.%)	5	7.5	10
Microwave Power (W)	250	500	750
Microwave Time (min)	10	15	20

Figure 2 (a) outlines a graphical summary of underlying steps within the treatment method along with the impact of each step on microstructure of fiber as shown in Fig. 2 (b). Detailed evaluation of process parameters and results will be presented in Sect. 3.

Characterization

The analysis of functional groups of the chemical changes of the fibers was obtained by FTIR spectrometer. Samples were scanned over the range 400 to 4000 cm^{-1} with a total of 64 scans at a resolution of 4 cm^{-1} at an ambient temperature ($25\text{ }^{\circ}\text{C}$). Figure 3 shows a flow chart for a structured process to determine the fiber chemical composition following a reported extractive method in the literature (Jiang et al. 2010; Wang et al. 2015; Watkins et al. 2015). First, the untreated and treated fibers are dried at 105°C for 24 hours. After that, the samples, each sample weighs approximately 2 g, are prepared independently for the biomass composition analysis. The untreated fiber was firstly dewaxed using benzene/ethanol. Boiling water was used for further decomposition of water-soluble substances. Diluted ammonium oxalate solution assisted in extraction of pectin. Further, boiling Potassium hydroxide solution was responsible for separating the hemicellulose. Lignin was extracted by boiling Hydrogen Peroxide (H_2O_2) solution. Eventually, the volume of cellulose was determined by subtracting the total measured chemical composition from 100%. The method was performed on three samples in each step and the average value was recorded.

To evaluate crystallinity of the untreated and treated fibers, chopped fibers were examined at an ambient temperature through step scanning with an X-ray powder diffractometer. Measurements were carried out at 40 kV and 20 mA, with a detector mounted on a goniometer scanning scale from 5° to 60° , at a scanning speed of $5^{\circ}\text{ min}^{-1}$ using monochromatic $\text{CuK}\alpha$ radiation ($\lambda = 1.5406\text{ nm}$). The crystallinity degree then was then measured using an empirical method which is explained in (Aly et al. 2012). For mechanical characterization, estimation of cross-sectional area of fiber was accomplished using an optical microscope with a magnification power of 300-1000x. Then, tensile test was carried out according to ASTM-C155 using a universal testing machine with a 5 kN load cell at a constant speed of 1 mm/s and gauge length of 28. Five samples of each category (UT, CH, AH) were tested, and results were recorded. Thermogravimetric and Derivative Thermogravimetric Analyses (TGA/DTG) of the untreated

and treated fibers were carried out using a thermogravimetric analyzer (TGA Q 500 TA Instrument). Samples were placed in an aluminum pan and heated in the range from 20°C to 550°C, at a rate of 10°C/min under inert atmosphere of N₂ with a flow rate of 50 mL/min to ensure full decomposition of sample.

Results And Discussion

Process Optimization

Surface Dewaxing

Microwave assisted dewaxing has been configured based on wax and impurities extraction rate as verified by using FTIR. Figure 4 shows that rate of dewaxing is directly proportional to the microwave power, and as such medium and high microwave powers are recommended for higher rate of wax extraction. The low power is also capable to extract wax but at a lower rate. Therefore, a medium power was selected as it is sufficient to improve the rate of wax removal with lower energy consumption while maintaining fiber structure and preventing damage that could occur from excessive microwave power. Overall, it is found that the optimal time which maximizes fiber dewaxing is 60 minutes.

Also, FTIR as shown in Fig. 5, indicates that the major internal chemical composition of the fiber was not altered because of dewaxing which preserved the fiber structural integrity. FTIR spectra for all experiments were almost consistent, confirming that no additional absorption bands were applied to the fiber. The characteristic peaks refer to an individual component of natural fiber such as wax, cellulose, hemicellulose, lignin, pectin and water molecules. The peak at around 1730 cm⁻¹ is due to carbonyl (C = O) stretching of acetyl groups in hemicellulose and ester bonds of carboxyl groups in lignin and hemicellulose (Lu and Oza 2013). The complete dewaxing is verified by the disappearance of the (C = O) after one hour of treatment time using microwave assisted heating. The same process requires 4 hours in case of conventional heating assisted dewaxing. The use of Hydroxyl group (OH) is substantial for modifying the reactivity of fiber surface for further chemical crosslinking with matrix material.

Acetylation

The acidified sodium chloride solution is used to sterilize, purify, and bleach the dewaxed fibers. Figure 6 displays the degree of acetylation as a function of time at three microwave power levels. It is noted that the maximum degree of acetylation is attained at high microwave power after 20 minutes of reaction time. However, medium heating power was adopted for this analysis as it has an acceptable degree of acetylation that would not weaken the structural integrity of the fiber as opposed to the use of high power. Acetylation decreases the hygroscopic characteristic of natural fiber and become more stable due to the replacement of OH groups in DPF by acetyl groups and thus enhance hydrophobicity. This also can be indicated by OH group intensity reduction as shown in Fig. 7. Based on the FTIR of AH option, it is also found that the spectra were not potentially changed after treatment except for intensity reduction of the

peak at 2855 cm^{-1} which is related to hemicellulose content (Bezazi et al. 2014). Thus, the aim of acylation to bleach fibers and remove portion of amorphous biomass is confirmed. This finding was not observed in the case of CH which indicate superiority of using microwave to bleach the fiber and achieve higher yield in a short time.

Mercurization

After applying DoE analysis, the optimal conditions to maximize the crystallinity degree were found at lower concentrations of NaOH and medium levels of microwave power for a time of 20 minutes. Results indicate that high concentrations of NaOH and high microwave power tend to degrade the crystallinity degree. This was reflected by massive damage to the cellulose structure due to the increased removal rate of non-cellulosic biomass. The FTIR spectra for final yield of the treatment method is presented in Fig. 8. Bands 1336 and 1316 cm^{-1} show significant differences in their absorbances. These are the bands assigned to both crystalline celluloses (Cel I and Cel II), and after treatment those band became more intense in the treated DPF than in the untreated fibers especially in case of microwave assisted heating. In addition, the absorption peaks at 3328 cm^{-1} attributed to OH stretching vibration, 2900 cm^{-1} attributed to CH stretching vibration, 1430 cm^{-1} attributed to CH_2 and OCH plane bending vibration, 1370 cm^{-1} attributed to CH deformation vibration, and 896 cm^{-1} attributed to anomeric carbon vibration can be observed in the spectra of treated DPF. All these are characteristic peaks of cellulose (brown shaded in Fig. 8), indicating extraction of highly rich cellulose fiber (Zhao et al. 2015). Hence, higher strength, improved hydrophobicity is achieved compared to the untreated fiber. The differences between microwave and conventional heat assisted treatment can be depicted as the higher intensity of the cellulose backbone (1030 cm^{-1}) and OH group (around 3325 cm^{-1}), the lower intensity of hemicellulose peak (2855 cm^{-1}). Therefore, the modified DPF using microwave power is anticipated to be more compatible with the base polymer resulting in improved bio-composite.

In summary, the current technique consists of three steps that should be applied in the specified sequence to achieve an optimal performance, including configured and optimized input variables. Each step is taken to accomplish a particular objective in preparation for the next step; surface dewaxing is used to extract both wax and impurities to expose the wall cell to react with imposed functional groups; acetylation is conducted to bleach and sanitize the fiber to improve its processability in the mercurization step and removes the amorphous cellulose. Mercurization is used to further remove/alleviate amorphous biomass and regulate the penetration level of NaOH-based ionic solution to react with OH group in hemicellulose and lignin to change fiber surface characteristics and increase the content of crystalline cellulose. However, considering the processing time and yield it is found that AH treatment produces better bleached and purified DPF in a shorter time ($\sim 1.5\text{ hr}$) with a reduction of ($\sim 80\%$) compared to the CH treatment. Figure 9 portrays a flow-chart illustrating the multi-step chemical treatment method of DPF using CH and AH routes.

Biomass Composition

Biomass composition (%) of both untreated and treated fibers after each step using both CH and AH procedures are shown in Fig. 10. The untreated DPF contains mainly cellulose, lignin and hemicellulose and small quantities of wax, impurities, and pectin. After dewaxing, it is found that mostly wax, impurities, and pectin are removed from DPF in both CH and AH with slightly larger removal in case of AH as indicated by the higher percentage of remaining biomass after dewaxing. Due to acetylation, cellulose content is further increased because of the partial extraction of amorphous biomass which is mainly lignin. The most crucial step is mercerization in which NaOH is used to remove the optimal percentage of amorphous biomass without affecting fiber integrity. After biomass composition analysis, it is shown that cellulose content is increased and DPF became rich in cellulose because of reduction of the extractives and amorphous biomass removal. Moreover, removal of the amorphous material is more evident in the case of AH due to the ability of alkali solution to penetrate inside fiber subsurface.

Morphological

Scanning Electron Microscope (SEM) micrographs were obtained and interpreted to evaluate the effect of the treatment steps on fiber morphology and internal structure. The untreated surface in Fig. 11 clearly shows a cover of wax layer and other impurities. After dewaxing, roughness of this outer layer is reduced due to wax and impurities removal as confirmed by FTIR. A major removal of impurities and less rough surface are observed in the case of AH compared to CH case, which indicates a higher efficiency of the microwave power to clean the fiber surface. On the other hand, dewaxing did not result in any fiber damage or deterioration, as it only took place at the fiber surface. The unaltered cross section due to dewaxing as confirmed by SEM images (Fig. 11) reveals that the fiber structure is well preserved.

Figure 12 demonstrates the surface morphology and cross section of the acetylated fibers. Compared to the untreated and dewaxed fiber, it is noted that AH-acetylation eliminates the deposits residing at fiber surface and therefore makes a smoother surface. The microwave power stimulated acetylation to bleach surface and extract further amorphous biomass including pectin and lignin. Whereas CH-acetylation showed no major effect to the dewaxed surface. Similarly, the magnified micrographs of cross section show a minor change in case of AH to the fiber interior subsurface where the elementary fiber become stuffed and squeezed which might be due to biomass removal. This action was not clearly observed in the case of CH, as the microwave power forced the functional groups to penetrate the fiber interior structure.

Figure 13 shows images of the fiber surface and cross section of treated DPF in comparison to untreated DPF after implementation of mercerization, which is the final step of the developed process. It is evident from these micrographs that the surface topography of DPF looks rougher and cleaner in this step of treatment compared to the previous steps. This is because mercerization eliminates a substantial amount of amorphous biomass mainly lignin and hemicellulose. In addition, the fiber backbone structure is preserved, and the fiber surface becomes fibrillated supporting compatibility with synthetic polymer thereby improving fiber/polymer interfacial adhesion. It is worthwhile to mention that the treatment process carefully controls the removal of hemicellulose and lignin to prevent fiber pulping (pure

dissimilated cellulose) and maintain fiber structural integrity. In this regard, SEM micrographs confirmed the potency of developed method to enhance the surface topography and preserves the fiber integrity. SEM of cross-section morphology reveals that circular bundles which form structural strength/integrity of fiber were potentially affected by the treatment method. As clearly shown in the corresponding micrographs in Fig. 13, the holes are collapsed and closed due to full removal of amorphous biomass. As lignin was removed, the middle lamella became more packed which cause a gradual elimination of microvoids as indicated in the square in the magnified images.

Crystallinity Degree

Figure 14 displays XRD patterns of untreated and treated DPF taken after completion of entire treatment process. Results show that both CH and AH procedures did not adversely influence the physical structure of DPF, since both XRD patterns show identical peaks at 21° and 18° for I_{002} and I_{am} , respectively. Clearly, the crystallinity degree has been increased owing to the treatment process which partially remove the amorphous biomass, mostly hemicellulose and lignin and promote the crystalline cellulosic content. In specific, crystallinity degree is increased by 32% when microwave is used as a heating source, whereas conventional heating improves crystallinity by 19%. Such a difference is due to the higher removal rate of non-cellulosic biomass in AH technique.

Hydrophobicity

Referring to Table 2 and comparing to untreated DPF, it is obvious that treated DPF has achieved a significant drop in water absorption especially in the AH case. This is due to the removal of biomass that absorbs water and/or moisture at a higher rate compared to cellulose (Balla et al. 2019). Even though CH-based process was able to cut water absorption in half, the AH treated fiber is often known to be the optimal option to minimize water absorption as the cellulose content is further increased. The use of higher hydrophobic fiber improves the fiber/polymer compatibility that will ultimately enhance the bio-composite strength and durability.

Table 2
Summary of average values of physical and mechanical properties of DPF before and after treatment.

	Water Absorption (%)	S_{UT}^* (MPa)	E^{**} (GPa)	δ_{EL}^{***} (%)
UT	10	80	7	0.016
CH	5	297	13	0.033
AH	3	444	24	0.027

* S_{UT} : Tensile strength; **E: Elastic modulus; *** δ_{EL} : Elongation at fracture

Tensile Properties

Among mechanical properties of interest are the tensile strength, modulus, and elongation at break of DPF, which were substantially increased in case of treated fibers owing to the treatment effect. Figure 15 exhibits stress-strain diagrams for the untreated and treated fibers. The elastic modulus, tensile strength and elongation at break can be extracted from the plot. It is evident from the figure that fiber processed by AH option yielded the maximum strength, but with a reduced ductility. Maximum ductility is achieved in the CH derived fiber case, but its strength is roughly 33% less than the AH-derived fiber. The trade-off between strength and ductility is a typical material behavior which is observed in this work. Clearly, the AH-based fiber is more brittle compared to the CH-based fiber as modified microstructure contribute to less ductility and high strength.

Table 2 summarizes the average values of physical and mechanical properties of untreated and treated fibers. The results presented in the table confirm that the multi-step chemical treatment process with microwave-assisted heating results in major increase in elastic modulus and tensile strength. The tensile strength improved by around 5.5-fold compared to the untreated fibers when AH is used and by 3.7-fold compared to untreated fiber when CH is used. Similarly, the elastic modulus of treated fiber increased by 3.4 factor in relative to untreated fiber when AH is used and 86% increase in the case of CH option. A substantial improvement in strength and modulus is highly valuable for the manufacture of polymer-based bio-composites. Thanks to optimized content of ingredients (cellulose, lignin, hemicellulose) and the efficient elimination of nonbeneficial components (wax, impurities), as verified by SEM images (Sect. 3.3). The elimination of such unfavorable surface contaminants further decreases the discontinuity and structural defects of the fiber. The successful completion of the current treatment process with the aid of heating, the fibrils are rearranged along the main axis, resulting in an improved resistance to deformation along the fiber axis. The experimental findings suggest that the inclusion of hemicellulose and other forms of waxy compounds up to a certain threshold will expand fiber ductility.

The current DPF is compared in terms of strength with similar date-palm fibers and other types of fibers (extracted from different trees) which were treated with various forms of mercerization (Table 3). It can be concluded from the data presented in Table 3 with confidence that the new treatment procedure has accomplished a much better improvement in tensile properties.

Table 3
Effect of various treatment methods on tensile strength of natural fibers.

Fiber Type	Treatment Procedure	S _{UT} (MPa)		Reference
		Untreated	Treated	
DPF-CH	Figure 9.	80	297	Current Work
DPF-AH	Figure 9.	80	444	Current Work
Date Palm Petiole	2% NaOH	174	243	(Lassoued et al. 2018)
Date Palm Stem	1% NaOH	176	310	(Aly-Hassan et al. 2015)
Abaca	5% NaOH	755	847	(Cai et al. 2015)
Banana	5% NaOH	780	536	(Mejia Osorio et al. 2012)
Coconut	20% NaOH	186	281	(Arsyad 2017)
Flax	5% NaOH	630	627	(Aly et al. 2012)
Husk	5% NaOH	109	135	(Wang et al. 2014)
Sisal	2 M NaOH	284	386	(Kim and Netravali 2010)
Sugarcane	5% NaOH	170	205	(Hossain et al. 2014)

Thermo-Gravimetric Analysis (TGA)

Figure 16 shows the thermal decomposition of the untreated and treated DPF when heated from ambient temperature to 500°C. TGA displays the ratio of weight loss over the temperature range, and derivative thermogravimetric (DTG) displays the rate of mass loss, of which peaks can be interpreted as highest degree of temperature decomposition. TGA graphs show that both untreated and treated DPF experienced an initial weight loss under 110°C due to moisture and volatile substances removal. Furthermore, degradation of bio-mass components starts at 210°C and 240°C for untreated and treated DPF, respectively. Such results imply that the treatment process improves the thermal stability of fiber. In reference to DTG curves, the untreated DPF exhibited two major degradation peaks which are associated to hemicellulose and cellulose degradation, respectively (Filho et al. 2020). However, the treated fiber exhibited only one degradation peak which is related to cellulose. The minor shoulder appeared in case of CH at 285°C is attributed to degradation of hemicellulose. A similar shoulder was not detected in the case of AH as cellulose content was dominant over other types of biomasses and therefore hemicellulose degradation is overlapped within the cellulose range. Findings confirm the non-crystalline biomass elimination as the fiber degradation tend to follow similar degradation characteristics of pure cellulose (Lanjewar et al. 2019). Finally, lower char residue content of untreated DPF (20.5%) in comparison with treated DPF (26.6%) can be explained by higher volatilization of waxes, impurities, and other volatile biomasses from the untreated fiber.

Conclusion

This paper presents a novel process to chemically modify natural fibers derived from date palm pedicels agro-residues. The method comprises well-structured processing steps including optimization of input parameters in each step, and effectively uses microwave-assisted heating to accelerate modification of fiber. Furthermore, it completely purifies the fiber from wax, impurity, and odor thus, the process has the benefit of increased processability and decreased hydrophobicity of fiber which promote its durability and widen its horizon of applications. Thus, the reported method will promote new opportunities to develop value-added products using natural fibers sourced from date palm waste. The effective use of chemicals enhances the fiber surface tribology, which increases the efficiency of OH group to be functionalized for high-performance bio-composite synthesis.

Further research is required to extend the invented method to other lignocellulosic fibers to improve their characteristics and post-processability. Characterization of mechanical, physical thermal properties is crucial to prepare such a class of materials for commercialization and to generate new opportunities for developing and manufacturing load bearing products.

Declarations

Funding

No funding was received for conducting this study.

Conflict of Interest

The authors declare no competing financial interest.

Ethics approval

This article does not contain any studies with human participants or animals performed by any of the authors.

Consent for publication

Consent to publication was obtained from all individual participants included in the study.

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Figures

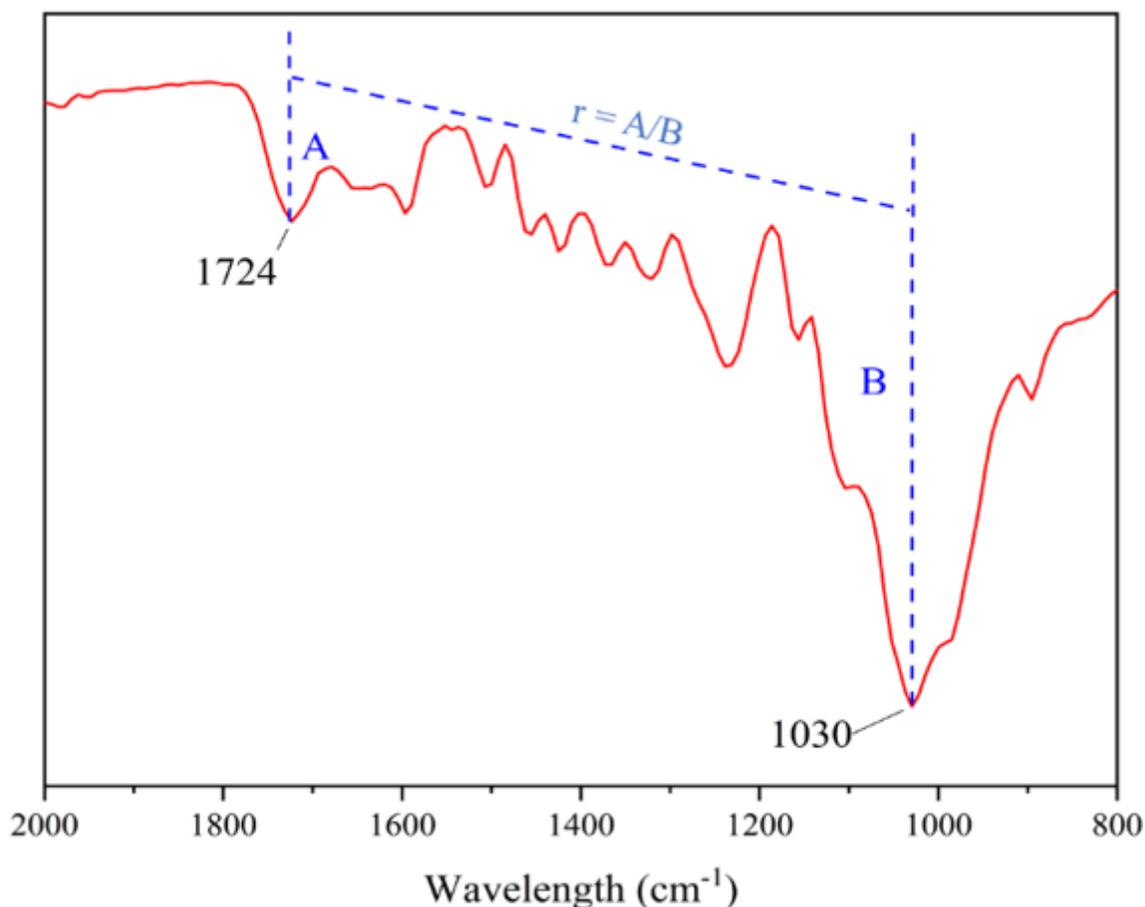


Figure 1

FTIR spectrum of acetylated DPF using AH option.

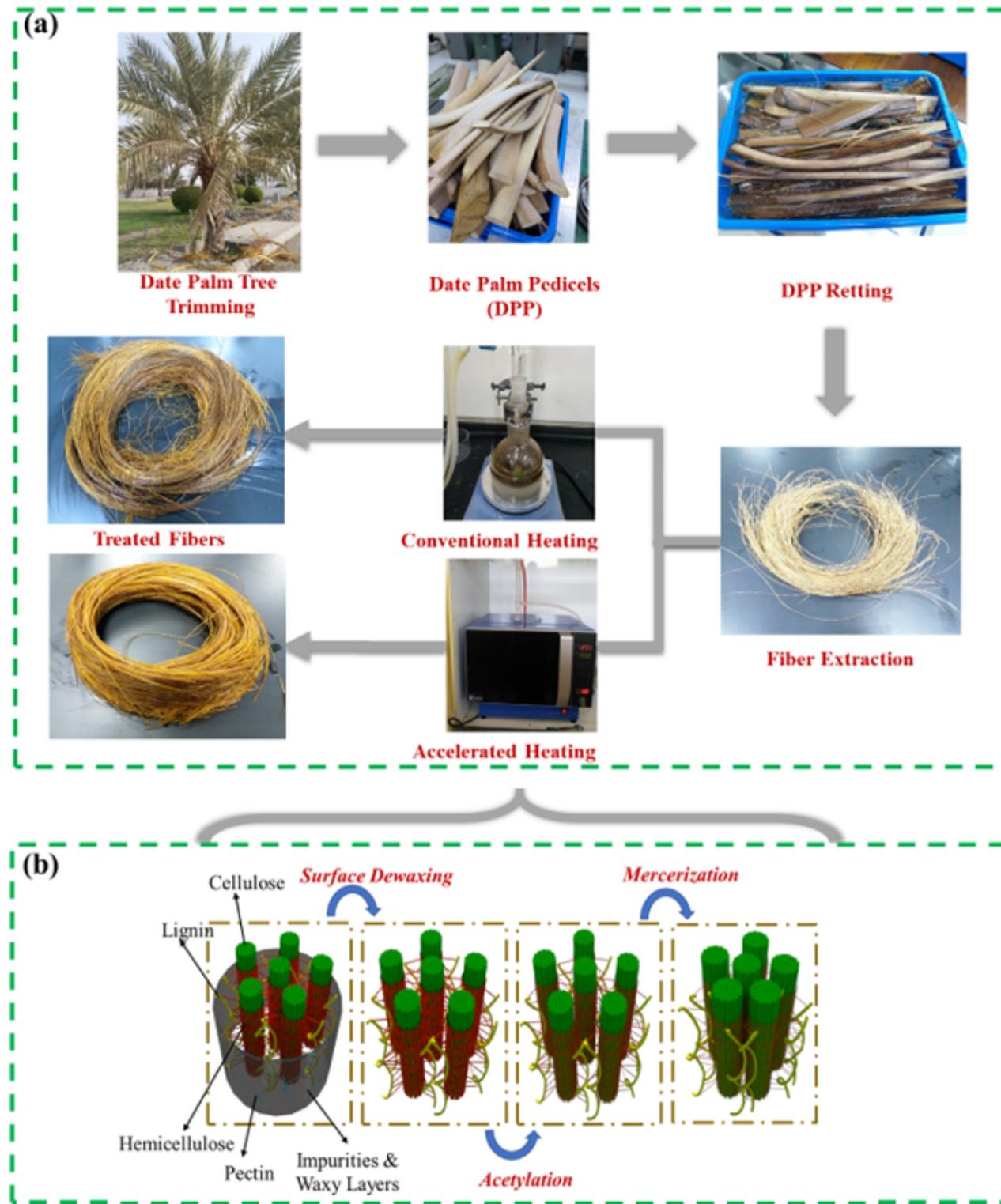


Figure 2

(a) Graphical presentation of the chemical treatment process, and (b) process effect on DPF microstructure.

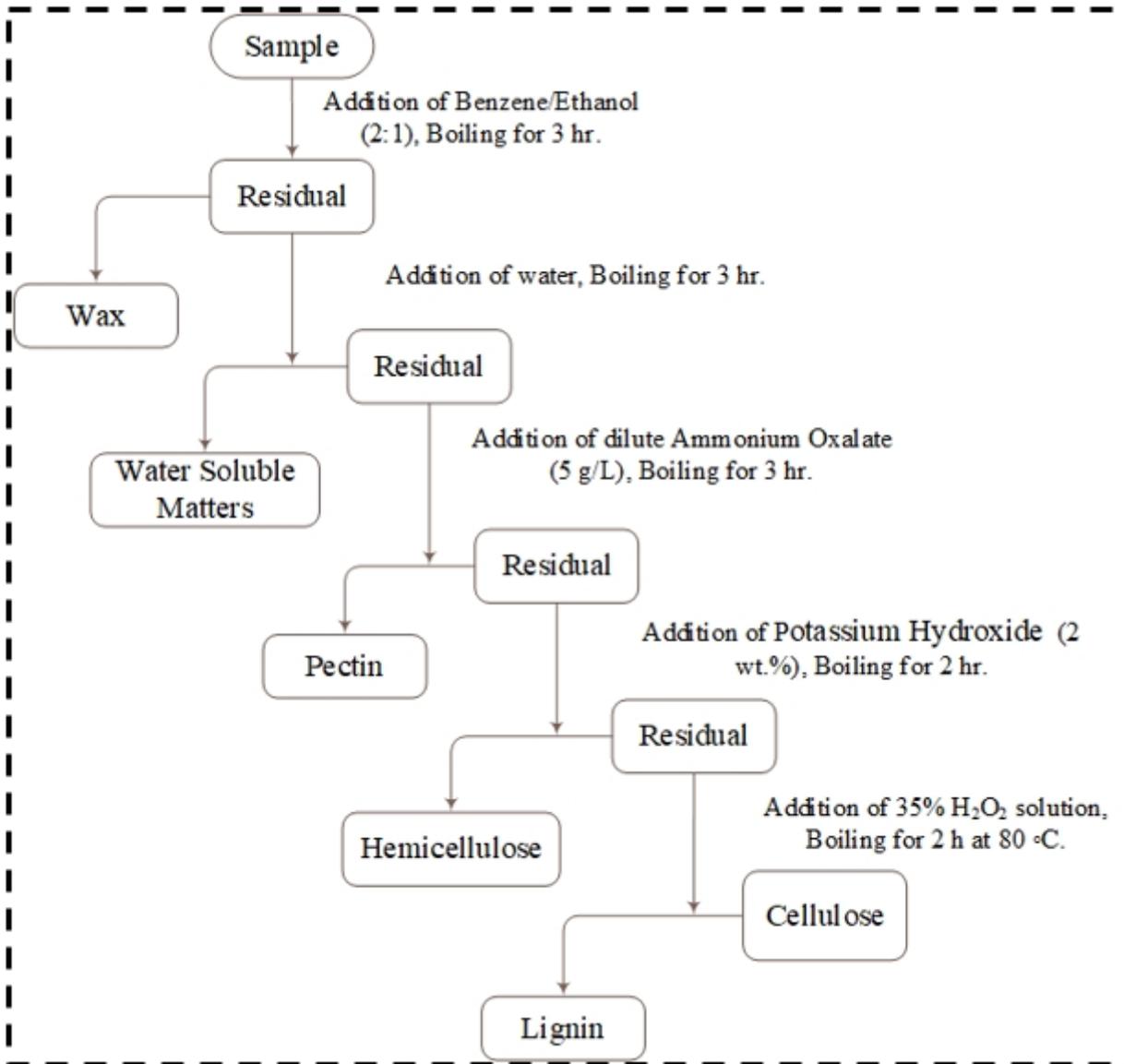


Figure 3

Schematic diagram for the biomass composition analysis.

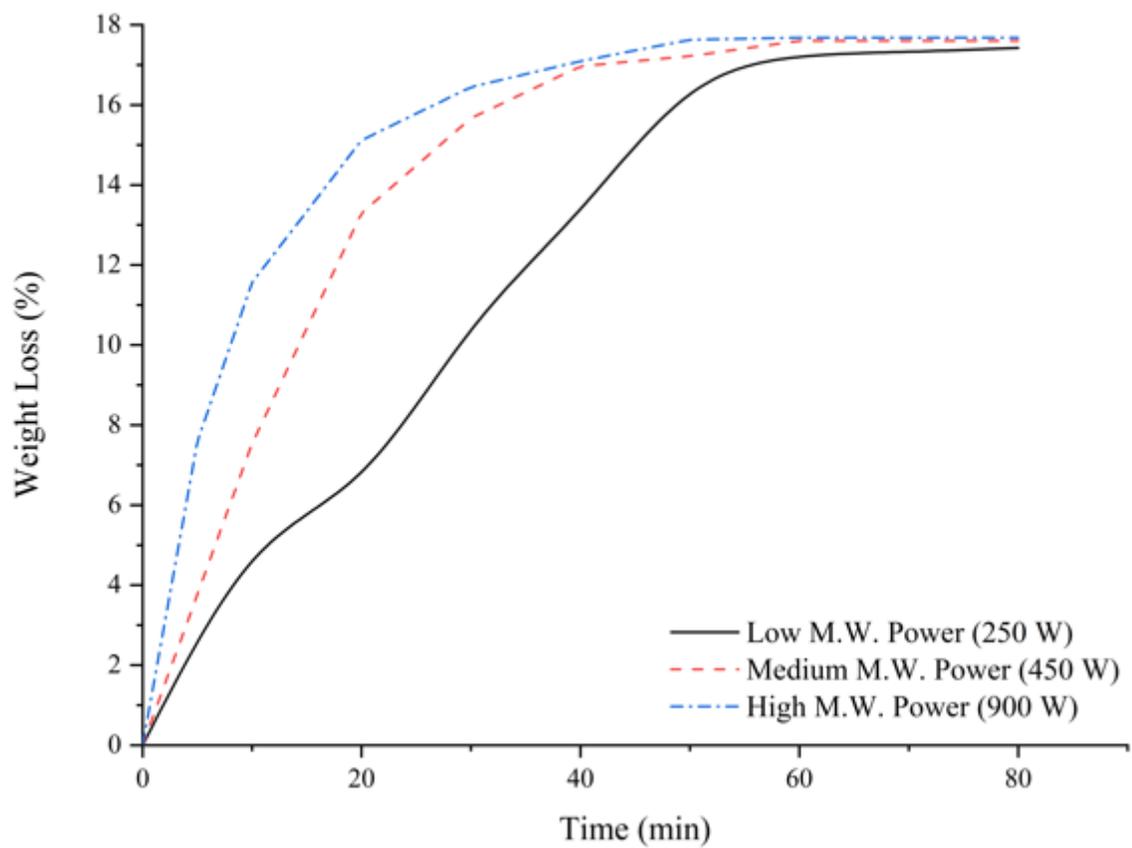


Figure 4

Effect of microwave power in AH dewaxing process on fiber.

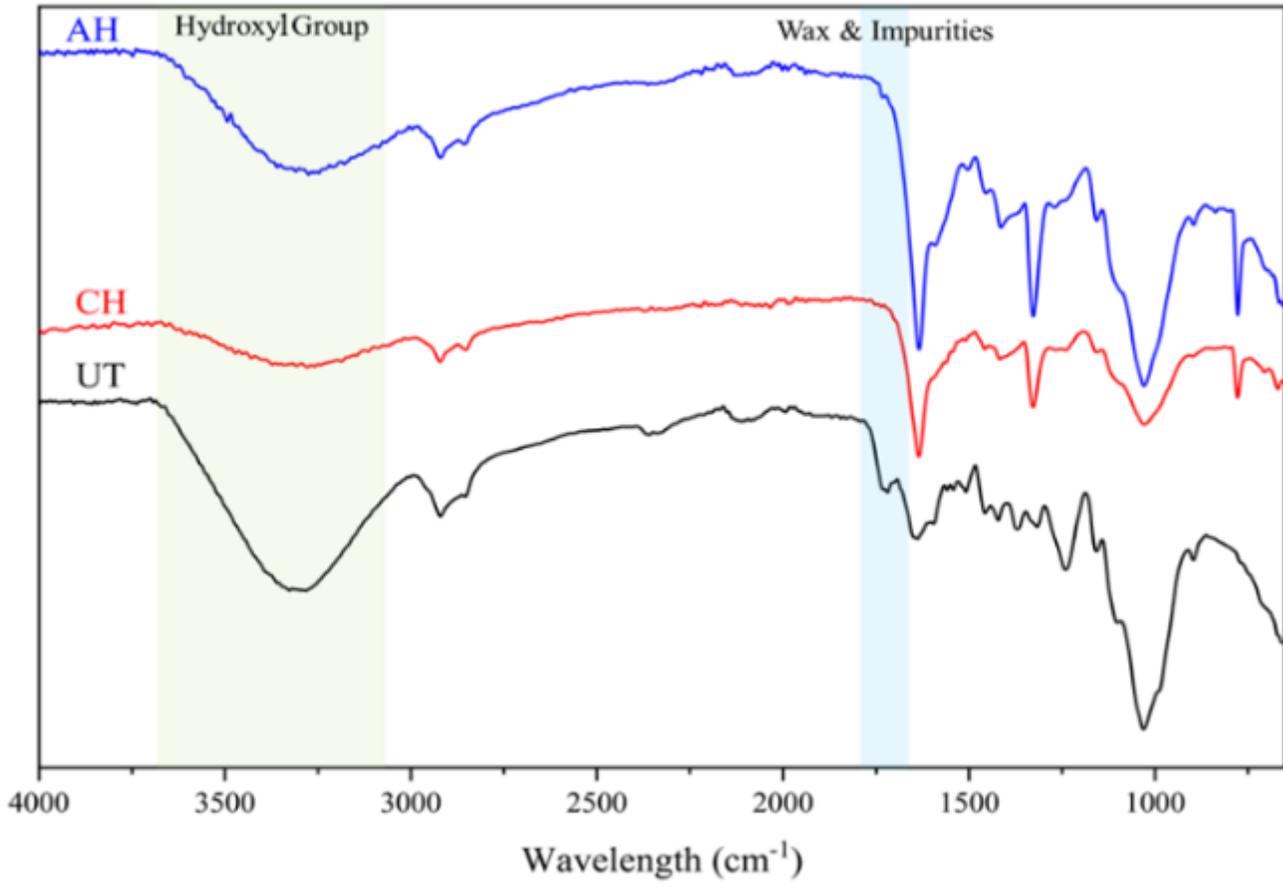


Figure 5

FTIR spectra of original (UT) and dewaxed (AH and CH) samples.

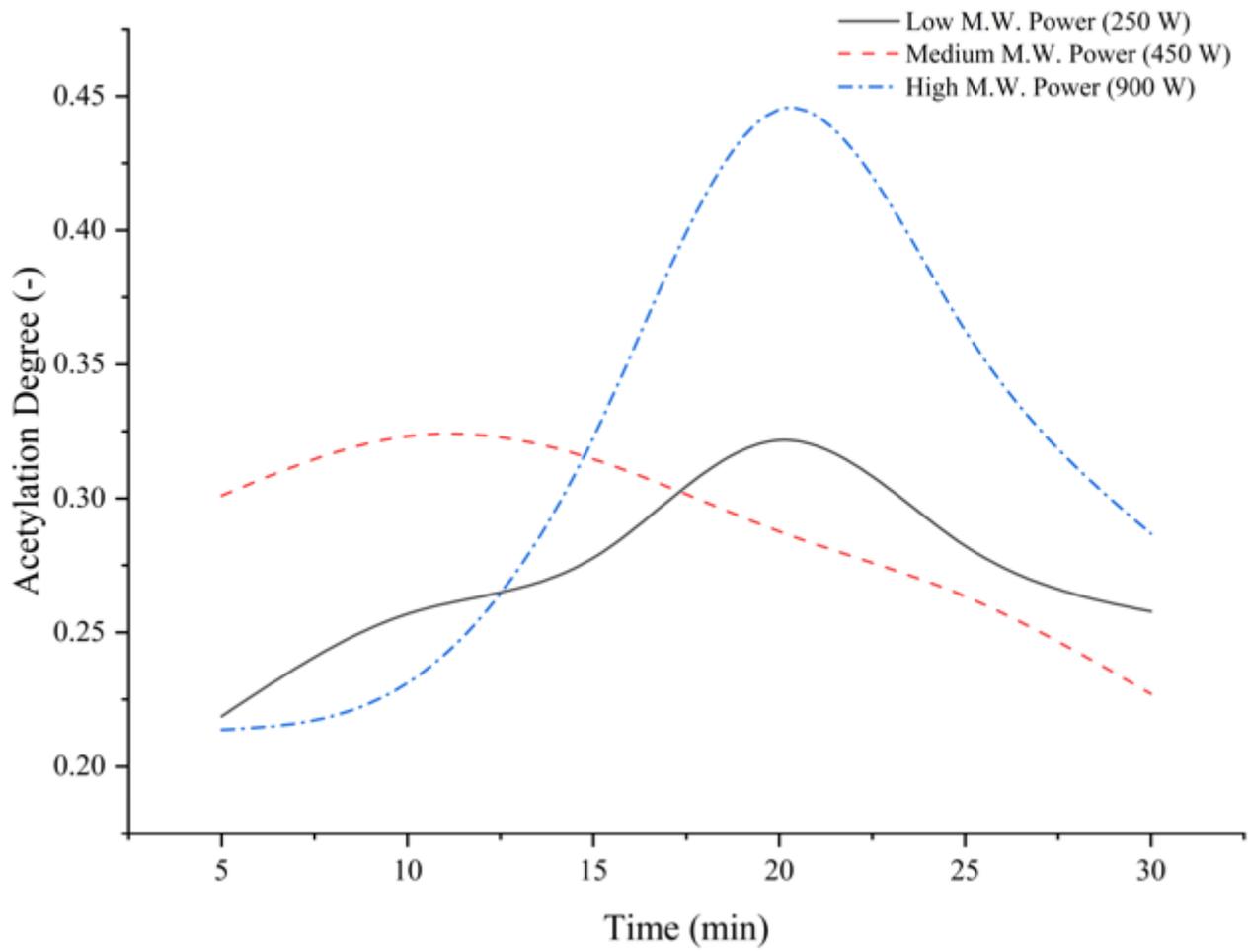


Figure 6

The time effect on acetylation.

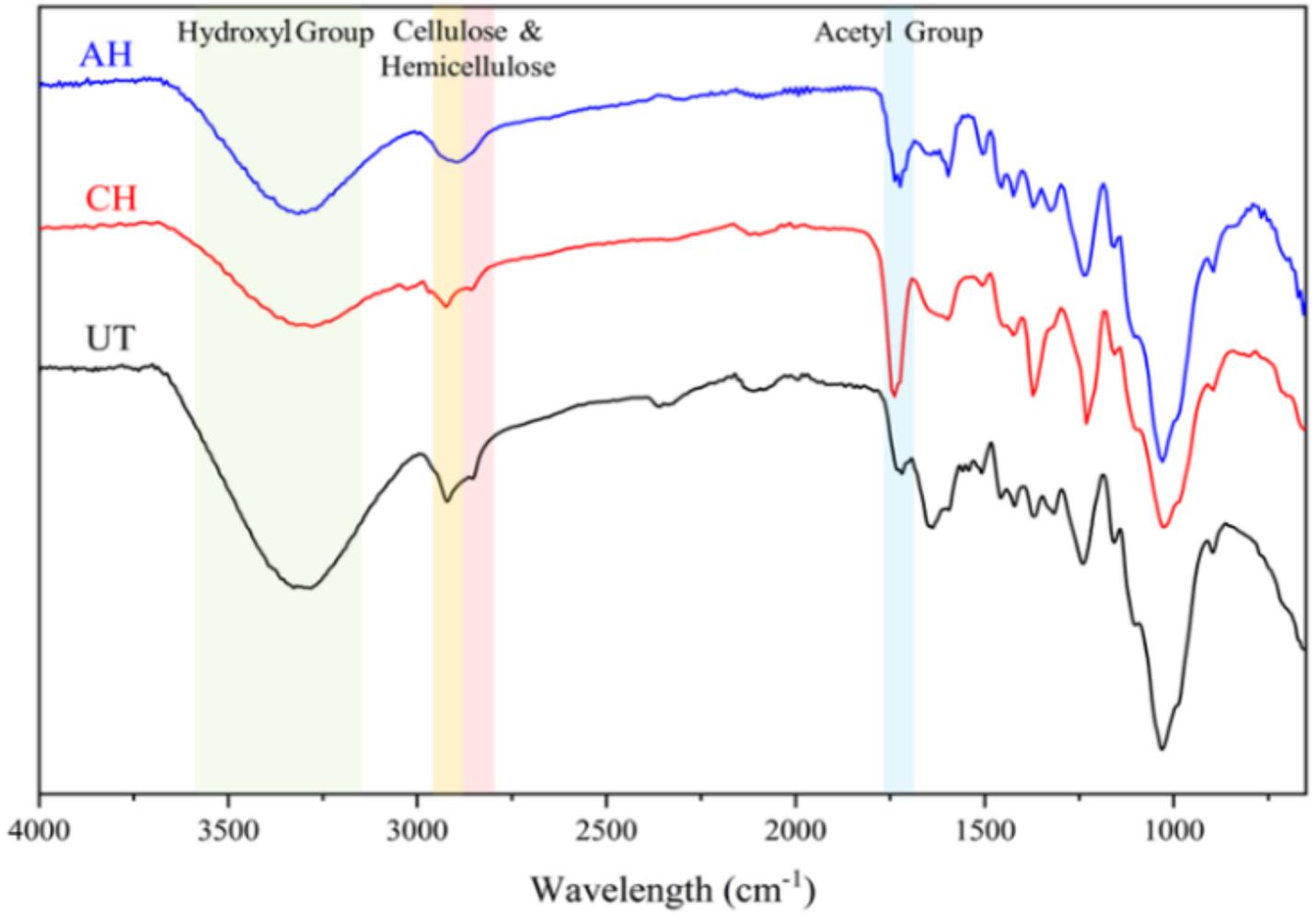


Figure 7

FTIR spectra of acetylated samples.

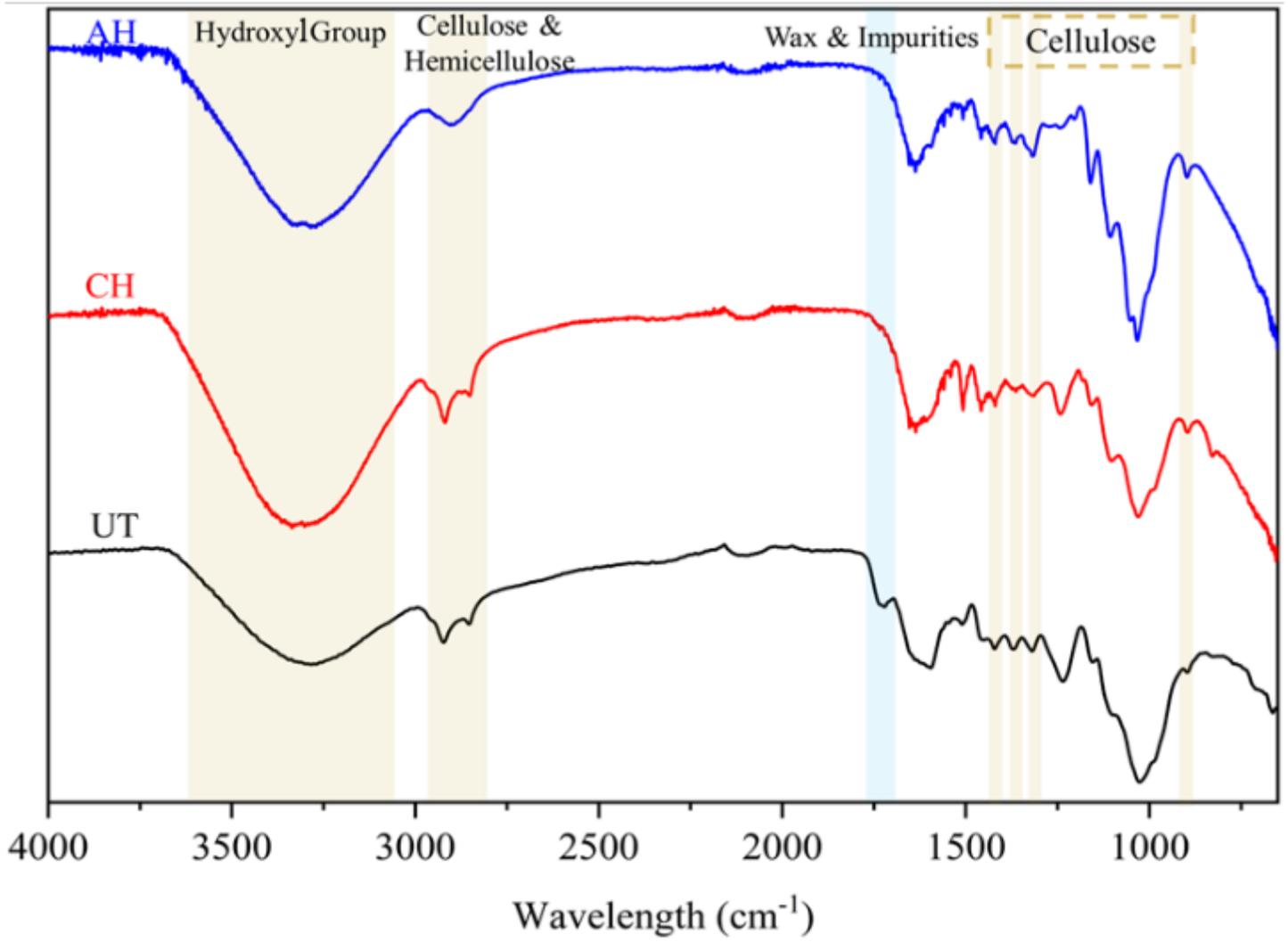


Figure 8

FTIR spectra of untreated and treated DPF in case of CH and AH.

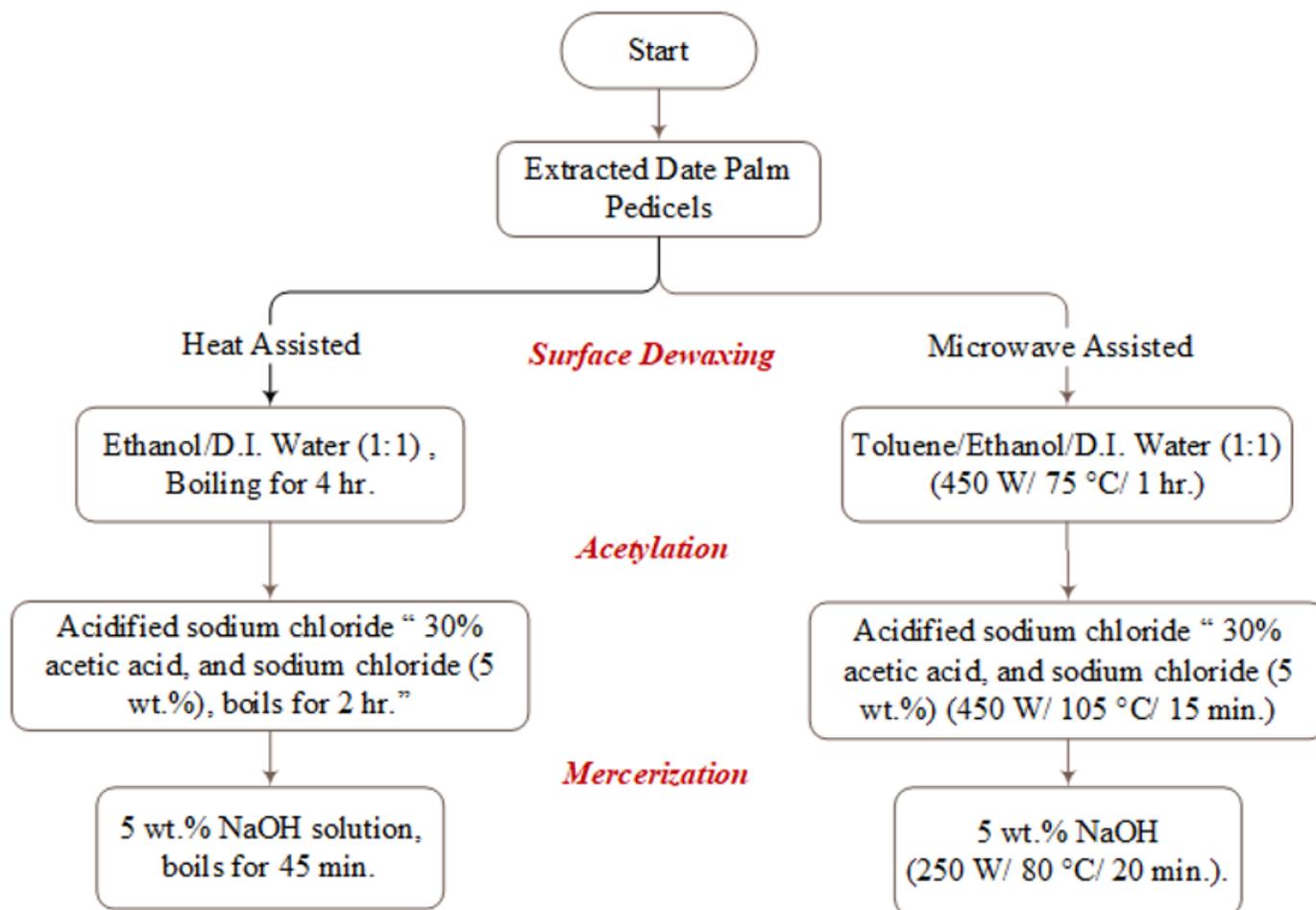


Figure 9

Flowchart illustrating new multistep chemical treatment method using CH and AH.

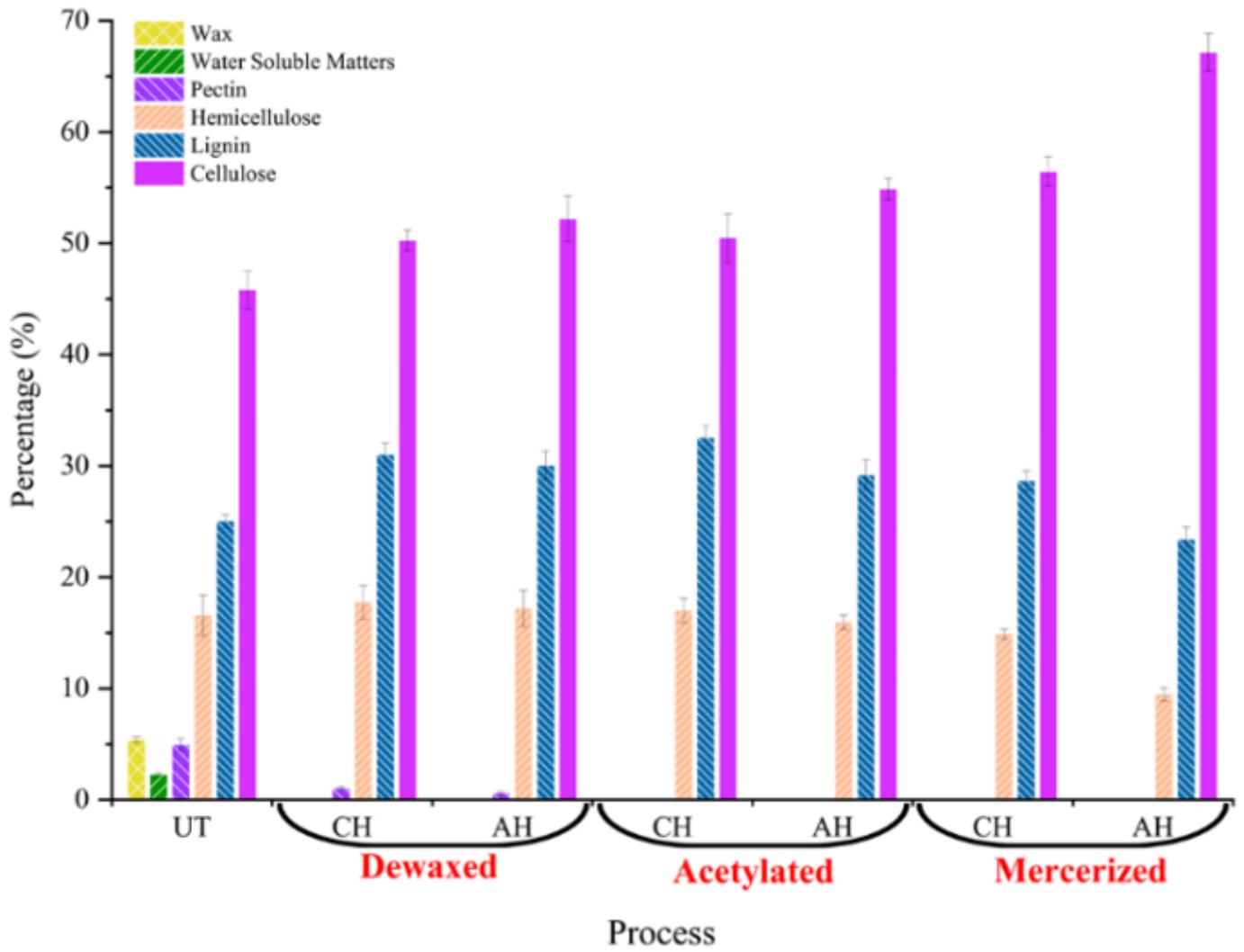


Figure 10

Biomass composition (%) of untreated and treated DPF after each treatment step.

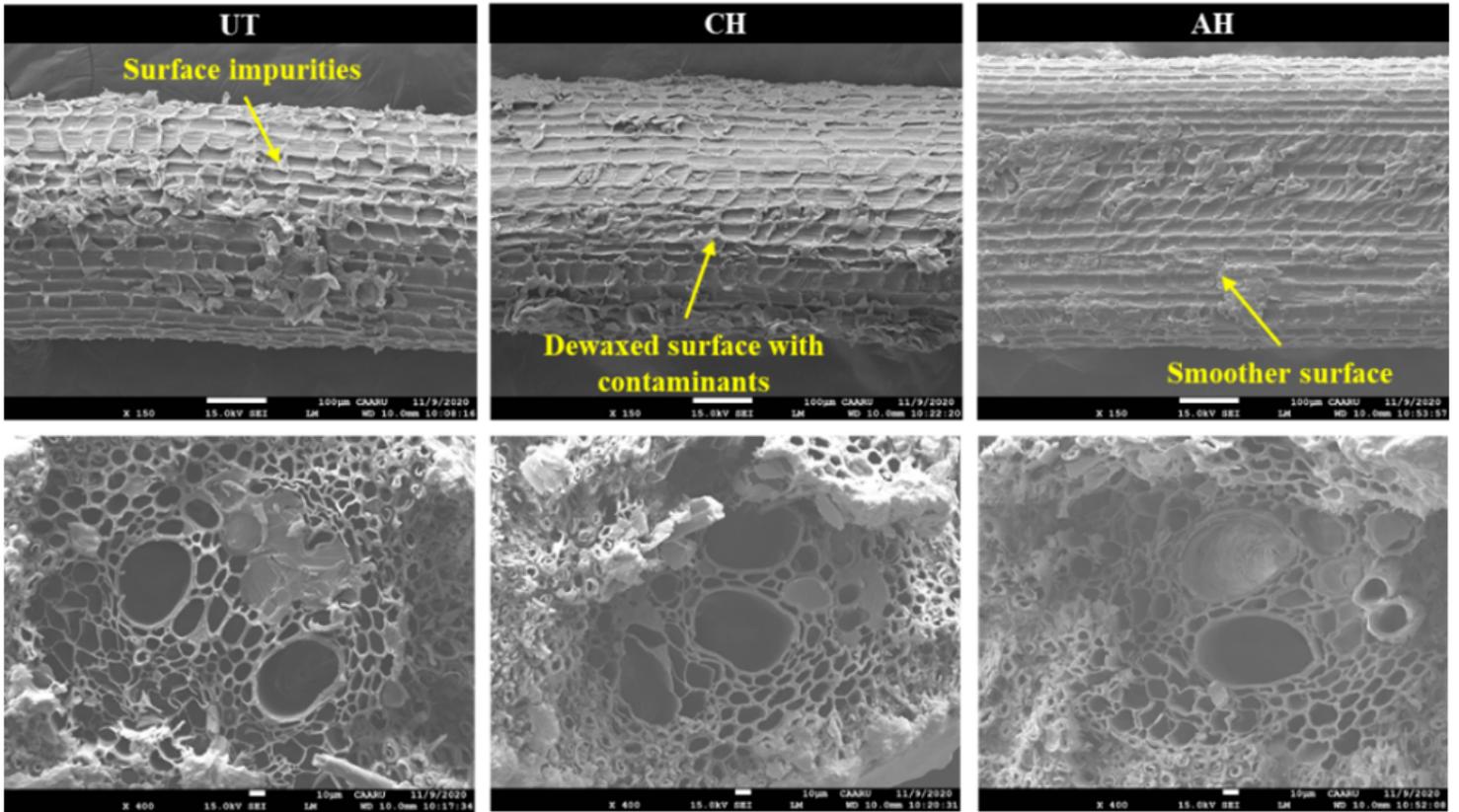


Figure 11

Untreated and Dewaxed DPF images using SEM.

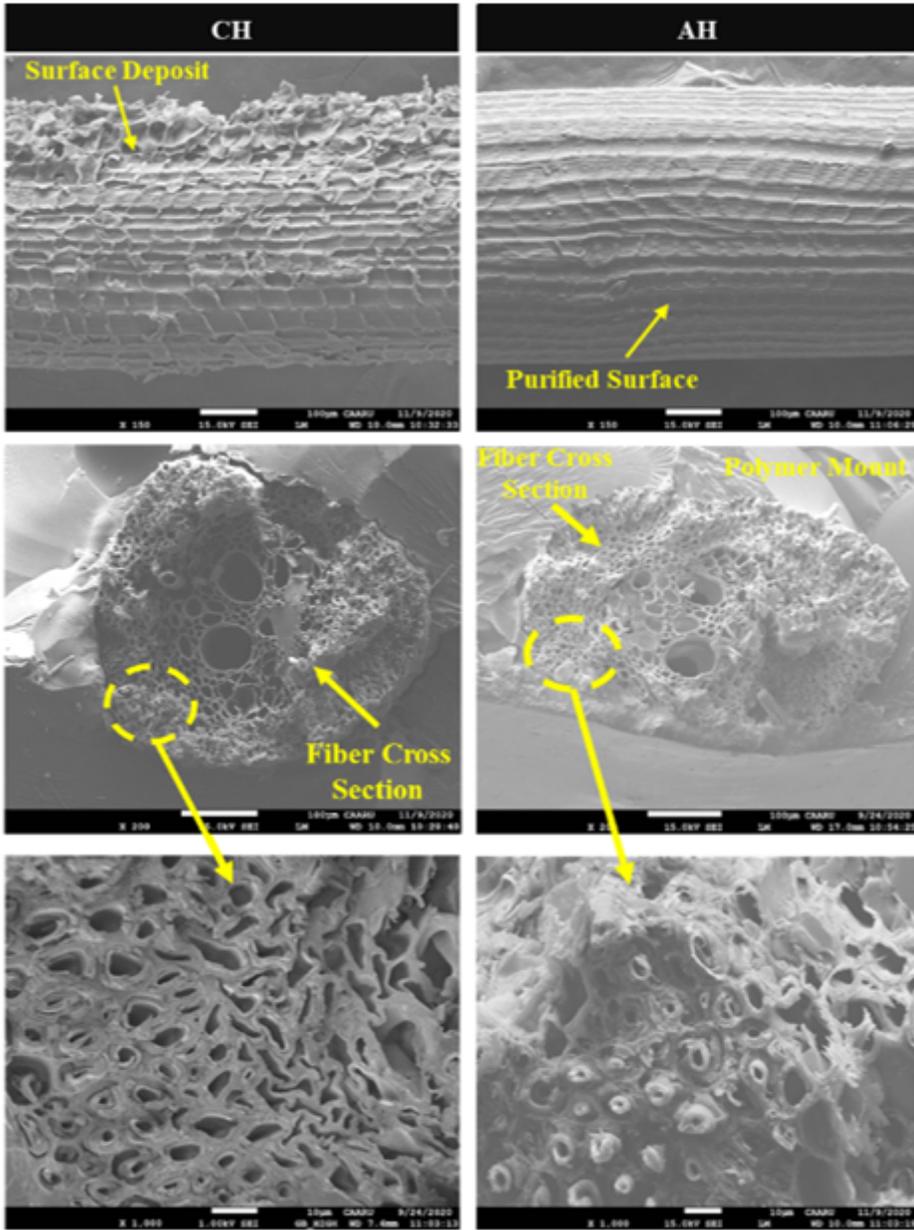


Figure 12

SEM images of the acetylated fiber (after dewaxing).

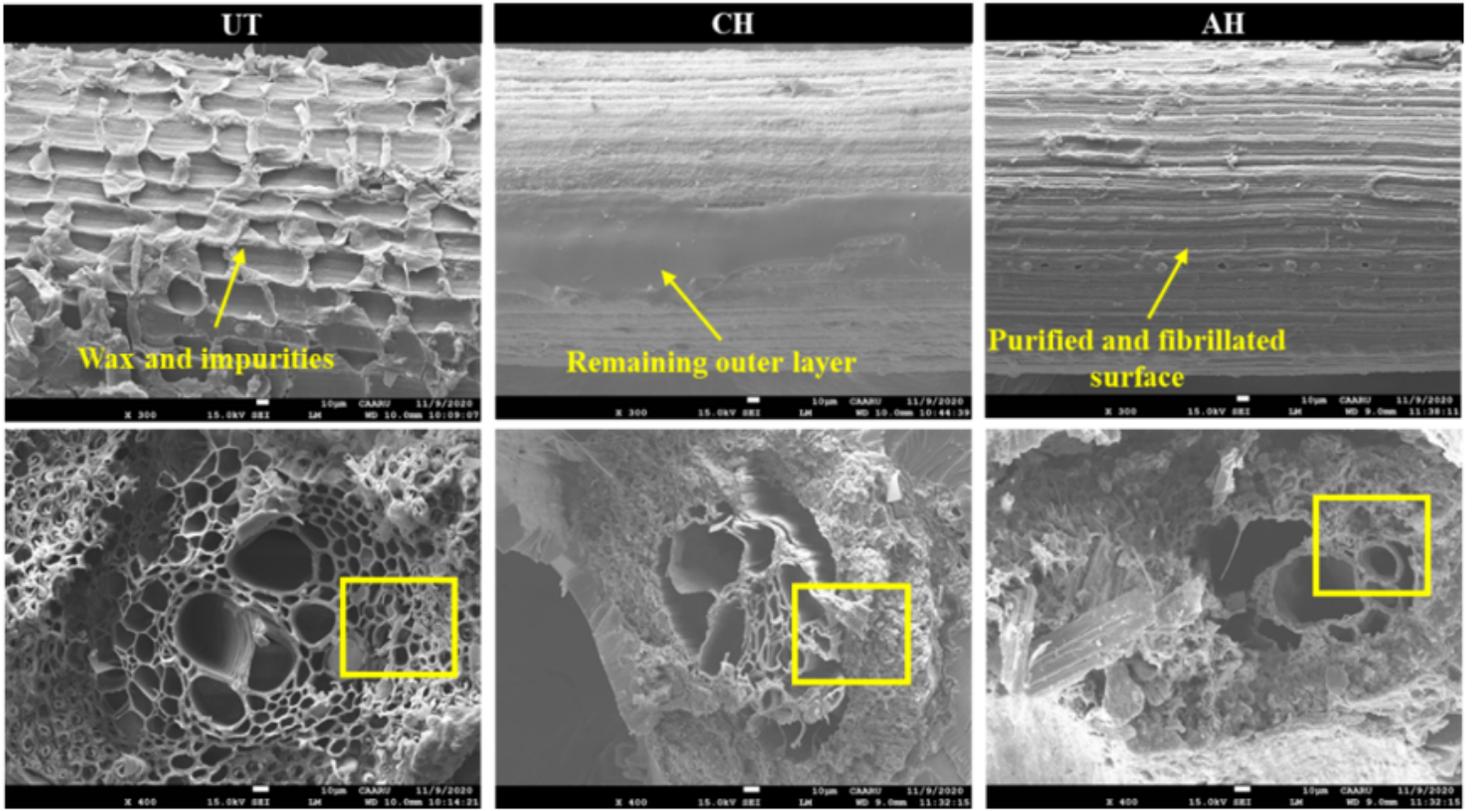


Figure 13

SEM images of untreated and treated (mercerized) DPF.

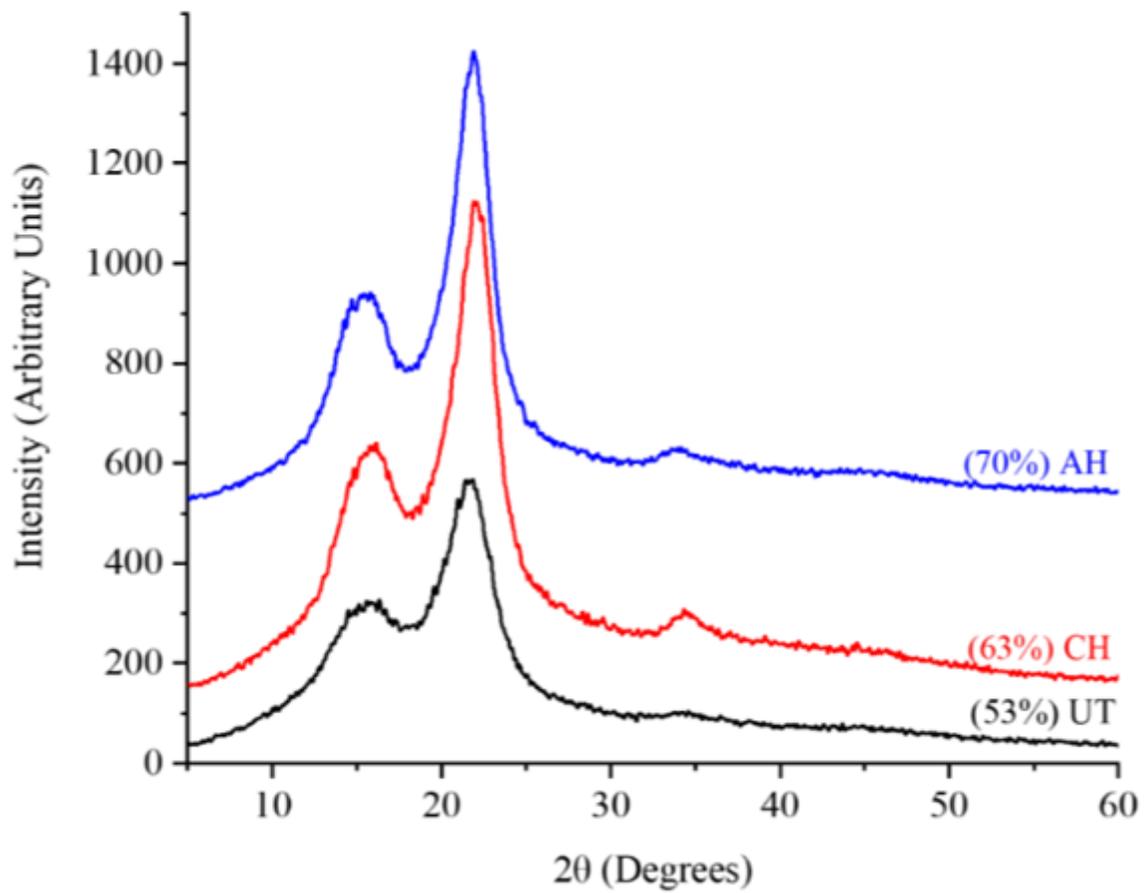


Figure 14

XRD of untreated and treated DPF and crystallinity degree.

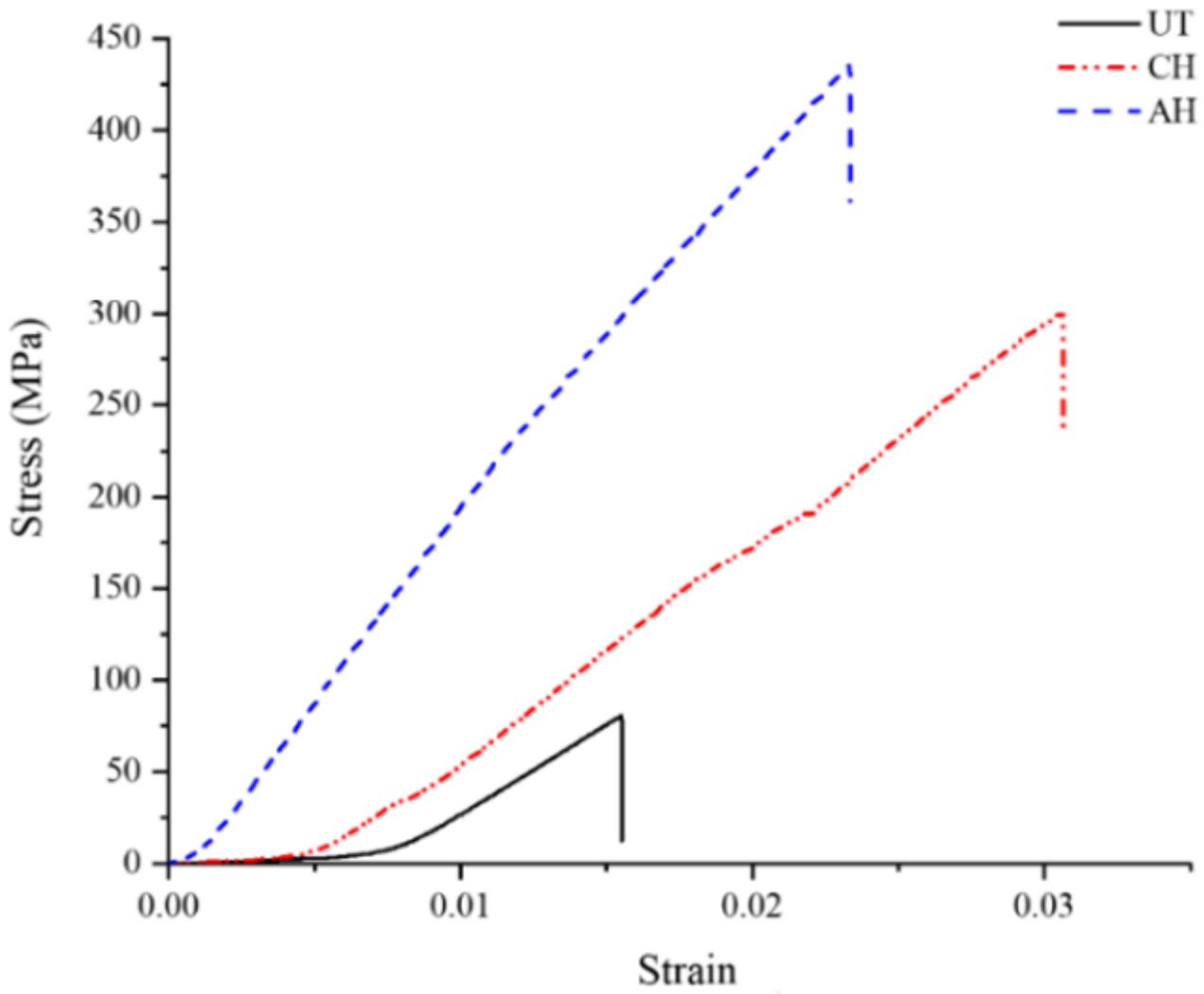


Figure 15

Stress-Strain curves of untreated and treated DPF.

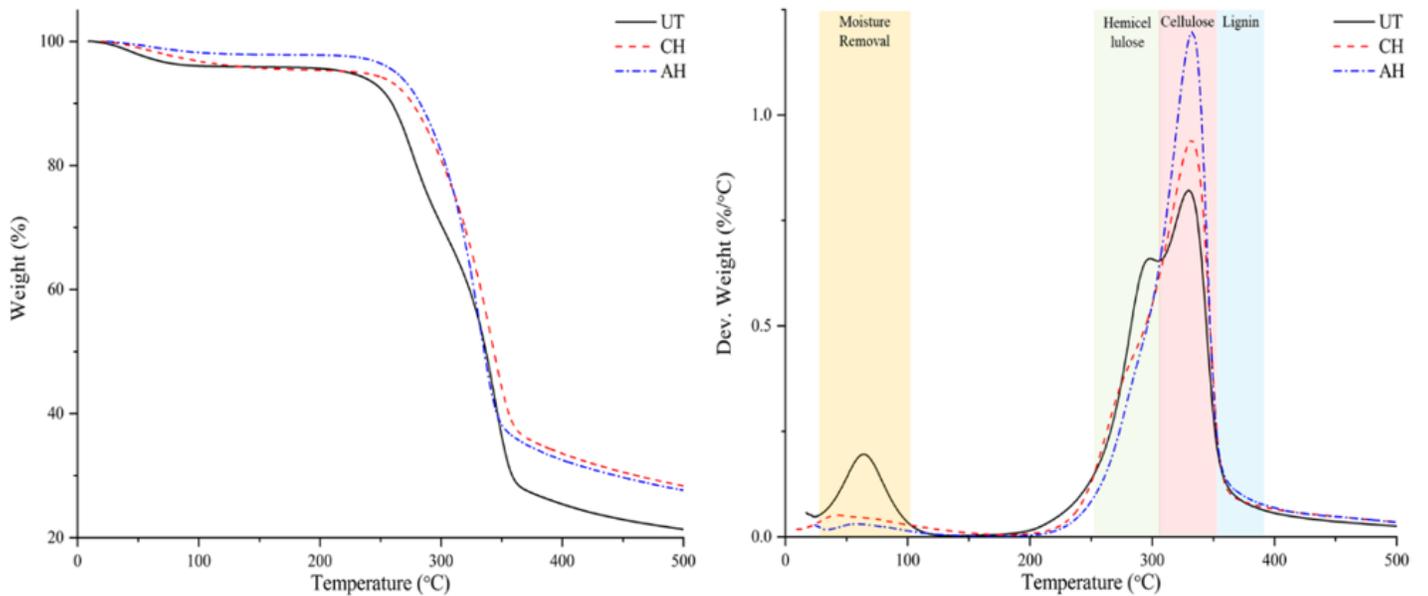


Figure 16

TGA and DTG of untreated and treated DPF.