

# We are IntechOpen, the world's leading publisher of Open Access books Built by scientists, for scientists

6,900

Open access books available

186,000

International authors and editors

200M

Downloads

Our authors are among the

154

Countries delivered to

TOP 1%

most cited scientists

12.2%

Contributors from top 500 universities



WEB OF SCIENCE™

Selection of our books indexed in the Book Citation Index  
in Web of Science™ Core Collection (BKCI)

Interested in publishing with us?  
Contact [book.department@intechopen.com](mailto:book.department@intechopen.com)

Numbers displayed above are based on latest data collected.  
For more information visit [www.intechopen.com](http://www.intechopen.com)



# Influence of Size Classifications on the Structural and Solid-State Characterization of Cellulose Materials

*Oluyamo Sunday Samuel and Adekoya Mathew Adefusika*

## Abstract

Influence of size classification on the properties of cellulose materials has been a subject of neglect over the years. Researchers had the opinion that there exist no significant difference between the characteristics of bulk particulate materials and sizes of their constituents. However, it has been affirmed that increase in crystallinity index, increases the strength properties of cellulose materials. Therefore, there is need to establish the influence of size classification as it affects the properties of cellulose materials. This study focused on the influence of size classifications on the structural and solid State characterization of cellulose obtained from wood dust. The structure of the cellulose composed principally of crystalline cellulose (I and II) and amorphous cellulose. The crystallinity and the inter-planar spacing revealed different structural properties for the two size classifications. The elemental composition consists of Carbon (C), Oxygen (O), Sodium (Na) and Chlorine (Cl) with Carbon having the highest percentage. The surface morphology of the isolated cellulose appears fiber-like for the size classifications examined. The isolated cellulose exhibits good mechanical and solid state properties with promising applications in device utilization. Within the limit of the research, size classification is noted to influence the characteristics of the cellulose materials.

**Keywords:** cellulose, size classification, crystallinity, structural characterization, solid state properties

## 1. Introduction

Wood is one of the hard fibrous structural tissue and abundant natural materials on earth. It is an organic material with a composition of cellulose, hemicellulose, and lignin which has been used for many years as a basic need in construction materials and other purposes [1–3]. The effect of particle sizes on the thermal and mechanical properties of wood had gained popularity in recent years due to its importance in improving the insulation properties of materials [4].

The environmental degeneration caused by solid waste from different activities had been a challenge to the waste management throughout the world. Nigeria, with a population over 180 million as at 2018, has the largest producer of residue and solid waste in Africa.

One important waste from wood is the wood dust. This by-product usually constitutes menace to man and his environment as the material is usually disposed of sometimes indiscriminately in different locations which most often constitute environmental pollution [3]. Studied had shown that if well harness, wood dust may attract economic values to the country rather than the usual pollution.

Cellulose is a formation of the composite, a versatile and widely natural-based material in nature that consists of glucose molecules which has various uses to man and used by man for thousands of years as building material, or energy source. It is a polymer that contains crystallites and displayed para-crystalline morphology [5]. The linear molecules are linked laterally by hydrogen bonds to form linear bundles which give rise to the crystalline structure. It has become one of the material's serving mankind for centuries and major subject in the history of polymer science in developing nation's economic and determination of polymeric crystal structures. Today, it is an important material which is widely used in industries (paper, pharm, food, etc.) and it has also served as an economic output in many countries of the world.

It has a general formula  $(C_6H_{10}O_5)_n$ , found in plants as microfibril and isolated from wide range of species from higher plants such as wood to green algae [6, 7]. It is a polymer that composed of amorphous and crystalline regions which varies depending on the plant species. Cellulose can be isolated from plants and non-plant sources. Isolation can be from a variety of sources such as (cotton, hemp, jute, sugarcane bagasse, rice straw, durra stalk, groundnut shell, etc.). The composition of chemical and cell dimensions depend on plants, origin and isolation method.

Cellulose can exist in its derivative forms namely rayon, cellulose acetate, cellulose nitrate, and ethyl cellulose. It is the main component of about half to one-third of plant tissues and categorized into three namely,  $\alpha$ -cellulose,  $\beta$ -cellulose and  $\gamma$ -cellulose [8]. Solubility and precipitation nature are the major category upon which cellulose is based. In plants, it composed of a linear homopolymer of 1,4- $\beta$ -glucopyranose units associated with hydrogen bonding and as a semicrystalline structure that is found and circulated from highly developed trees to primitive organisms [9, 10]. The chemical repeating unit is the  $\beta$ -1,4-linked glucose and structural repeat is  $\beta$ -cellobiose [11]. The repeating unit in cellulose is the anhydrocellulobiose and half a degree of polymerization (DP) gives rise to the number of repeating per molecule. It is higher in native cellulose than other group of cellulose which is usually due to the purification procedures. Van der Waals forces and hydrogen bonds tightly bound the glucose to each other to form crystalline structures called Elementary fibril. This consists of around 40 glucan chains, 40 Å widths, 30 Å tick and 100 Å long [12].

There are two main regions found in cellulose fibers. These are crystalline and amorphous. Crystalline are regions with a high order of microfibrils while less order of microfibrils is called amorphous. Amorphous material are materials that lack definite shape or formless. These regions vary proportions among the plants species. For this reason, the properties of cellulose materials depend largely on the material. The versatility of cellulose makes it important in it usage. The method of isolation or treatment, sources of cellulose give rise to different polymorphs with only a few exceptions. This may be due to molecular orientation and hydrogen bonding [3]. The polymorphic forms can be grouped into four: I, II, III, and IV which can be determined by XRD pattern. The first model of the crystalline structure of native cellulose of a monoclinic unit cell was developed by Mayer and Mish [13]. All native cellulose crystalline consists of CI with only a few exceptions and exhibits the same crystalline structure. It composed of two distinct allomorphs I $\alpha$ (triclinic) and I $\beta$ (monoclinic) depending on the biological origins. I $\alpha$ structure is metastable and dominated polymorph for most algae and bacteria, whereas I $\beta$ is dominant for

higher plant cell wall cellulose and in tunicates.  $\alpha$  can be converted to  $\beta$  in alkaline solution by hydrothermal treatments at a temperature of 260°C. Native cellulose is organized in fibrils, which are represented by the association of cellulose molecules. The native cellulose of higher plants possesses a high degree of polymerization (DP) of up to 10,000  $\beta$ - anhydroglucose residues [14]. This indicates that the molecular weight is above 1.5 million (g/mol). The increase in crystalline regions increases the rigidity and decreases the elasticity of the polymeric substance. The accessibility of cellulose molecules affects the ratio of the crystalline region and the amorphous region in the cellulose structure [15–18].

Modification of cellulose is identified by addition of crystalline allomorphs, II, III, and IV. Mercerization (Alkali treatment) and regeneration (solubilization and subsequent recrystallization) are the two main methods of preparing celluloses II.

However, when the time and the amount of chemical introduced in the treatment of native cellulose are not restricted to a predetermined pattern, it results to the production of cellulose (I and II).

Treatments with liquid ammonia with celluloses I and II produces celluloses III<sub>I</sub> and III<sub>II</sub>. In addition, heating of III<sub>I</sub> and III<sub>II</sub> produces celluloses IV<sub>I</sub> and IV<sub>II</sub> [19, 20].

Structure and morphology of cellulose give a clearer picture of understanding the behavior of cellulose during chemical modification. It also gives understanding on the morphological changing of materials after hydrolysis. There are three structural levels that describe the complex structure of cellulose. These are molecular level (molecular mass, potential intramolecular and chemical constitution), supra-molecular levels (crystal structure and intermolecular hydrogen-bonding system) and morphological levels (organization of crystals into microfibrils, the existence of different cell wall layers in the fibers, and other cellulose morphologies). These levels determine both chemical and mechanical properties of cellulose.

One of the parameters used to study the total cellulose present in cellulosic materials is the crystallinity Index [21–25]. In addition, the presence of crystallinity in cellulose contributes greatly to its physical, chemical and mechanical properties [22, 26, 27]. The crystallinity index of cellulosic material has an influence on the stiffness, rigidity and the strength of the material. The increase in the crystallinity index (CI) is associated with high potential mechanical property and increase reinforcing capability of a cellulose material. Several techniques have been used to measure the crystallinity index. These techniques include; XRD, solid-state <sup>13</sup>C NMR, infrared (IR) spectroscopy and Raman spectroscopy. The crystallinity index has been used for years in interpreting cellulose changes after treatment (physicochemical and biological). It has been reported that crystallinity index varies significantly depending on the measurement method. Among these methods, XRD is the mostly employed. Three different methods are commonly employed in calculating the CI from the raw spectrographic data on the XRD [23, 28–32]. The first method was established by [33], proposed for cellulose I. In this method, consideration was based on the ratio of the peak height between the intensity of the crystalline and the total intensity after subtraction of background signal at 18° (2 $\theta$ ) degrees. The idea behind the Segal equation is that there are no crystalline peaks near 18° for cellulose I, therefore any observed intensity would be due to amorphism region. [33] found a maximum at 18° but other authors have found maxima at values even higher, such as 20–22° [34]. Thus, [30] showed that a perfectly crystalline cellulose would still only give a Segal CrI value of 92% when the crystal is approximately the size of a good cotton crystal (FWHM = 1.7°). Furthermore, for a 100% amorphism, a pattern would have to be completely flat; something that never happens. Because there is no fundamentally sound method that is well proven for crystallinity determination, Segal method results remain fairly simple to obtain and give helpful information. Segal with other methods (peak de-convolution method, and amorphous subtraction) all has fundamental flaws.

Available researches into isolation of cellulose focused mostly on thermal and mechanical properties. Hitherto, there is no available information about the influence of size classifications on the structural and solid state characterization of cellulose materials. Although, some school of thought have the notion that size classification has no significant influence on the properties of cellulose materials.

This effect is however well established for wood bulk and particle materials [2, 35, 36]. It is important to note that cellulose particles are presented in differing sizes in material processes and applications.

*Guarea thompsonii* is a species of plant from the family of *Meliaceae*. It is a hard wood that is naturally durable, resistant to impregnation, medium shrinkage and has a desired compressive advantage for concrete as a structural material.

Therefore in the present research, cellulose particles isolated from *Guarea thompsonii* are classified into two categories while the structural and solid state characterizations are determined.

## 2. Method

### 2.1 Material

Wood specie (*Guarea thompsonii*) was selected from a sawmill in the area of research and authenticated at the Department of Forestry and Wood Technology, Federal University of Technology, Akure (FUTA), Nigeria. The sample was processed into wood dust and sieved into two size classifications (424–599  $\mu\text{m}$  and 600–849  $\mu\text{m}$ ) at the Department of Materials and Metallurgical Engineering (FUTA). Analytical grades chemical used were Sodium chlorite ( $\text{NaClO}_2$ ) (Sigma-Aldrich, Steinheim, Germany), sodium hydroxide ( $\text{NaOH}$ ) (British Drug House, Darmstadt, Germany), and acetic acid (Sigma-Aldrich, Steinheim, Germany).

### 2.2 Pre-treatment of material

The obtained sample after processed to wood dust were sieved using a Wiley mechanical sieve shaker (Pascal Engineering, Sussex, England) and the wood dust with two size classifications (425–599  $\mu\text{m}$  and 600–849  $\mu\text{m}$ ) were obtained.

### 2.3 Pulping procedure

The two classifications of the wood dust were pulped in a water bath at 90°C under atmospheric pressure with the ratio of wood to liquor of 1:20, using 20%  $\text{NaOH}$  for 90 minutes. The pulped was obtained by filtration after digestion and washed thoroughly with water until it was free of residual alkali. The pulp yield was oven-dried at 105°C to a constant weight and stored for further processing.

### 2.4 Bleaching procedure

1000 mL of hot distilled water, 12 g of  $\text{NaClO}_2$ , and 3 mL of acetic acid were added to approximately 20 g of oven-dried pulp sample in a 2-L Erlenmeyer flask. The flask was covered and the mixture heated in a water bath at 70°C for 30 minutes with intermittent stirring. After the first 30 minutes in the water bath, another 12 g of  $\text{NaClO}_2$  and 3 mL of acetic acid was added with intermittent stirring and sustained for another 30 minutes before switching the bath off. The sample was allowed to settle down for 24 hours in the water bath. After digestion, the bleach was obtained by filtration and washed thoroughly with water until it was free of residual alkali and chlorine. The obtained sample was dried at 105°C to a constant weight.

### 3. Theoretical consideration

#### 3.1 X-ray diffraction method

One of the reliable techniques to determine the crystal structure of any material is the X-ray diffraction (XRD). The crystalline phase of the material can be obtained by examine the diffraction patterns.

The XRD patterns of the two size classifications were obtained using a Philips PW 3710 X'pert Pro diffractometer (Philips Analytical, Almelo, Netherlands) with a Cu-K $\alpha$  monochromator of wavelength,  $\lambda = 1.540598 \text{ \AA}$ m, in the range of 10–50° (2 $\theta$ ) generated at 15 kV. All experiments were repeated twice and duplicate X-ray analyses were performed.

The Interplanar spacing ( $d$ -spacing) was calculated from the Bragg equation using [37, 38].

$$n\lambda = 2d \sin \theta \quad (1)$$

where  $n$  is the order of reflection,  $\lambda$  is the wavelength of the incident X-rays (m),  $d$  is the interplanar spacing of the crystal, and  $\theta$  is the Bragg's angle (°).

The crystallinity index ( $CrI$ , %) was obtained from the XRD diffraction pattern. The patterns were engaged to determine the crystallinity parameters of cellulose derived from different size classifications of the wood dust samples. The crystallinity index was calculated according to [33], as followed Eq. (2),

$$CrI = \frac{I_{020} - I_{am}}{I_{020}} \times 100 \quad (2)$$

where  $I_{020}$  is the maximum intensity of the lattice diffraction and  $I_{am}$  is the low intensity peak at the amorphous region of the baseline at 2 $\theta$ , approximately 18°.

The average crystallite sizes ( $L$ ) in the isolated cellulose samples were calculated from the XRD line broadening using the Scherrer's equation

$$L = \frac{K \times \lambda}{H \times \cos \theta} \quad (3)$$

where  $K$  is a constant whose value is given as 0.91,  $\theta$  is the Bragg's angle (°), and  $H$  is the intensity of the full width at half maximum (FWHM) corresponding to a high intensity peak of the diffraction plane.

The Surface chain  $W$  occupying a layer that is approximately 0.57 nm thick is given as

$$W = \frac{(L - 2h)^2}{L^2} \quad (4)$$

The determination of the monoclinic and triclinic structure for the two size classifications was calculated from the method developed by [37]. The isolated cellulose was categorized into I $\alpha$  or I $\beta$  predominant form by employing discriminant analysis. The function which discriminates between them (the monoclinic and triclinic structure) is given as:

$$Z = 1693d_1 - 902d_2 - 549 \quad (5)$$

where  $d_1(nm)$  is the  $d$ -spacing of the I $\beta$  ( $\bar{1}\bar{1}0$ ) peak and  $d_2(nm)$  is the  $d$ -spacing of the I $\beta$  (110) peak.  $Z > 0$  indicates that cellulose is rich in the I $\alpha$  form and  $Z < 0$  indicates that I $\beta$  is the predominant form.

### 3.2 FTIR spectroscopy measurement

Fourier transform infrared (FTIR) spectroscopy is a mature analytical technique employed to examine the microscopic area of a materials. FTIR spectra of powder samples of cellulose were obtained using a Thermo Nicolet 5700 FTIR spectrometer (Nicolet, Madison, WI, USA). The Spectra were acquired over the range 500–4000  $\text{cm}^{-1}$  at a resolution of 2  $\text{cm}^{-1}$  for samples in pellet form prepared by mixing 1.0 mg of powder samples with 200.0 mg KBr spectroscopic grade. The spectra obtained were used for rapid information about the chemical structure of the cellulose samples.

### 3.3 Scanning Electron Microscope Measurement

The morphological characterization of composite materials can be investigated by the Scanning Electron Microscopy (SEM). It is a popular and powerful technique for imaging the surface of a material (surface topology, morphology and chemical composition) [39]. The image resolution depends on the property of the electron and the electron probe interaction with the specimen. SEM analysis was performed on the isolated cellulose obtained of the two size classifications using FEI NOVA 200 NanoSEM equipment (FEI Company, Hillsboro Oregon, USA) with an accelerating voltage of up to 30 kV and a resolution of up to 1 nm to observe the morphology of the cellulose obtained.

## 4. Results

### 4.1 X-ray diffraction (XRD) of cellulose

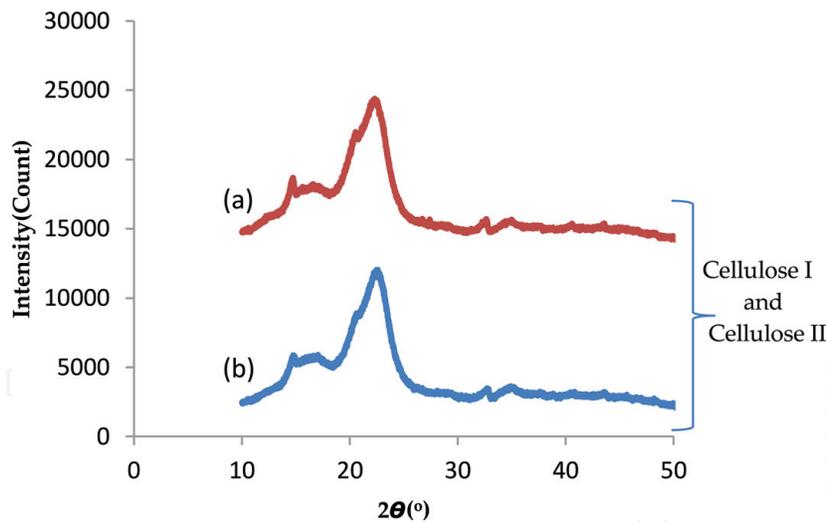
X-ray diffraction (XRD) is a method generally used to determine the crystallinity of materials, interplanar distances etc. The free hydroxyl groups present in the cellulose macromolecules are likely to be involved in a number of intramolecular and intermolecular hydrogen bonds, which may give rise to various ordered crystalline arrangements [40, 41].

In order to evaluate and determine the intensities of the diffraction bands, establish the crystalline and amorphous areas and determine the crystallite sizes of the cellulose, the X-ray diffractogram (**Figure 1**) was adopted. The crystallographic planes from the diffractogram are labeled according to the cellulose structure.

The diffractograms showed that 12.414–14.755°C ( $2\theta$ ), 16.910–17.127°C ( $2\theta$ ), 18.01°C ( $2\theta$ ), 22.107–22.591°C ( $2\theta$ ) and 34.908–35.075°C ( $2\theta$ ) reflections were assigned to reflection assigned to the ( $\bar{1}10$ ) crystallographic plane, (110) crystallographic plane, amorphous phase, (020) crystallographic plane and (004) crystallographic [37].

The X-ray diffraction pattern generated to evaluate the crystallinity of the cellulose samples in **Figure 1** showed a peak at  $2\theta = 22.591$  and  $22.308^\circ\text{C}$  for *Guarea thompsonii* of 425–599  $\mu\text{m}$  and 600–849  $\mu\text{m}$  of size classifications. These distinct peaks obtained for the XRD of the samples is an indication that the cellulose are crystalline in nature.

The diffraction peak of cellulose shown around  $2\theta = 12.414$ – $14.755^\circ\text{C}$  was assigned to the crystalline plane of ( $\bar{1}10$ ) for cellulose type I and II. Moreover, it is interesting that cellulose showed the doublet in the intensity of the main peaks ( $2\theta = 20.2$ – $20.4^\circ\text{C}$ ) which were also assigned to the crystalline planes of (110) for cellulose type I and II. This shows that mixture of cellulose I and II has been the most stable structure of technical relevance and can be produced by two processes:



**Figure 1.** XRD of isolated cellulose obtained for different size classifications for *Guarea thompsonii* (a) 425–599  $\mu\text{m}$  (b) 600–849  $\mu\text{m}$ .

regeneration (solubilization and recrystallization) and mercerization (aqueous sodium hydroxide treatments) [27].

From this result, it can be concluded that alkali treatment in pretreatment process led to the change of cellulose allomorph from type I; the native cellulose found in nature, to type I and II; the regenerated cellulose which is the most stable crystalline form. Moreover, the acid hydrolysis which removed the amorphous region out from the cellulose led to re-crystallization was the main cause to obtain the obvious peaks of doublet intensity at  $2\theta = 20.2\text{--}20.4^\circ\text{C}$ .

#### 4.1.1 Investigation of the crystallites structure of cellulose by XRD

The average dimensions of the elementary crystallites perpendicular to the  $(1\bar{1}0)$ ,  $(110)$  and  $(020)$  crystallographic planes of the mixture of cellulose I and cellulose II with amorphous can be calculated using the Scherrer's equation, by measuring the full widths at half maximum (FWHM) of the different diffraction peaks, assuming that the finite size of crystallites dominate the broadening of the X-ray reflections. These values have to be considered as a lower bound since instrumental broadening and possible imperfections of the crystal lattice are neglected by the method.

The results obtained from the XRD profiles of samples in **Figure 1** are presented in **Tables 1** and **2**. In this, only the length perpendicular to the  $(020)$  plane could be calculated since the  $(1\bar{1}0)$  and  $(110)$  reflections in the XRD profile overlapped each other. An average crystallite length of about 1.742 nm and 1.748 nm were obtained *Guarea thompsonii* of 425  $\mu\text{m}$  – 599  $\mu\text{m}$  and 600  $\mu\text{m}$  – 800  $\mu\text{m}$  size classifications.

The inter-planar spacing (d-spacing) values of the cellulose spectra for most prominent peak with crystal plane of preferred orientation along the  $(020)$  were 3.933 Å and 3.982 Å for *Guarea thompsonii* of 425–599  $\mu\text{m}$  and 600–849  $\mu\text{m}$

Classification ( $\mu\text{m}$ )	$(1\bar{1}0)$		$(110)$		$(004)$		$(020)$	
	$2\theta$	d (Å)	$2\theta$	d (Å)	$2\theta$	d (Å)	$2\theta$	d (Å)
425–599	14.754	5.999	17.044	5.198	34.908	2.568	22.591	3.933
600–849	14.721	6.013	16.726	5.293	34.992	2.562	22.308	3.982

**Table 1.** Band position ( $2\theta$ ) and d-spacing of crystalline cellulose of *Guarea thompsonii*.

Classification ( $\mu\text{m}$ )	$L(020)$ (nm)	Cr.I	W	Z-Values
425–599	1.750	56.89	0.12	–2.229
600–849	1.749	54.09	0.12	–8.428

**Table 2.**

Parameters obtained from the XRD analysis of the cellulose samples of *Guarea thompsonii*.

respectively. Other values of inter planar spacing for the remaining crystallographic planes were depicted in **Table 1**.

The crystalline interior chains W for the isolated samples were calculated by the fraction of cellulose chains contained in the interior of the crystallites. It was estimated as 0.12 for the two samples isolated in **Table 2**. This indicated that the proportion of crystallite interior chains, W, is similar for both samples examined.

**Table 2** showed the calculated Z- values for the two size classifications (*Guarea thompsonii*). The values of the estimated isolated cellulose obtained were less than zero ( $Z < 0$ ). This shows that the cellulose samples belong to I $\beta$  (monoclinic) dominant.

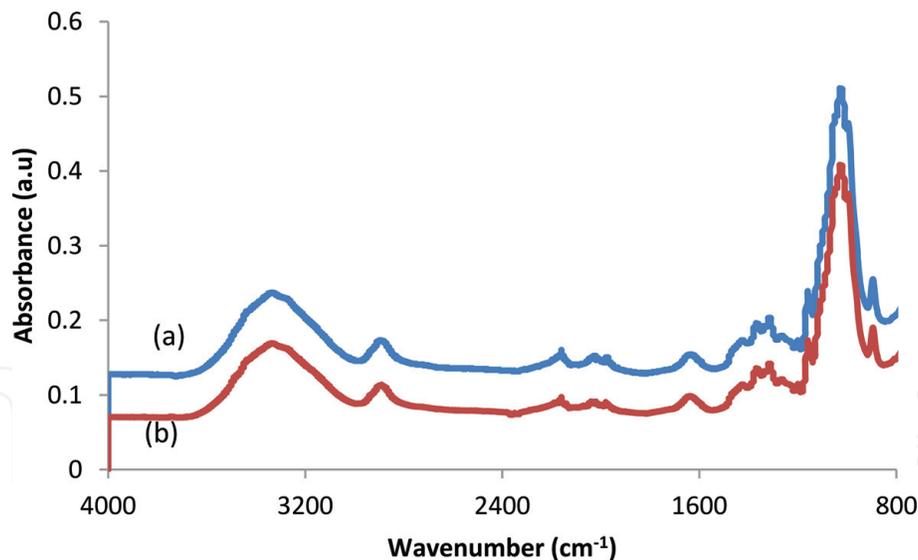
The degree of cellulose crystallinity is one of the most important crystalline structure parameters. The crystallinity index (CrI) calculated according to [33] showed that the crystallinity of cellulose obtained for 425–599  $\mu\text{m}$  size classification was higher than that obtained for 600  $\mu\text{m}$  to 849  $\mu\text{m}$  size classification. The CrI value are 56.89 and 54.09% for *Guarea thompsonii* cellulose obtained from different size classifications of 425–599  $\mu\text{m}$  and 600–849  $\mu\text{m}$ , respectively. In contrast, 60.4% for *Eucalyptus grandis* and 62.6% for *Pinus taeda* were also recorded in a study of structural characteristics and thermal properties of native cellulose [41]. This high percentage in crystallinity index might be associated with the reduction in the corresponding amorphous state of the material due to the probable dissociation of the bonds as a result of pulping. This can also be due to significant increase in surface area-to-volume ratio of the molecules, and with the crystalline size of the samples reducing to nanoscale. The difference in the values obtained may be due to the chemical treatments for purification and crystalline or amorphous standard [42]. Moreover, high crystallinity index is associated to increase stiffness, rigidity and strength of the isolated cellulose obtained. As a result, sample with 425–599  $\mu\text{m}$  size classifications has high potential mechanical property and reinforcing capability than sample with 600–849  $\mu\text{m}$  size classifications [43].

#### 4.2 Fourier infra-red spectroscopy (FTIR) of cellulose

**Figure 2** shows the functional groups and the band positions for the isolated cellulose prepared by Fourier Infra-red Spectroscopy (FTIR).

Two main absorbance regions were pronounced by the samples considered. The absorbance regions were in the range of approximately 1110–870  $\text{cm}^{-1}$  and 3630–2960  $\text{cm}^{-1}$  wave number. The intensities regions were high in 425–599  $\mu\text{m}$  size classifications than 600–849  $\mu\text{m}$  due to the surface to volume ratio of the atom exposed to the FTIR machine.

The strong dominant broad peaks in the region are obtained from 3630 to 2960  $\text{cm}^{-1}$  which is commonly observed for hydrogen. The absorption around 3320  $\text{cm}^{-1}$  corresponds to the vibration of H-bonded OH groups while a peak near 2890  $\text{cm}^{-1}$  is assigned to C-H stretching vibration. These inter- and intra-molecular hydrogen bonds in the cellulose has a strong influence on the physical and mechanical properties of the cellulose. The band near 1160  $\text{cm}^{-1}$  is representative of the anti-symmetric bridge stretching of C—O—C groups. The band near 1300  $\text{cm}^{-1}$



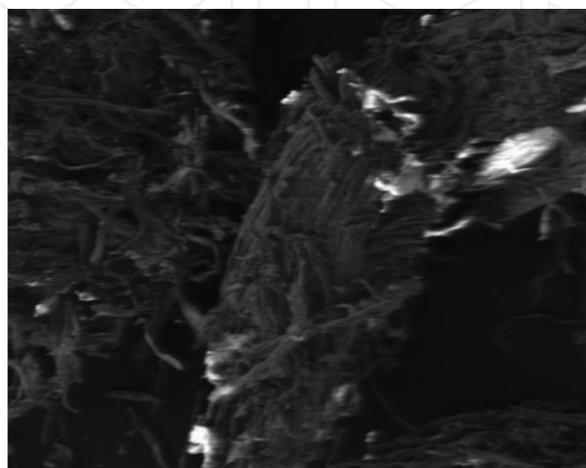
**Figure 2.**  
FTIR spectra of isolated cellulose of different size classifications for *Guarea thompsonii* (a) 425–599  $\mu\text{m}$  (b) 600–849  $\mu\text{m}$ .

could be ascribed to  $\text{CH}_2$  wagging vibrations in the cellulose while the absorption at  $1200\text{ cm}^{-1}$  belongs to the  $\text{C—O—H}$  in-plane bending at C-6 [44, 45]. The band peak at  $1610\text{ cm}^{-1}$  can be assigned as OH bending due to absorbed water because the OH bending mode is strongly perturbed by bound water [46]. Strong peaks at  $1020$  and  $1090\text{ cm}^{-1}$  are indicative of  $\text{C—O}$  stretching at C-3,  $\text{C—C}$  stretching and  $\text{C—O}$  stretching at C-6 [45]. Finally, the absorbance peak observed at  $899\text{ cm}^{-1}$  was assigned to the symmetric  $\text{C—O—C}$  stretching of  $\beta$  (1 $\rightarrow$ 4)-glycosidic linkage.

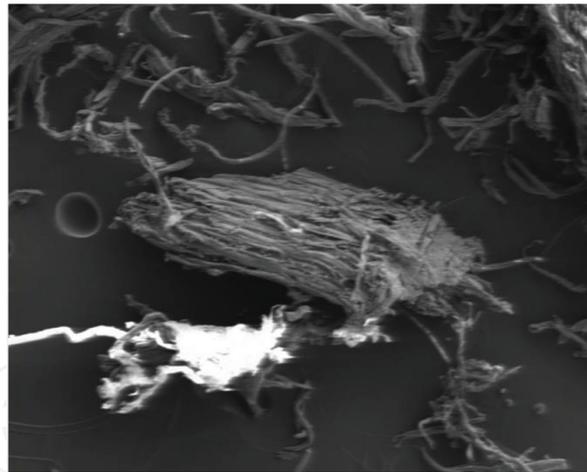
### 4.3 Scanning Electron Microscopy (SEM)

The Scanning Electron Microscopy (SEM) was an effective method for investigating the morphological characteristics of the composites. **Figures 3** and **4** show the SEM image of *Guarea thompsonii* of isolated cellulose obtained.

As shown in **Figures 3** and **4**, SEM images of the isolated cellulose of two samples depicted strings of fibers. This was in agreement with other authors' findings, although the length of the fiber may differ [47–52].



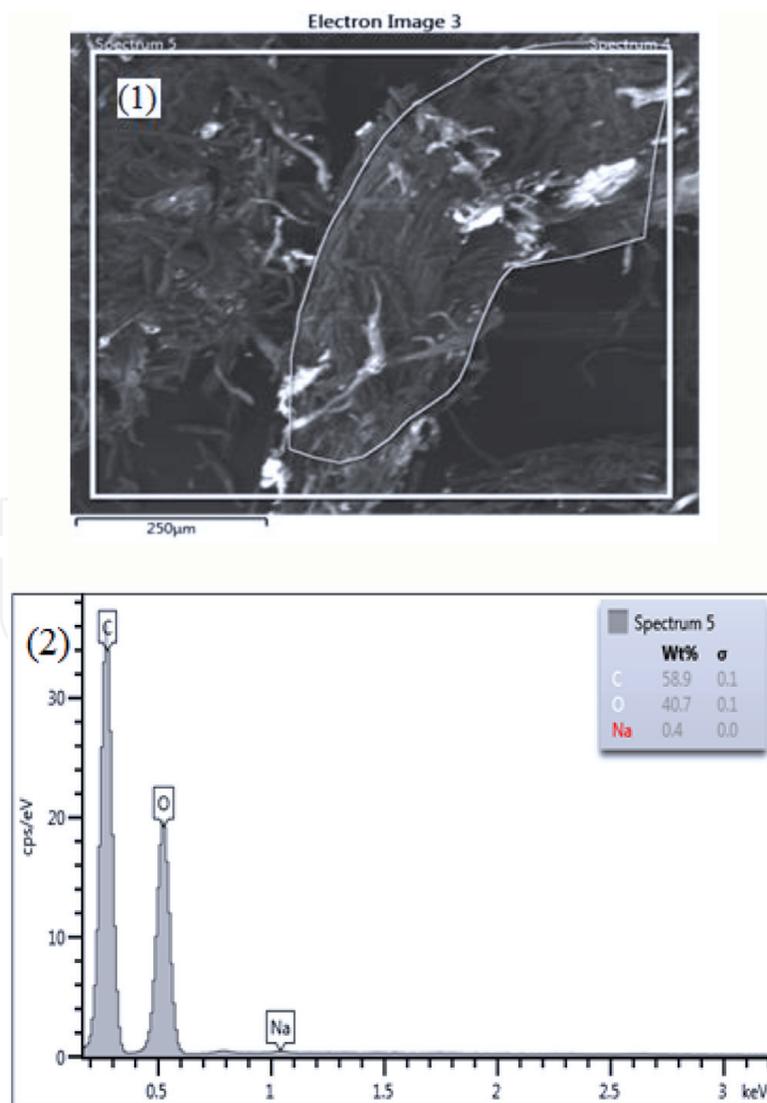
**Figure 3.**  
SEM image of isolated cellulose for *Guarea thompsonii* of 425–599  $\mu\text{m}$  size classifications.



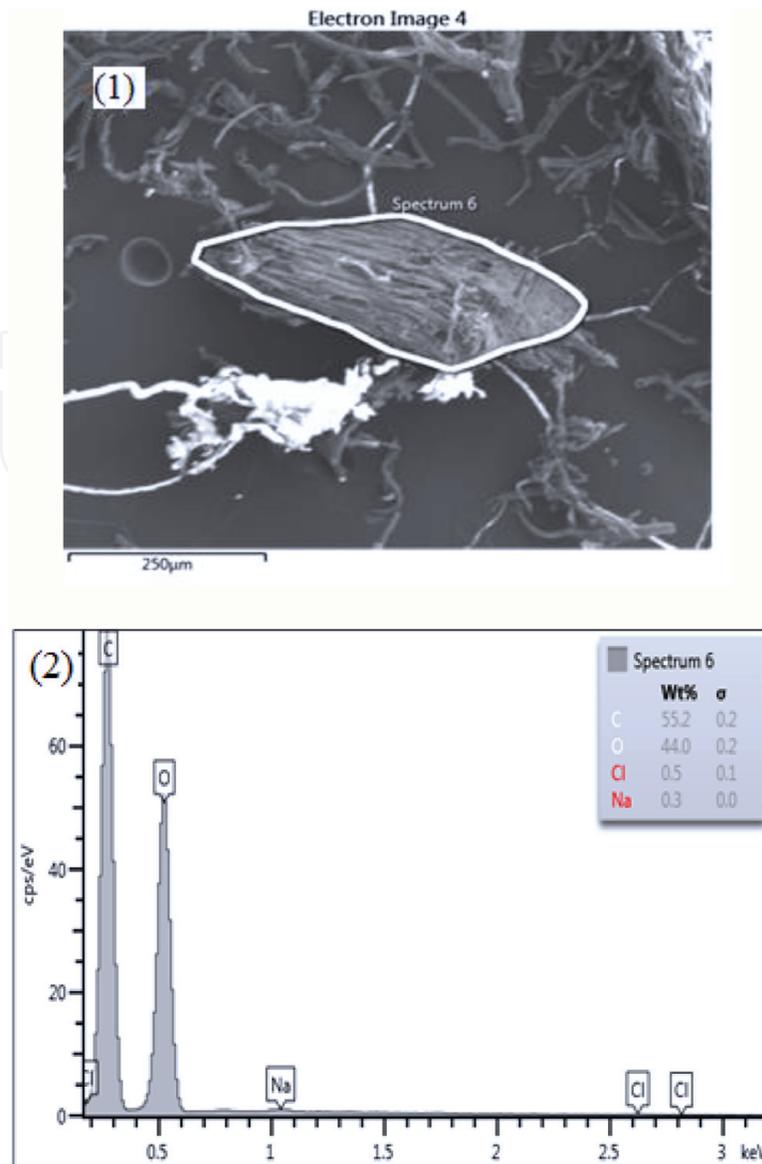
**Figure 4.**  
SEM image of isolated cellulose for *Guarea thompsonii* of 600–899  $\mu\text{m}$  size classifications.

#### 4.4 Energy dispersive X-ray diffraction (EDX)

Energy dispersive X-ray diffraction (EDX) attached with SEM was used for elemental analysis of isolated cellulose. The EDX spectra (as shown in **Figures 5 and 6**) peaks correspond to the energy levels for which the carbon (C), oxygen (O),



**Figure 5.**  
(1) SEM image of isolated cellulose for *Guarea thompsonii* of 425–599  $\mu\text{m}$  size classifications; and (2) EDX spectrum of isolated cellulose for *Guarea thompsonii* of 425–599  $\mu\text{m}$  size classifications.



**Figure 6.**  
 (1) SEM image of isolated cellulose for *Guarea thompsonii* of 600–899 μm size classifications; and (2) EDX spectrum of isolated cellulose for *Guarea thompsonii* of 600–899 μm size classifications.

chlorine (Cl), and Sodium (Na) were identified with carbon having the highest percentage among the elements observed in the spectra. The elemental compositions for the samples with size classifications 425–599 μm were 58.9 wt % carbon, 40.7 wt % oxygen and 0.4 wt % sodium and 55.2 wt % carbon, 44.0 wt % oxygen, 0.5 wt % chlorine and 0.3 wt % sodium for 600–899 μm. The impurities present could have been due to the NaClO<sub>2</sub> that was used in the bleaching process.

## 5. Conclusion

Wood dusts from two size classifications were isolated from XRD, FTIR, SEM and EDX. The isolated cellulose obtained is the mixture of cellulose I and II and amorphous with fiber-like shape. The two celluloses examined has a preferred orientations along the (020) plane for the most prominent peaks with a crystallinity index of 56.89% for size range 425–599 μm and 54.09% for size range 600–849 μm. The crystallinity index is high in 425–599 μm size classifications compare to 600–849 μm. This indicates that the strength properties is higher in 425–599 μm size classification and have reinforcement ability than 600–849 μm. It is important to

note that size classifications played a major role on the crystallinity index on the sample examined which is a basic factor that determine how high the mechanical properties is in a material.

## Acknowledgements

We express our gratitude to the Material and Engineering Research Institute (MERI), Sheffield Hallam University (UK) for their support and excellent work done during the analyses of the powder material; The Federal University of Technology, Akure, Nigeria and Edo University, Iyamho, Nigeria are also appreciated for their support during the preparation of the samples.

## Conflict of interest

No conflict of interest.

## Author details

Oluyamo Sunday Samuel<sup>1</sup> and Adekoya Mathew Adefusika<sup>1,2\*</sup>

1 Condensed Matter and Statistical Physics Research Unit, Department of Physics, Federal University of Technology, Akure, Nigeria

2 Department of Physics, Edo University Iyamho, Edo State, Nigeria

\*Address all correspondence to: mathewadekoya14@gmail.com

## IntechOpen

© 2019 The Author(s). Licensee IntechOpen. This chapter is distributed under the terms of the Creative Commons Attribution License (<http://creativecommons.org/licenses/by/3.0>), which permits unrestricted use, distribution, and reproduction in any medium, provided the original work is properly cited. 

## References

- [1] Belgacem MN, Gandini A. The surface modification of cellulose fibres for use as reinforcing elements in composite materials. *Composite Interfaces*. 2005;12(1–2):41-75. DOI: 10.1163/1568554053542188
- [2] Oluyamo SS, Adekoya MA. Effect of dynamic compression on the thermal conductivities of selected wood products of different particle sizes. *International Research Journal of Pure and Applied Physics*. 2015;3(1):22-29
- [3] Adekoya MA, Oluyamo SS, Oluwasina OO, Popoola AI. Structural characterization and solid state properties of thermal insulating cellulose materials of different size classifications. *BioResource*. 2018;13(1): 906-917. DOI: 10.15376/biores13.1.906-917
- [4] Oluyamo SS, Bello OR. Particle sizes and thermal insulation properties of some selected wood materials for solar device applications. *IOSR-JAP*. 2014; 6(2):54-58. DOI: 10.9790/4861-0903011417
- [5] Hosemann R. Crystallinity in high polymers, especially fibres. *Polymer*. 1962;3:349-392. DOI: 10.1016/0032-3861(62) 90093-9
- [6] Varshney VK, Naithani S. Chemical functionalization of cellulose derived from nonconventional sources. In: Kalia S et al., editors. *Cellulose Fibers: Bio- and Nano-Polymer Composites*. Berlin Heidelberg: Springer-Verlag; 2011. pp. 43-60
- [7] Trache D, Donnot A, Khimeche K, Benelmir R, Brosse N. Physico-chemical properties and thermal stability of microcrystalline cellulose isolated from alfa fibres. *Carbohydrate Polymers*. 2014;104:223-230
- [8] Sun JX, Sun XF, Zhao H, Sun RC. Isolation and characterization of cellulose from sugarcane bagasse. *Polymer Degradation and Stability*. 2004;84:331-339. DOI: 10.1016/j.polyimdegradstab.2004.02.008
- [9] Klemm D, Philipp B, Heinze T, Heinze U, Wagenknecht W. *Comprehensive Cellulose Chemistry, Volume 1, Fundamentals and Analytical Methods*. Weinheim, Germany: Wiley-VCH; 1998
- [10] Sèbe G, Ham-Pichavant F, Ibarboure E, Koffi ALC, Tingaut P. Supramolecular structure characterization of cellulose II nanowhiskers produced by acid hydrolysis of cellulose I substrates. *Biomacromolecules*. 2012;13(2):570-578. DOI: 10.1021/bm201777j
- [11] Varrot A, Macdonald J, Stick RV, Pell P, Gilbert HJ, Davies GJ. Distortion of a cellobio-derived isofagomine highlights the potential conformational itinerary of inverting  $\beta$ -glucosidases. *Chemical Communications*. 2003: 946-947
- [12] Bidlack J, Malone M, Benson R. Molecular structure and component integration of secondary cell walls in plants. *Proceedings of the Oklahoma Academy of Science*. 1992;72:51-56
- [13] Sehaqui H, Zhou Q, Ikkala O, Berglund LA. Strong and tough cellulose nanopaper with high specific surface area and porosity. *Biomacromolecules*. 2012;12(10):3638-3644. DOI: 10.1021/bm2008907
- [14] Hon DNS, Shiraishi N. *Wood and Cellulosic Chemistry*. New York: M. Dekker; 1991
- [15] Ishikawa A, Sugiyama J, Okano T. *Fine Structure and Tensile Properties of*

Ramie Fibers in the Crystalline Form of Cellulose I, II and III. Vol. 81. Wood Research; 1994. pp. 16-18

[16] Oh SY, Dong IY, Shin Y, Hwan CK, Hak YK, Yong SC, et al. Crystalline structure analysis of cellulose treated with sodium hydroxide and carbon dioxide by means of X-ray diffraction and FTIR spectroscopy. Carbohydrate Research. 2005;**340**:2376-2391. DOI: 10.1016/j.carres.2005.007

[17] Matheus P, Vinícios P, Ademir JZ. Structural Characteristics and Thermal Properties of Native Cellulose. Rijeka, Croatia: Intech Open Science; 2013. pp. 45-68

[18] Wicklein B, Salazar-Alvarez G. Functional hybrids based on biogenic nanofibrils and inorganic nanomaterials. Journal of Materials Chemistry A. 2013;**1**: 5469-5478. DOI: 10.1039/C3TA01690K

[19] Pérez S, Mazeau K. Conformations, structures, and morphologies of celluloses. In: Dimitriu S, editor. Polysaccharides: Structural Diversity and Functional Versatility. New York: Marcel Dekker, Inc.; 2005. pp. 41-68

[20] Zugenmaier P. Crystalline Cellulose and Cellulose Derivatives: Characterization and Structures. Springer Series in Wood Science. Berlin, Heidelberg: Springer-Verlag; Cellulose; 2008. pp. 101-174

[21] Al-Zuhair S. The effect of crystallinity of cellulose on the rate of reducing sugars production by heterogeneous enzymatic hydrolysis. Bioresource Technology. 2008;**99**: 4078-4085

[22] Andersson S, Serimaa R, Paakkari T, Saranpää P, Pesonen E. Crystallinity of wood and the size of cellulose crystallites in Norway spruce (*Picea abies*) WJ. Wood Science. 2003;**49**:531-537. DOI: 10.1007/s10086-003-0518-x

[23] Cao Y, Tan HM. Study on crystal structures of enzyme-hydrolyzed cellulosic materials by X-ray diffraction. Enzyme and Microbial Technology. 2005;**36**:314-317

[24] Lavoine N, Desloges I, Dufresne A, Bras J. Microfibrillated cellulose - its barrier properties and applications in cellulosic materials: A review. Carbohydrate Polymers. 2012;**90**(2): 735-764. DOI: 10.1016/j.carbpol.2012.05.026

[25] Hall M, Bansal P, Lee JH, Realff MJ, Bommarius AS. Cellulose crystallinity-a key predictor of the enzymatic hydrolysis rate. The FEBS Journal. 2010; **277**:1571-1582

[26] Ryu D, Lee SB, Tassinari T. Effect of crystallinity of cellulose on enzymatic-hydrolysis kinetics. Abstracts of Papers of the American Chemical Society. 1981; **182**:58-60

[27] Moon RJ, Martini A, Nairn J, Simonsen J, Youngblood J. Cellulose nanomaterials review: Structure, properties and nanocomposites. Chemical Society Revision. 2011;**40**: 3941-3994

[28] Bansal P, Hall M, Realff MJ, Lee JH, Bommarius AS. Multivariate statistical analysis of X-ray data from cellulose: A new method to determine degree of crystallinity and predict hydrolysis rates. Bioresource Technology. 2010; **101**:4461-4471

[29] Driemeier C, Calligaris GA. Theoretical and experimental developments for accurate determination of crystallinity of cellulose I materials. Journal of Applied Crystallography. 2011;**44**:184-192

[30] French AD, Cintron MS. Cellulose polymorphy, crystallite size, and the Segal crystallinity index. Cellulose. 2013;**20**:583-588. DOI: 10.1007/s10570-012-9833-y

- [31] Park S, Baker JO, Himmel ME, Parilla PA, Johnson DK. Cellulose crystallinity index: Measurement techniques and their impact on interpreting cellulase performance. *Biotechnology for Biofuels*. -2010;**3**(1): 10. DOI: 10.1186/1754-6834-3-10
- [32] Thygesen A, Oddershede J, Lilholt H, Thomsen AB, Stahl K. On the determination of crystallinity and cellulose content in plant fibres. *Cellulose*. -2005;**12**(6):563-576. DOI: 10.1007/s10570-005-9001-8
- [33] Segal L, Creely JJ, Martin AE, Conrad CM. An empirical method for estimating the degree of crystallinity of native cellulose using the X-ray diffractometer. *Textile Research Journal*. 1959;**29**(10):786-794. DOI: 10.1177/004051755902901003
- [34] Agarwal UP, Reiner RS, Ralph SA. Cellulose I crystallinity determination using FT-Raman spectroscopy: Univariate and multivariate methods. *Cellulose*. 2010;**17**(4):721-733. DOI: 10.1007/s10570-010-9420-z
- [35] Jaya H, Omar MF, Akil HA, Ahmad ZA, Zulkepli NN. Effect of particle size on mechanical properties of sawdust-high density polyethylene composites under various strain rate. *BioResources*. 2016;**11**(3):6489-6504. DOI: 10.15376/biores.11.3.6489-6504
- [36] Oluyamo SS, Aramide TM, Adekoya MA, Famutimi OF. Variation of bulk and particle thermal properties of some selected wood materials for solar device applications. *IOSR Journal of Applied Physics (IOSR-JAP)*. 2017;**9**(3):14-17. DOI: 10.9790/4861-0903011417
- [37] Wada M, Okano T. Localization of  $\alpha$  and  $\beta$  phases in algal cellulose revealed by acid treatments. *Cellulose*. 2001;**8**:183-188. DOI: 10.1023/A:1013196220602
- [38] Kim UJ, Eom SH, Wada M. Thermal decomposition of native cellulose: Influence on crystallite size. *Polymer Degradation and Stability*. 2010;**95**: 778-781. DOI: 10.1016/j.polymdegradstab.2010.02.009
- [39] Azzaoui K, Mejdoubi E, Lamhamdi A, Jodeh S, Hamed O, Berrabah M, et al. Preparation and characterization of biodegradable nanocomposites derived from carboxymethyl cellulose and hydroxyapatite. *Carbohydrate Polymers*. 2017;**167**:59-69. DOI: 10.1016/j.carbpol.2017.02.092
- [40] Popescu MC, Popescu CM, Lisa G, Sakata Y. Evaluation of morphological and chemical aspects of different wood species by spectroscopy and thermal methods. *Journal of Molecular Structure*. 2011;**988**:65-72. DOI: 10.1016/j.molstruc.2010.12.004
- [41] Poletto M, Pistor V, Zattera AJ. Structural characteristics and thermal properties of native cellulose. In: Van de Ven T, Gdbout L, editors. *Cellulose-Fundamental Aspects*. Rijeka, Croatia: Intech Open Science; 2013. pp. 45-68.4
- [42] Gümüşkaya E, Usta M, Kirci H. The effects of various pulping conditions on crystalline structure of cellulose in cotton linters. *Polymer Degradation and Stability*. 2003;**81**(3):559-564. DOI: 10.1016/S0141-3910(03)00157-5
- [43] Johar N, Ahmad I, Dufresne A. Extraction, preparation and characterization of celluloses fibres and nanocrystals from rice husk. *Industrial Crops and Products*. 2012;**37**:93-99. DOI: 10.1016/j.indcrop.2011.12.016
- [44] Cao Y, Tan HM. Structural characterization of cellulose with enzymatic treatment. *Journal of Molecular Structure*. 2004;**705**:189-193. DOI: 10.1016/j.molstruc.2004.07.010
- [45] Liu CF, Xu F, Sun JX, Ren JL, Curling S, Sun RC, et al. Physicochemical characterization of

- cellulose from perennial ryegrass leaves (*Lolium perenne*). Carbohydrate Research. 2006;**341**:2677-2687. DOI: 10.1016/j.carres.2006.07.008
- [46] Oh SY, Yoo D, Shin Y, Seo G. FTIR analysis of cellulose treated with sodium hydroxide and carbon dioxide. Carbohydrate Research. 2005;**340**: 417-428. DOI: 10.1016/j.carres.2004.11.027
- [47] El-Sakhawy M, Hassan ML. Physical and mechanical properties of microcrystalline cellulose prepared from agricultural residues. Carbohydrate Polymer. 2007;**67**(1):1-10. DOI: 10.1016/j.carbpol.2006.04.009
- [48] Adel AM, Abou-Youssef H, El-Gendy AA, Nada AM. Carboxymethylated cellulose hydrogel; sorption behavior and characterization. Nature and Science. 2010;**8**(8):244-256. DOI: 10.7537/marsnsj080810.29
- [49] Ibrahim MM, Agblevor AF, El-Zawawy W. K: Isolation and characterization of cellulose and lignin from steam-exploded lignocellulosic biomass. BioResources. 2010;**5**(1): 397-418. DOI: 10.15376/biores.5.1.397-418
- [50] Pereira PHP, Voorwald HCJ, Cioffi MOH, Mulinari DR, Da Luz SM, Da Silva MLCP. Sugarcane bagasse pulping and bleaching: Thermal and chemical characterization. BioResources. 2011; **6**(3):2471-2482
- [51] Morgado DL, Frollini E. Thermal decomposition of mercerized linter cellulose and its acetates obtained from a homogeneous reaction. Polímeros. 2011;**21**(2):111-117. DOI: 10.1590/S0104-14282011005000025
- [52] Oluwasina O, Lajide L, Owolabi B. Microcrystalline cellulose from plant waste through sodium hydroxide-anthraquinone-ethanol pulping. Bio