



## Characterization of microcrystalline cellulose extracted from walnut and apricots shells by alkaline treatment

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**Abstract:** In this study, microcrystalline cellulose (MCC) was isolated from walnut and apricot shells (WS and AS) as agricultural wastes in order to use it as reinforcement in polymer composites. The microcrystalline cellulose was extracted by alkaline treatment and bleached by peroxide as an environmentally friendly treatment, called walnut cellulose (WC) and apricot cellulose (AC). The chemical composition of the samples was set according to the Technical Association of Pulp and Paper Industry (TAPP). After treatments, the  $\alpha$ -cellulose content increased by about 23 % for the two used cellulose sources. The structural and morphological properties of the samples were investigated by Fourier transform infrared spectroscopy in the attenuated total reflectance mode (ATR-FTIR), optical microscopy (OM), X-ray diffraction and scanning electron microscopy (SEM). The crystallinity index values evaluated for WC and AC *via* X-ray diffraction were 86.4 and 80.3 %, respectively. The alkaline soluble fractions of walnut (ASW) and apricot (ASA) shells were recovered and characterized by OM and ATR-FTIR spectroscopy. Furthermore, their chemical composition was analyzed. The characterization and the properties of the WC and AC were similar to those of commercial MCC and MCC prepared in the literature from wood and some agricultural wastes.

**Keywords:** agricultural wastes; walnut shell; apricot shell; microcrystalline cellulose; crystallinity index.

### INTRODUCTION

Over the last decade, biopolymers and natural fiber-reinforced plastics have become an interesting alternative to replace petroleum-based plastics. For this, academic and industrial research use wastes generated by agriculture in order to develop environmentally friendly composites with good thermal stability,<sup>1,2</sup> mechanical properties<sup>1–3</sup> and biodegradable.<sup>2</sup> These wastes consist of fibrous

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vegetable tissues, mainly composed of cellulose, hemicelluloses and lignin, as the major constituents, considered as bio-composites, and of pectin, waxes, proteins, lipids, ash and extractive compounds that could be found as minor constituents.<sup>3</sup> Generally, a good dispersion of cellulose in the matrix of polymers enhance their mechanical and barrier properties of gases and vapors.<sup>4</sup> Thus, some researchers used lignin as a coupling agent to improve the interfacial adhesion between cellulose fibers and polymer matrices, and to enhance the thermal stability of the composites.<sup>5</sup> Recall that cellulose is the most abundant renewable polymer in the world. In cellulose fibers, the cellulosic chains are arranged in ordered crystallites and less-ordered amorphous regions, while lignin and hemicelluloses are amorphous components. Cellulose fibers found their first application in paper and clothing manufacturing, due to their high stiffness, low density and low cost. Cellulose fibers can be extracted from various agricultural wastes, such as wood, bamboo, flax, wheat,<sup>6</sup> almond shell,<sup>7</sup> corn stover,<sup>8</sup> sisal,<sup>9</sup> etc. For this, researchers developed different processes and treatments to valorize it, such as cellulose fibers,<sup>10</sup> cellulose crystals,<sup>7</sup> microfibrils,<sup>11</sup> microcrystalline cellulose,<sup>12</sup> nanofibrils,<sup>6</sup> or cellulose nanocrystals.<sup>13</sup>

The isolation of cellulose fibers requires the removal of other components, such as lignin, hemicelluloses and pectin, from natural fibers. However, the physicochemical properties depend on the method used and the raw material. Rajeshkumar *et al.*<sup>14</sup> proposed an environmental process to extract cellulose fibers from petioles of *Phoenix Sp.* plants. It consists of immersing these petioles in water for 15 to 20 days, then peeling them manually. On the other hand, Belouadah *et al.*<sup>15</sup> proposed the same procedure followed by manually brushing using a comb with metal teeth for *Lygeum spartum L.* Some researchers extracted cellulose fibers just by alkaline treatment.<sup>16</sup> However generally, the microcellulose fibers extracted by chemical treatment undergo alkaline treatment in order to remove the hemicelluloses and soluble lignin fraction, followed by bleaching with sodium hypochlorite in the presence of acetic acid, in order to remove the lignin part.<sup>17</sup> Chen *et al.*<sup>6</sup> and Reddy *et al.*<sup>11</sup> first dewaxed their samples with benzene and ethanol mixture using a Soxhlet apparatus. Cellulose crystals were extracted from almond shells by the same process but not dialyzed, vacuum-dried<sup>7</sup> and the cellulose nanocrystals derived from walnut shell were isolated using the same process reinforced by homogenization and ultrasonication processes.<sup>13</sup> Microcrystalline cellulose was isolated from palm oil fronds by an original process starting with alkaline hydrogen peroxide, followed by mechanical fibrillation, and then a solution of sodium chlorite was added to set pH to 4–5. Finally, this fiber was hydrolyzed in hydrochloric acid and the microcrystalline cellulose was dialyzed than dried in a vacuum oven.<sup>18</sup> Others precipitated the soluble lignin, after alkaline treatment or after alkaline peroxide, using HCl.<sup>19</sup>

The aim of the present work was to extract microcrystalline cellulose (MCC) by alkaline treatment and environmentally peroxide bleaching and remove the residual fraction after alkaline treatment from two agricultural residues (apricot and walnut shells). To that end, an alkaline treatment was carried out to remove the hemicelluloses and the soluble lignin fraction in order to isolate it, and peroxide bleaching was carried out to eliminate the insoluble lignin and isolate the MCC. To date, MCC has not been extracted from apricot shell and very few studies on walnut shells have been reported (Zheng *et al.*,<sup>20</sup> Harini *et al.*<sup>21</sup> and Hemmati *et al.*<sup>13</sup>). The structure and morphology of this cellulose were characterized by different techniques, *i.e.*, optical microscopy, scanning electron microscopy (SEM), Fourier transform infrared spectroscopy (FTIR), and X-ray diffraction (XRD). The removed fraction after alkaline treatment was analyzed using optical microscopy and FTIR spectroscopy. The chemical compositions of fibers and removed fraction were analyzed as per TAPPI standards.

## EXPERIMENTAL

### *Materials*

Walnut (WS) and apricot (AS) shell fibers were used as native cellulose fibers. The fruits come from a local market in Algiers (Algeria). Hydrogen peroxide (H<sub>2</sub>O<sub>2</sub>, 50 %) was purchased from Distrim. Hydrochloric acid (37 %) and sulfuric acid (96 %) were purchased from Carlo Erba Reagents. Sodium hydroxide (98 %) and acetic acid (99.8 %) were supplied by Scharlab S.L. Methanol was purchased from Chromanorm®. Ethanol (≥ 97 %), Ferrouin solution (C<sub>36</sub>H<sub>24</sub>FeN<sub>6</sub>O<sub>4</sub>S, 0.1 wt. %) and sodium metasilicate pentahydrate (≥ 97 %) were supplied by Sigma–Aldrich. Benzene (99.5 %) was purchased from Riedel de Haen. Dichloromethane (CH<sub>2</sub>Cl<sub>2</sub>, 99.8 %) was supplied by VWR Prolabo. Sodium chlorite (80 %) was purchased from Fluka, Potassium dichromate (99.97 %) and ferrous ammonium sulfate hexahydrate ((NH<sub>4</sub>)<sub>2</sub>Fe(SO<sub>4</sub>)<sub>2</sub>·6H<sub>2</sub>O) were respectively supplied by Labosi and Biochem.

### *Extraction of cellulose fibers from walnut and apricot shells*

The walnut and apricot shells were washed with distilled water five times at 90 °C during 2 h. After filtration, these shells were directly crushed with a knife grinder. Dried product was sieved and chemically purified according to the following steps: The powders of the walnut and apricot shells (WS and AS), between 40 and 100 μm in size, were treated with 5 % NaOH for 4 h at 80 °C in order to remove the alkaline-soluble components, such as soluble lignin, hemicelluloses and pectin. After this treatment, the fibers were centrifuged and washed with distilled water. The residual fibers were then dried in a hot air oven at 80 °C and crushed with a knife grinder. The definition of the sample code used is illustrated in Table I.

### *Recovery of alkaline soluble fraction*

The alkaline soluble fraction of the WS and AS was precipitated at pH 1.5 adjusted by HCl from the supernatant solution of the alkaline treatment. The solution was centrifuged and washed with distilled water and the residual fraction was recuperated and dried at 60 °C.

### *Preparation of microcrystalline cellulose from walnut and apricot shells*

The dried powder treated with alkaline solution was bleached using alkaline peroxide as an environmentally friendly treatment. In the alkaline media, H<sub>2</sub>O<sub>2</sub> allows the formation of the hydroperoxide anion (–OOH) that reacts strongly with the chromophores and phenolic ring-

conjugated ethylenic or carbonyl groups present in lignin and remove them permanently.<sup>22,23</sup> The powder was treated with 5 % hydrogen peroxide solution in the presence of 1 % of sodium metasilicate pentahydrate, the pH of this solution was adjusted at  $9.7 \pm 0.3$  using 10 % of NaOH solution. During the first hour, the fibers were treated without any heating (exothermic reaction). The reaction was realized at  $80^\circ\text{C}$  during the following three hours.<sup>22</sup>

TABLE I. Definition of each sample code

Sample code	Definition of sample code
WS	Walnut shell
WC	Walnut cellulose
WTA	Alkaline soluble fraction of walnut
AS	Apricot shell
AC	Walnut cellulose
APA	Alkaline soluble fraction of apricot shell

The fibers were treated with  $\text{H}_2\text{O}_2$  at pH  $9.7 \pm 0.3$ , four times successively, in order to remove the greatest amount of lignin from the fiber and to obtain white colored fibers. The treated fibers were isolated by centrifugation and washed 3 times after each treatment. After the last treatment, the fibers were washed to a constant pH. Then they were dried at  $60^\circ\text{C}$ , crushed with a knife grinder and sieved to produce fibers smaller than  $40\ \mu\text{m}$ .

#### *Fiber characterization*

The chemical composition of the untreated and chemically treated fibers was determined in accordance with the standards of the Technical Association of the Pulp and Paper Industry (TAPPI). The extractives were removed with an ethanol–benzene mixture according to the T 204 cm-97. The holocellulose content was determined as described by Yokoyama,<sup>24</sup> by the selective degradation of the lignin polymer by sodium chlorite at  $70^\circ\text{C}$  in the presence of acetic acid. The  $\alpha$ -cellulose content was determined by the removal of  $\beta$  and  $\gamma$ -cellulose from the holocellulose by alkaline extraction according to TAPPI T203 om-88. The acid soluble lignin content was analyzed by reaction with sulfuric acid using a standard method recommended in TAPPI-T222 om-88.

Fourier transform infrared spectroscopy (FTIR) measurements were performed using a Spectrum Two Perkin Elmer spectrometer, in the attenuated total reflectance (ATR) mode. The samples were scanned 10 times in the range  $650\text{--}4000\ \text{cm}^{-1}$ , with a resolution of  $4\ \text{cm}^{-1}$ . Microscopic photographs for the untreated WS, AS and for the WC, AC, WTA and APA were obtained using a Zeiss Axioskop coupled with an Optimas 1.5 picture analysis system. The magnification was  $100\times$  and  $1000\times$ . The surface morphologies of samples were investigated using a JEOL<sup>®</sup> JSM-6360 model with diverse voltages (5, 10, 15 kV) in order to observe the structure of the walnut and apricot shells (WS and AS) and the microcrystalline cellulose extracted from these shells (WC and AC). The crystallinity of the WS, WC, AS and AC was determined with a PANalytical XPERT-PRO diffractometer fitted with copper anode (Cu) under ( $\lambda = 1.5406\ \text{\AA}$ ) wavelength and at 40 kV and 45 mA generator settings. Sample analysis was performed in the range  $3\text{--}70^\circ$  of the  $2\theta$  region. The crystallinity index (*CrI*) was calculated by the empirical method proposed by the Segal formula (1959) for native cellulose:<sup>25</sup>

$$CrI = 100 \frac{I_{200} - I_{am}}{I_{200}} \quad (1)$$

where  $I_{200}$  denotes the maximum intensity of the 200 peak near to  $2\theta = 22.5^\circ$  and  $I_{am}$  is the intensity of the background scatter measures at  $2\theta$  value near  $18^\circ$ .

## RESULTS AND DISCUSSION

### *Chemical composition*

The chemical composition differs between different plants and different parts of the same plant, according to the geographic location, age, climate and soil conditions, among other factors. The chemical composition of the walnut and the apricot shells used for this study are presented in Table II.

TABLE II. Chemical analysis for untreated and treated wastes

Chemical composition	Content, %					
	Walnut			Apricot		
	WS	WC	WTA	AS	AC	APA
Extractives	4.3	1.8	13.7	4.4	1.3	11.2
Acid soluble lignin	35.4	13.4	66.7	32.5	11.0	60.9
Holocellulose	60.6	85.0	8.8	63.0	87.0	12.4
$\alpha$ -Cellulose	51.6	75.8	–	51.9	75.3	–
Hemicelluloses	9.0	9.1	–	11.1	11.7	–

The holocellulose contents found in walnut and apricot shell were in the range of 60.6 and 63 %, respectively. They were in the range found for Giant reed<sup>12</sup> and Posidonia balls<sup>26</sup> and somewhat higher those found for Posidonia leaves,<sup>26</sup> Sugar palm<sup>27</sup> and Peanut shell.<sup>28</sup> The contents of holocellulose and  $\alpha$ -cellulose increased after treatment in the WS and AS. This enhancement is due to the extractive removal lignin after the set treatments. The contents of holocellulose were enhanced by 24.4 and 24 %, respectively, for WS and AS after treatment. At the same time, the content of  $\alpha$ -cellulose increased by 18.2 and 23.4 % for WS and AS, respectively.

The acid soluble lignin contents found, respectively, for WS and AS fibers were 35.4 and 32.5 %. These results seem considerably higher than the lignin content in bamboo and wood, (20–25 %)<sup>6</sup> and lower than that found in piassava, ( $\approx$ 48 %).<sup>29</sup> It is similar to the lignin content in Coir fiber (36.14 %)<sup>30</sup> and Peanut shell (35.2 %).<sup>27</sup>

The untreated fiber of walnut and apricot shells had a higher percentage of lignin, and a lower percentage of  $\alpha$ -cellulose than WC and AC; the bleaching treatment cannot eliminate the total lignin content. The alkaline soluble fractions of the walnut and apricot shells (WTA and APA) contain considerably more lignin than hemicelluloses and the extracted fractions.

### *ATR-FTIR spectroscopic characterization*

To study the effect of the treatment process on the composition of both WS and AS, FTIR spectra of each sample before treatment (the raw cellulose fiber),

after treatment (treated cellulose fiber) and after isolation of lignin (soluble alkaline fraction) were recorded and are compared in Fig. 1a and b.

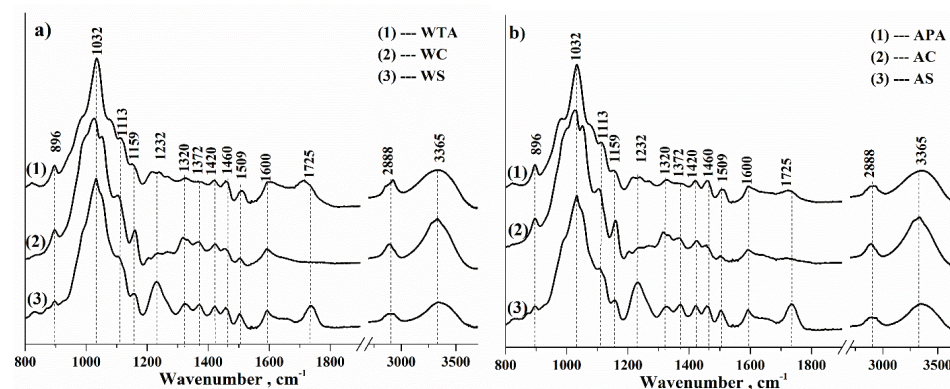


Fig. 1. FTIR spectra of WS, WC and WTA (a) and AS, AC and APA (b).

This FTIR spectroscopic analysis is in good agreement with the chemical analysis for all the samples. A wide band at around  $3360\text{ cm}^{-1}$ , due to the stretching vibration of the hydrogen bonded hydroxyl group, is observed. It becomes more prominent for the AC and WC samples as the results of the increased availability and content of cellulose (75 %  $\alpha$ -cellulose), *i.e.*, newly exposed cellulose hydroxyl groups.<sup>31,32</sup> The band at around  $2900\text{ cm}^{-1}$  for all samples is attributed to the aliphatic C–H stretching vibration in methyl ( $\text{CH}_3$ ) and methylene ( $\text{CH}_2$ ) functional groups. The band at  $1460\text{ cm}^{-1}$  represents the aromatic ring C=C of the phenyl propane group of lignin.<sup>33</sup> and the asymmetric bending of  $-\text{CH}_3$  group,<sup>34</sup> while the band at  $1422\text{ cm}^{-1}$  corresponds to vibrations of the aromatic skeletal and ring breathing with C–O stretching in lignin.<sup>35</sup> The intensity of these bands decreased after the treatment due to the removal of lignin. The band at around  $1730\text{ cm}^{-1}$  was only observed in the spectra of WS and AS. It is attributed to the stretching vibration of the carbonyl group (C=O) of hemicelluloses present in these shells.<sup>17,34</sup> This band disappeared after alkaline and bleaching treatments, but just decreased for WTA and APA due to the reduction of hemicelluloses content. These results are in good agreement with the variation of the chemical composition according to the treatments performed, which are summarized in Table II. The bands at  $1600$  and  $1509\text{ cm}^{-1}$ , significantly reduced in WC and AC, are attributed to symmetrical stretching vibration of C=C in plane of the aromatic ring present in lignin.<sup>34</sup> The band at  $1230\text{ cm}^{-1}$ , which is present in the spectra of WS and AS, is attributed to the out of plane stretching vibration of C–O of aryl group present in the lignin part. It was significantly reduced in the WC and AC samples. This reduction is due to the removed lignin in WC and AC, as seen in Table II. The bands at  $1320$  and  $1370$



$\text{cm}^{-1}$  are attributed, respectively, to the asymmetric and symmetric stretching of  $-\text{COOH}$  and the bonding of  $\text{CH}_2$ .<sup>36</sup> The band at  $1032 \text{ cm}^{-1}$  is attributed to the stretching mode of  $\text{C}-\text{O}-\text{C}$  bonds in the glucosidic rings.<sup>36</sup> The absorption band at  $1155 \text{ cm}^{-1}$  is attributed to  $\text{C}-\text{O}-\text{C}$  asymmetric stretch vibration in cellulose and hemicelluloses, while the band at  $894 \text{ cm}^{-1}$  is ascribed to the stretching vibration of  $\text{C}_1-\text{O}-\text{C}$  of  $\beta$ -(1-4)-glycosidic linkages in cellulose I and the amorphous fraction. The absorbance of these bands is more intense after alkaline and bleaching treatment due to the increase of cellulose content. The band at around  $840 \text{ cm}^{-1}$  associated with the band at  $894 \text{ cm}^{-1}$  observed in WTA and APA confirmed the presence of 1,2,4-tetrasubstituted aromatics in the lignin.<sup>37</sup>

#### *Optical microscopy*

Microscopic photographs at magnifications of  $100\times$  and  $1000\times$  were taken for the untreated WS, AS and for WC, AC, WTA and APA (Fig. 2). After the bleaching treatment, microscopic photographs at the same magnifications as for WC and AC were taken.

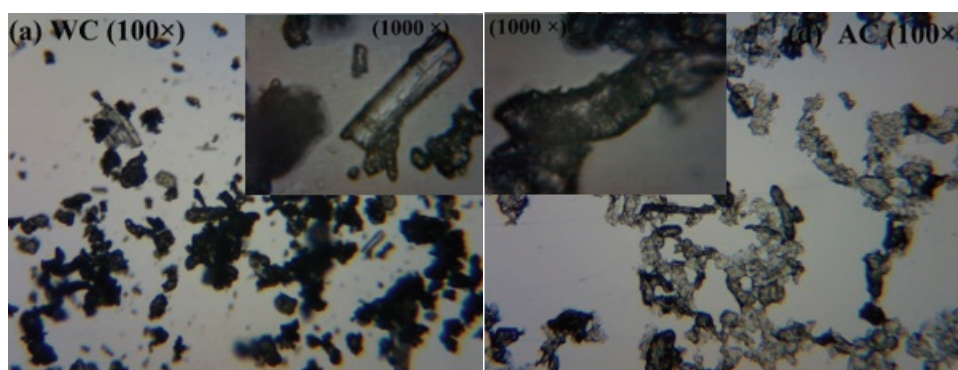


Fig. 2. Optical microscopic images of microcrystalline cellulose WC (a) and AC (b).

The microscopic photographs of untreated WS and AS show that a mixture of almost totally spherical shapes and some cylindrical shapes of inhomogeneous size, notably for the AS, were obtained. After treatment, microcrystalline cellulose particles of more cylindrical shape, in particular for WC, were obtained (Fig. 2). Their surface was smoother than that of the initial cellulose.

#### *X-Ray diffraction analysis*

The XRD results for microcrystalline cellulose derived from WS and AS before and after treatment are illustrated in Fig. 3a and b, respectively. The X-ray diffraction patterns of the samples are a combination of the diffraction of the amorphous region from lignin and hemicelluloses and ordered crystallites and less-ordered amorphous regions from the cellulosic chains. The XRD analysis of these samples (WS, WC, AS and AC) revealed a typical cellulose I $\beta$  structure.<sup>38</sup>

They have three common peaks for all samples located at  $2\theta$  14.8, 16.2 and  $22.4^\circ$  characterized the presence of cellulose  $I_\beta$ .<sup>38,39</sup>

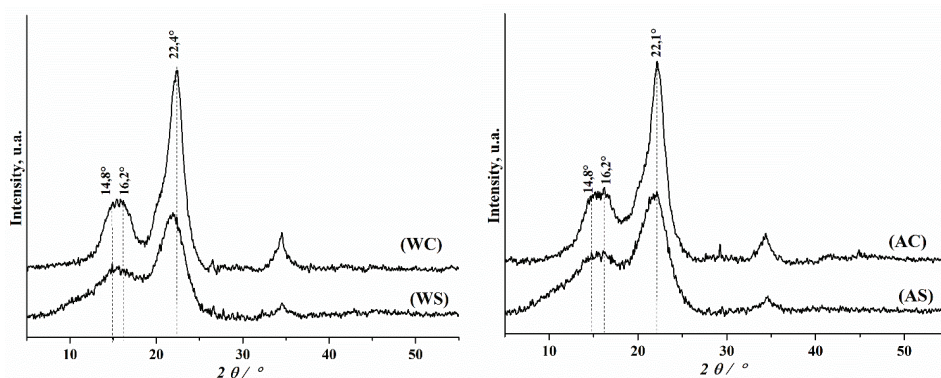


Fig. 3. XRD patterns for WS and WC (a) and AS and AC (b).

The peaks located at  $2\theta$  of 14.8 and  $16.2^\circ$  have the positions of (101) and (101-) crystallographic plane reflections, respectively,<sup>40,41</sup> the peak located at  $22.4^\circ$  has the positions of (200) crystallographic plane reflections.<sup>38,39</sup> The XRD results fit well with cellulose  $I_\beta$  crystals and the degrees of crystallinity were calculated using the Eq. (1). After chemical treatment of the raw materials, the crystallinity index increased from 55.2 and 60.4 %, for WS and AS to 86.4 and 80.3 % for WC and AC, respectively. Similar values were reported in literature.<sup>42</sup> These results indicate that after the treatments, the regions with a random amorphous structure, such as hemicelluloses and lignin, had dissolved and were removed.<sup>43</sup>

#### SEM characterization

SEM characterization was performed in order to evaluate the changes in surface morphology of WS and AS after treatment. Images of WS, AS, WC and AC are shown at  $2000\times$  magnification in Fig. 4.

The SEM images of the raw materials (WS and AS) show non-uniform particles with a rough and irregular surface. This irregularity may be attributed to the presence of impurities, waxes, hemicelluloses and lignin on its surface.<sup>44</sup> The presence of pores is clearly visible on the surface of WS and AS. After treatment, more uniform particles could be seen. The WC and AC have, respectively, more cylindrical and spherical shapes but not a completely rough surface due to the hydrogen-bonding present between the cellulose fibers.<sup>45</sup>

From the SEM micrographs of WS, AS, WC and AC (Fig. 4), it is evident that a profound morphological change in the shape of the raw material occurred after treatment. Yet, a high level of aggregation and agglomeration was observed due to the hydrophilic nature of the cellulose surface. The morphological shapes



of the microcrystalline cellulose WC and AC are similar to those of commercial microcrystalline cellulose reported by many authors, such as Karim.<sup>45</sup>

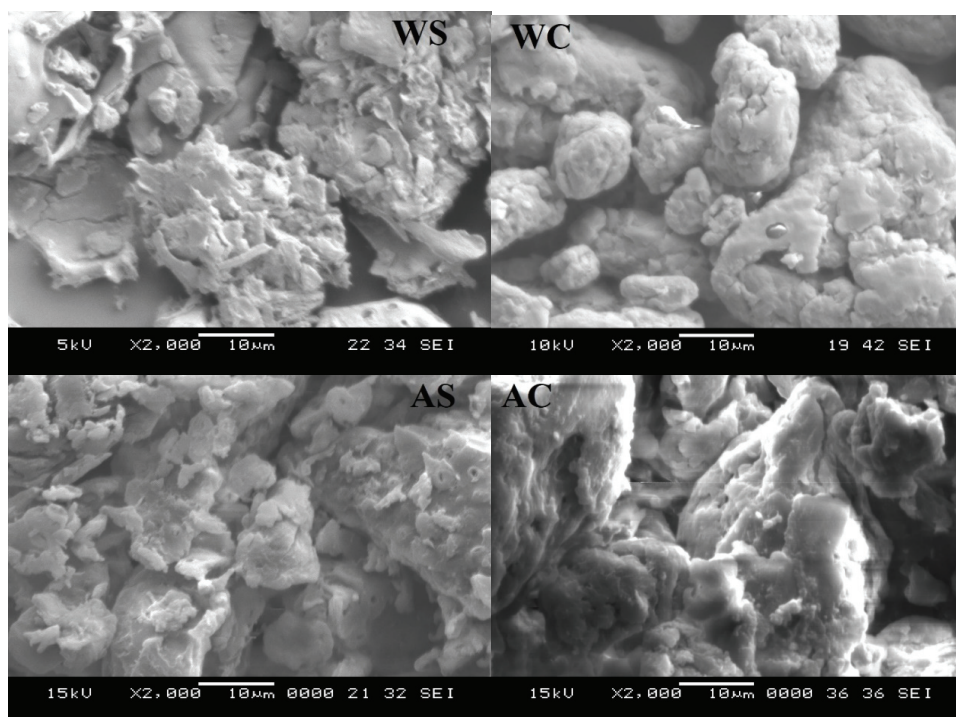


Fig. 4. Scanning electron microscopy images of different fiber surfaces (WS, WC, AS and AC).

#### CONCLUSIONS

MCC was successfully prepared from walnut and apricot shells, using only mercerization (NaOH) and bleaching with alkaline peroxide. The chemical compositions of WS and AS are very similar considering the nature of their similar sources. The chemical composition analysis showed an increasing trend in the holocellulose and  $\alpha$ -cellulose contents after the chemical treatment. The  $\alpha$ -cellulose content changed from 51.6 % in WS to 75.8 % in WC and from 51.9 % in AS to 75.3 % in AC. The ATR-FTIR spectra and chemical compositions are in agreement and show, on the one hand, that the ratio of lignin had considerably decreased by 35.4 and 32.5 % in WC and AC, respectively, but are not completely eliminated by the method used in this work. On the other hand, they show that alkaline treatment allows considerable quantities of extractable lignin to be extracted. This alkaline soluble fraction can easily be recovered by acid precipitation. Microscopic characterizations showed the morphological changes occurring on WS and AS fibers during the treatment. The X-ray diffraction analysis

showed a high crystallinity index (calculated by the Segal formula) of 86.4 and 80.3 % for WC and AC, respectively. This crystallinity index is good enough and in agreement with the crystallinity index of commercial MCC.

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## ИЗВОД

## КАРАКТЕРИЗАЦИЈА МИКРОКРИСТАЛНЕ ЦЕЛУЛОЗЕ ЕКСТРАХОВАНЕ ИЗ ЉУСКИ ОРАХА И КОШТИЦА КАЈСИЈЕ АЛКАЛНИМ ТРЕТМАНОМ

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У овом раду је микрористална целулоза (МСС) изолована из љуски ораха (WS) и коштица кајсије (AS) као пољопривредног отпада у циљу добијања пунилаца за ојачавање полимерних композита. МСС је екстрахована алкалним третманом и избељена пероксидом као еколошки прихватљивим поступком, чиме су добијени узорци целулозе WC (од љуске ораха) и AC (од коштице кајсије). Хемијски састав узорака је одређен према стандардима Техничке асоцијације индустрије целулозе и папира (TAPP). Након третмана, садржај  $\alpha$ -целулозе се повећао за око 23 % за оба коришћена материјала. Структурна и морфолошка својства узорака су испитивана инфрацрвеном спектроскопијом са Фуријеовом трансформацијом у тоталној рефлексiji (ATR-FTIR), оптичком микроскопијом (OM), рендгенском дифракционом анализом (XRD) и скенирајућом електронском микроскопијом (SEM). Индекс кристалинности, одређен на основу резултата XRD анализе, износи 86,4 % за WC и 80,3 % за AC. Алкално растворљиве фракција WS и AS (ASW и ASA, редом) су издвојене и карактерисане оптичком микроскопијом и ATR-FTIR спектроскопијом. Такође, одређен је и хемијски састав. Карактеристике WC и AC су сличне карактеристикама комерцијалних МСС и узорака целулозе који су у литератури припремљени од дрвета и неких пољопривредних отпадних материјала.

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## REFERENCES

1. N. Saba, A. Safwan, M. L. Sanyang, F. Mohammad, M. Pervaiz, M. Jawaid, O. Y. Alothman, M. Sain, *Int. J. Biol. Macromol.* **102** (2017) 822 (<https://doi.org/10.1016/j.ijbiomac.2017.04.074>)
2. K. G. Satyanarayana, G. G. C. Arizaga, F. Wypych, *Prog. Polym. Sci.* **34** (2009) 982 (<https://doi.org/10.1016/j.progpolymsci.2008.12.002>)
3. M. El Achabya, Z. Kassaba, A. Barakat, A. Aboukassd, *Ind. Crops Prod.* **112** (2018) 499 (<https://doi.org/10.1016/j.indcrop.2017.12.049>)
4. L. Ugarte, A. Santamaria-Echart, S. Mastel, M. Autore, R. Hillenbrand, M. A. Corcuera, A. Eceiza, *Ind. Crops Prod.* **95** (2017) 564 (<http://dx.doi.org/10.1016/j.indcrop.2016.11.011>)
5. S. S. Nair, P.-Y. Kuo, H. Chen, N. Yan, *Ind. Crops Prod.* **100** (2017) 208 (<http://dx.doi.org/10.1016/j.indcrop.2017.02.032>)
6. W. Chen, H. Yu, Y. Liu, Y. Hai, M. Zhang, P. Chen, *Cellulose* **18** (2011) 433 (<http://dx.doi.org/10.1007/s10570-011-9497-z>)

7. N. Maaloul, R. B. Arfi, M. Rendueles, A. Ghorbal, M. Diaz, *J. Mater. Environ. Sci.* **8** (2017) 4171 ([http://www.jmaterenvironsci.com/Document/vol8/vol8\\_N11/438-JMES-2556-Maaloul.pdf](http://www.jmaterenvironsci.com/Document/vol8/vol8_N11/438-JMES-2556-Maaloul.pdf))
8. L. A. S. Costa, D. de J. Assis, G. V. P. Gomes, J. B. A. da Silva, A. F. Fonsêca, J. I. Druzian, *Mater. Today-Proc.* **2** (2015) 287 (<https://doi.org/10.1016/j.matpr.2015.04.045>)
9. J. I. Morán, V. A. Alvarez, V. P. Cyras, A. Vázquez, *Cellulose* **15** (2008) 149 (<https://doi.org/10.1007/s10570-007-9145-9>)
10. M. Maache, A. Bezazi, S. Amroune, F. Scarpac, A. Dufresne, *Carbohydr. Polym.* **171** (2017) 163 (<http://dx.doi.org/10.1016/j.carbpol.2017.04.096>)
11. J. P. Reddy, J.-W. Rhim, *J. Nat. Fibers* **15** (2018) 465 (<https://doi.org/10.1080/15440478.2014.945227>)
12. A. F. Tarchoun, D. Trache, T. M., Klapötke, M. Derradji, W. Bessa, *Cellulose* **26** (2019) 7635 (<https://doi.org/10.1007/s10570-019-02672-x>)
13. F. Hemmati, S. M. Jafari, M. Kashaninejad, M. B. Motlagh, *Int. J. Biol. Macromol.* **120** (2018) 1216 (<https://doi.org/10.1016/j.ijbiomac.2018.09.012>)
14. G. Rajeshkumar, V. Hariharan, T. P. Sathishkumar, *J. Ind. Text.* **46** (2016) 667 (<https://doi.org/10.1177/1528083715591581>)
15. Z. Belouadah, A. Ati, M. Rokbi, *Carbohydr. Polym.* **134** (2015) 429 (<http://dx.doi.org/10.1016/j.carbpol.2015.08.024>)
16. P. Senthamaraikannan, M. Kathiresan, *Carbohydr. Polym.* **186** (2018) 332 (<https://doi.org/10.1016/j.carbpol.2018.01.072>)
17. I. M. Fareez, N. A. Ibrahim, W. M. H. W. Yaacob, N. A. M. Razali, A. H. Jasni, F. A. Aziz, *Cellulose* **25** (2018) 4407 (<https://doi.org/10.1007/s10570-018-1878-0>)
18. A. F. Owolabi, M. K. M. Haafiz, Md. S. Hossain, M. H. Hussind, M. R. N. Fazita, *Int. J. Biol. Macromol.* **95** (2017) 1228 (<http://dx.doi.org/10.1016/j.ijbiomac.2016.11.016>)
19. R. C. Sun, X. F. Sun, J. L. Wen, *J. Agric. Food Chem.* **49** (2001) 5322 (<http://dx.doi.org/10.1021/jf010645y>)
20. D. Zheng, Y. Zhang, Y. Guo, J. Yue, *Polymers* **11** (2019) 1130 (<https://doi.org/10.3390/polym11071130>)
21. K. Harini, C. C. Mohan, *Int. J. Biol. Macromol.* **163** (2020) 1375 (<https://doi.org/10.1016/j.ijbiomac.2020.07.239>)
22. R. C. Sun, J. M. Fang, J. Tomkinson, *Ind. Crop. Prod.* **12** (2000) 71 ([https://doi.org/10.1016/S0926-6690\(00\)00039-X](https://doi.org/10.1016/S0926-6690(00)00039-X))
23. S. I. Mussatto, G. J. M. Rocha, I. C. Roberto, *Cellulose* **15** (2008) 641 (<https://doi.org/10.1007/s10570-008-9198-4>)
24. T. Yokoyama, J. F. Kadla, H.-M. Chang, *J. Agric. Food Chem.* **50** (2002) 1040 (<https://doi.org/10.1021/jf011173q>)
25. L. Segal, J. J. Creely, A. E. Martin, C. M. Conrad, *Text. Res. J.* **29** (1959) 786 (<https://doi.org/10.1177/004051755902901003>)
26. F. Bettaieb, R. Khiari, M. L. Hassan, M. N. Belgacem, J. Bras, A. Dufresne, M. F. Mhenni, *Ind. Crops Prod.* **72** (2015) 175 (<https://doi.org/10.1016/j.indcrop.2014.12.038>)
27. R. Ilyas, S. Sapuan, M. Ishak, *Carbohydr. Polym.* **181** (2018) 1038 (<https://doi.org/10.1016/j.carbpol.2017.11.045>)
28. S. Naduparambath, E. Purushothaman, *Cellulose* **23** (2016) 1803 (<https://doi.org/10.1007/s10570-016-0904-3>)
29. J. R. M. d'Almeida, R. C. M. P. Aquino, S. N. Monteiro, *Compos., A* **37** (2006) 1473 (<https://doi.org/10.1016/j.compositesa.2005.03.035>)

30. A. Espert, F. Vilaplana, S. Karlsson, *Compos., A* **35** (2004) 1267 (<https://doi.org/10.1016/j.compositesa.2004.04.004>)
31. J.-H. Chen, J.-G. Liu, Y.-Q. Su, Z.-H. Xu, M.-C. Li, R.-F. Ying, J.-Q. Wu, *Carbohydr. Polym.* **206** (2019) 616 (<https://doi.org/10.1016/j.carbpol.2018.11.024>)
32. L. Bergamonti, M. Potenza, A. Haghghi Poshtiri, A. Lorenzi, A. M. Sanangelantoni, L. Lazzarini, P. P. Lottici, C. Graiff, *Carbohydr. Polym.* **231** (2020) 115773 (<https://doi.org/10.1016/j.carbpol.2019.115773>)
33. B. D. Mistry, *A Handbook of Spectroscopic Data Chemistry (UV, IR, PMR, 13CNMR and Mass Spectroscopy)*, Oxford Book Co., Jaipur, 2009, p. 42
34. C. Yadav, A. Saini, P. K. Maji, *Carbohydr. Polym.* **165** (2017) 276 (<http://dx.doi.org/10.1016/j.carbpol.2017.02.049>)
35. B. H. Stuart, *Infrared spectroscopy: fundamentals and applications*, Wiley, Chichester, 2004, p. 77
36. F. Niu, M. Li, Q. Huang, X. Zhang, W. Pan, J. Yang, J. Li, *Carbohydr. Polym.* **165** (2017) 197 (<http://dx.doi.org/10.1016/j.carbpol.2017.02.048>)
37. Boukir, S. Fellak, P. Doumenq, *Heliyon*. **5** (2019) e02477 (<http://dx.doi.org/10.1016/j.heliyon.2019.e02477>)
38. A. D. French, *Cellulose* **21** (2014) 885 (<http://dx.doi.org/10.1007/s10570-013-0030-4>)
39. M.-J. Chen, X.-Q. Zhang, A. Matharu, E. Li, R.-M. Melo, C.-F. Liu, Q.-S. Shi, *ACS Sustain. Chem. Eng.* **5** (2017) 7278 (<http://dx.doi.org/10.1021/acssuschemeng.7b01526>)
40. R. Liu, H. Yu, Y. Huang, *Cellulose* **12** (2005) 25 (<http://dx.doi.org/10.1007/s10570-004-0955-8>)
41. K. Singh, T. J. M. Sinha, S. Srivastava, *Int. J. Miner. Process.* **139** (2015) 51 (<http://dx.doi.org/10.1016/j.minpro.2015.04.014>)
42. K. Zhang, Y. Zhang, D. Yan, C. Zhang, S. Nie, *Cellulose* **25** (2018) 5049 (<http://dx.doi.org/10.1007/s10570-018-1928-7>)
43. R. Li, J. Fei, Y. Cai, Y. Li, J. Feng, J. Yao, *Carbohydr. Polym.* **76** (2009) 94 (<http://dx.doi.org/10.1016/j.carbpol.2008.09.034>)
44. A. P. B. Gonçalves, C. S. de Miranda, D. H. Guimarães, J. C. de Oliveira, A. M. F. Cruz, F. L. B. M. da Silva, S. Luporinia, N. M. José, *Mater. Res.* **18** (2015) 205 (<http://dx.doi.org/10.1590/1516-1439.366414>)
45. M. Z. Karim, Z. Z. Chowdhury, S. B. Abd Hamid, M. E. Ali, *Materials* **7** (2014) 6982 (<http://dx.doi.org/10.3390/ma7106982>).