

Egyptian Journal of Chemistry

http://ejchem.journals.ekb.eg/



Synthesis and Description of Cellulose Nano-crystals from Sugarcane Bagasse, Saw Dust and Paper Waste



Nagwa I. Elkhyat^{1*}, Salah M. A. Saad¹, Farhat F.A. Foda^{1*} and Abdalla E. El-Hadary¹

¹ Faculty of Agriculture, Biochemistry Dept. Benha University, Benha 13736, Egypt.

Abstract

Naturally occurring cellulose fiber serves as the exporter of cellulose nanocrystals (CNCs), a novel substance. A substantial quantity of interest in CNCs was sparked by their mechanical, optical, and chemical qualities. This study explores the possibility from agricultural and industrial wastes that are not commercially recyclable such as sugarcane bagasse (SCB), sawdust (SD), and paper waste (PW) as a newresources to produce CNCs. X- ray Diffraction Spectroscopy(XRD) revealed that the crystallinity index of cellulose isolated from SCB was 45.18% which increased to 77.53% for the CNCs. But, these values of crystallinity index were found to be 59.00% and 87.11% for cellulose and CNCs produced from SD. While, the crystallinity index of CNCs produced from paper waste by chemical method was found to be higher (90.00%) than mechanical method (68.29%) when compared with cellulose isolated was lowest value (47.66%). Morphological characterization (TEM) showed the CNCs with the average dimensions diameter (18nm to 58nm) and length (193nm to 378nm) of SCB, SD diameter (47.41nm to 55.77nm) and length (277.13 to 350.20 nm) for chemical method. While, PW showed that diameter (30 to 40nm) and length (250nm to 577nm) for chemical and mechanical methods, respectively. From the above-mentioned data, it can be concluded that the CNCs synthesized from different agricultural and industrial wastes which is considered a cause of environmental pollution, as it was highlighted and transformed into nanomaterial for biomedical and various industrial applications.

Keywords: Agriculture wastes; Cellulose; Cellulose Nanocrystals; Sugarcane bagasse; Sawdust; Paper industry.

1. Introduction

Because agricultural waste has the potential to drastically damage the ecology, there has been increased worry about it recently. Millions of agricultural wastes are created because of the rise in agricultural product output. Burning the product's leftovers releases a lot of pollutants, suspended particles, and greenhouse gases. Nonetheless, there are several useful components in industrial and agricultural waste that may be recovered and used to create a variety of products. Among the materials that may be made from these wastes is cellulose. [1-3].

Because of their exceptional biocompatibility, high tensile strength, specific surface area, and renewable nature, nanocrystals made from agricultural waste are widely employed in many different sectors. Acid hydrolysis, enzymatic hydrolysis, oxidation hydrolysis, and other mechanical techniques can be used to manufacture cellulose from biomass waste. [4,5].

In general, mechanical, chemical, chemo-mechanical, and a combination of these techniques are used to create nanocellulose materials. To create extremely crystalline forms of cellulose, cellulose is often hydrolyzed using strong acids to make cellulose nanocrystals, [6]. Nanocellulose can be synthesized either in the form of nanofiber or nanocrysyals. The size, shape, and surface chemistry of fabrication cellulose nanoparticles depend on the process parameters, [7].

Sugarcane bagasse has attracted increasing attention due to higher biomass yields. It is a byproduct of the sugarcane production industry, comprising cellulose (43.6%), hemicellulose (33.8%), and lignin (18.1%) on a dry weight basis [8].

Nanocrystalline cellulose (CNCs) extracted and characterized from sugarcane bagasse (SCB) by using sulfuric acid hydrolysis. The crystallinity index, morphology, and structural properties of the cellulose and nanocellulose were carried out using an X-ray diffractometer (XRD), scanning electron microscope (SEM), and fourier transform infrared spectroscopy (FT-IR), respectively. According to calculations, the crystallinity indexes of sugarcane, extracted cellulose, and CNCs were found to be 58.9%, 70.4% and 77.1%, respectively.SEM analysis revealed agglomerated rod-like CNCs[9].

Cellulose isolated from sawdust and rice husk waste by chemical hydrolysis process, and the yields were found to be 13.45% and 22.75% (w/w) of cellulose from rice husk and sawdust wastes, respectively. While, chemical process caused partial removal of hemicellulose and lignin from raw material these results were confirmed by the FTIR spectrum. Using (XRD), the structure and morphological characteristics of cellulose from sawdust and rice husk waste were examined and the

*Corresponding author e-mail: farhat.foda@fagr.bu.edu.eg.nagwa.elkhayat@fagr.bu.edu.eg., (**Nagwa. I. Elkhyat**) Received date 29 January 2025; Revised date 26 March 2025; Accepted date 14 April 2025

DOI: 10.21608/EJCHEM.2025.356465.11232

results showed that cellulose structure has been converted from amorphous to crystalline form after the bleaching process

Also, cellulose nanocrystals were synthesized from Albizialebbeekwood sawdust. The chemical analysis was performed to determine the amount of lignocellulosic components in sawdust. According to the data, cellulose (40.72%), holocellulose (65.10%), hemicellulose (24.38%), lignin (25.67%), and ash (1.10%).[11]

Cellulose nanocrystals (CNCs) extracted from wood sawdust using an oxidizing agent and acid hydrolysis and morphological characterization of the synthesis CNCs by (FTIR) approved cellulose functional structures in the CNCs, (XRD) analysis indicated a high crystallinity index (78%) and the transmission electron microscope (TEM) analysis showed that typical nano-dimensions of 2.1–10 nm for diameter and 150–350 nm for length [12].

As an alternative to industrial raw materials [13] reported that waste paper is an effective raw material because of its high cellulose content and widespread availability content (60-70%) with comparatively less hemicelluloses (10-20%) and lignin (5-10%) and the range of yields for nanocellulose generation was (1.5% to 64%) nanocelluloses were found to have dimensions between 2 and 100 nm and crystallinity indexes between 54% and 95% contingent on the type of waste paper used and the applied procedures.

Cellulose nanocrystals (CNCs) synthesized from waste paper by acid hydrolysis process from the obtained results, (FTIR) for nanocellulose showed that no new chemical bonds were formed during the synthesis of nanocellulose. While, XRD showed that the crystallinity index of the (CNCs) produced in the treated and untreated paper industry was 66% and 73.8%, respectively cellulose nanocrystals discovered a natural needle-like fiber with a length of 300 nm to 600 nm, a diameter of 10 nm to 30 nm, and an aspect ratio in the range of 10 to 60nanometer[14].

The current study objective was to create CNCs from sugarcane bagasse, sawdust, and industrial paper waste using a combination of mechanical (ball milling) and chemical (acid hydrolysis) methods. The CNCs were expected to have a high crystallinity index, be kindly stable, and have surface morphology (size and shape) that made them suitable for use as reinforcing factors in the synthesis of cellulose nanocrystals for a range of applications. Through the study of SEM, TEM, FTIR, and XRD, the surface morphology and topography of the CNCs produced from SCB, SD by chemical method and PW by chemical and mechanical method to overcomes the risks of chemical methods.

2. Materials and Methods

Ligno-cellulosic substrates including sugarcane bagasse (SCB), sawdust (SD), and industrial paper waste (PW). (SCB) supplied by the juice shop in Toukh ,Qalyubia. While, (SD) collected from a local furniture factory in Moshtohor village in Toukh, Qalyubia Governorate, and (PW) supplied by Lotus Pack Paper Factory in Banha. These raw materials were washed thoroughly with hot distilled water and allowed to dry in the air at room temperature. All chemicals used in these experiments were provided by Sigma and Al-Gomhoria Chem. Co. of high quality and purity. In the laboratory of the science faculty of Benha university.

Chemical analysis and extraction of cellulosefrom SCB, SD, and PW:

The chemical composition (cellulose, hemicellulose, and lignin) of the (SCB), (SD), and PW were determined according to the quantitative analysis method described by [15,16]. While, delignification and cellulose extraction from the above materials by the method described by [17].

Synthesis of cellulose nanocrystals (CNCs):

Cellulose nanocrystals (CNCs) were synthesized from cellulose obtained from SCB and SD using hydrolysis as described in literature [18], though with slight modifications also. The extracted cellulose hydrolyzed with 32% (w/v) of H₂SO₄ with a 1:25 g/mL ratio of cellulose to the dilute acid at room temperature for 24 h under constant stirring. This reaction was then quenched by addition of 10 fold deionized water to the reaction mixture, followed by centrifugation at 10,000 rpm for 15 min three times to elute the acidic solution. The supernatant was discarded and the cellulosic precipitate re-dispersed in deionized water and dialyzed against deionized water several times. The colloidal suspension was then sonicated in an ice bath sonicator for 1 h to homogenize the generated cellulose nanocrystals. The generated nanocrystals were further centrifuged at 6, 000 rpm for 30 min. This was then allowed to settle for 24 h then the water was replaced with acetone and centrifuged at 6,000 rpm for 30 min. The cellulose nanocrystals were finally oven dried in vacuum at 70 C overnight. Conversely, though, CNCs from PW were obtained through two pretreatments: acid hydrolysis was done with 40% (w/v) of H₂SO₄ at 50 C for 1 hour. Then the solution was centrifuged at 6,000 rpm for 10 min and dialyzed for five days, and mechanical grinding by ball milling for 4 hours using zeconia balls [19].

Characterization of cellulose nanocrystals

X-ray Diffraction Spectroscopy (XRD):

Provides the calculation for determining whether or not cellulose nanocrystals are, on average, crystalline. The intensity of the rays that have been diffracted is displayed as a function of the diffraction angles on the diffractogram. The following formula was used to get the crystallinity index (CrI) [20].

$$CrI\% = \frac{I_{200} - I_{am}}{I_{200}} X100$$
 (1)

Where I₂₀₀ was the intensity at the plane (200) and I_{am} was the minimum intensity between the planes 110 and 200. I₂₀₀ represented both crystalline and amorphous portions, whereas I_{am} represented the amorphous portion. The spectra (XRD) (model number XRD 6000) illustrate the intricate characteristics of the crystal planes. : The samples were scanned over the

range of $2\theta = 5-40^{\circ}$ with a scanning rate of 0.4° /min at room temperature. The Debye-Scherrer equation was utilized to get an

$$\mathbf{D} = \frac{K\lambda}{\beta \cos \theta} \mathbf{A}^{o} \tag{2}$$

Where: \mathbf{D} = the average crystalline size in \mathbf{A}^{o}

 \mathbf{K} = the Scherrer constant (0.9).

accurate reading of the crystallites nanoparticles size [21].

 $\lambda = X$ -ray wavelength (1.5406 A°) s.

 β = peak width of half maximum.

 θ = the Bragg diffraction angle

Scanning electron microscopy (SEM)

The morphology of the produced CNCs, including their size and shape, was analyzed with scanning electron microscopy (SEM) with the model number JEOL JSM 6510 Iv by the approach developed by [22].

Fourier Transform Infrared Spectroscopy (FTIR)

The FTIR spectra of the samples under investigation were recorded on an instrument model NicoletTM iS10 (Shimadzu FTIR 8400) according to the method described by [23].

Transmission Electron Microscopy (TEM):

Morphological properties and particle sizes of CNCs samples were analyzed using (Tecnai G2 20 S-twin) Transmission Electron Microscopy, in the nano-fiber laboratory in October city. [24].

Statistical analysis

All data were expressed as means and standard error and statistically analyzed using SAS (Statical Analysis Software) SAS (2004). Statistical significance of differences among different study groups was evaluated by one way analysis of variance (Danken) ($P \le 0.05$). Duncan's multiple range tests was used to differentiate between means to determine differences between means of treatments at significance rates of ($P \le 0.05$).

Results and Discussion:

Chemical composition and characterization of SCB, SD, and PW:

The data on the proximate chemical composition of the agricultural and industrial wastes such as SCB, SD, and PW are shown in **Table (1)**. The obtained resulted in the proximate analysis such as moisture, crude fat, and crude protein contacts. SCB showed the highest content of moisture (2.77 ± 0.173^{a}) , while the ash and cellulose contents were found to be higher in PW $(7.073\pm0.114^{a},55.81\pm0.262^{a})$. On the other hand, the higher lignin content for SD was found to be (36.45 ± 0.513^{a}) . These results are different from those reported by **[8, 11,19]**.According to these results, the SCB, SD, and PW have a great potential for value-added use as cellulose sources that are renewable.

Table (1): Chemical composition of raw wastes of Sugarcane bagasse (SCB), Sawdust (SD), and Paper waste (PW).

Raw wastes	Moisture%	Ash%	Crude Protein%	Crude Fat%	Cellulose (%)	Hemicellulose (%)	Lignin (%)
Sugarcane							
bagasse	2.77±0.173 a	1.073±0.114°	1.007±0.063 ^a	1.180±0.062 ^a	48.07±0.262°	30.09±0.180 ^a	15.02±0.513 ^b
(SCB)							
Saw dust							
(SD)	1.14±0.173 ^b	2.097±0.114 ^b	0.317±0.063 ^b	0.560±0.053 ^b	40.19±0.262 ^b	18.10±0.180°	36.45±0.513 ^a
Paper waste							
(PW)	1.03±0.173 ^b	7.073±0.114 ^a	0.137±0.063 ^b	0.395±0.075 ^b	55.81±0.262 ^a	20.35±0.180 ^b	13.33±0.513 ^b

A,b,c:No significance difference (p>0.05) between means of treatments, within the same column and with the same superscript letter.

X-ray Diffraction Spectroscopy (XRD):

The XRD test to functionedto consider the crystalline structure of cellulose and CNCs produced from SCB, SD, and PW. The obtained results are presented in **Table (2)**, and **Fig. 1 (a,b,and c)**. The data in **Fig. 1(a)** showed that the samples of CNCs prepared from sugarcane bagasse (SCB) exhibited a peak around $2\theta = 15.57^{\circ}$ and 23.14° which are supposed to present the cellulose structure. The cellulose crystals exhibit characteristic assignment of 110, 200, and 004 planes, respectively. In

Egypt. J. Chem. 68, SI: Z. M. Nofal (2025)

the case of CNCs, the remaining amorphous portion was eliminated after acid hydrolysis, resulting in an increase in the crystallinity index of the SCB from 45.18% to 77.53% **Table (2).**

These XRD-derived crystallinity degree results show a similar pattern to the FTIR-derived crystallinity analysis result. Furthermore, our results align with those reported by [9]. Because SCB had a larger concentration of the amorphous element, it showed the lowest CrI in the experimental results. Crystallinity rose because of chemical alteration that used sodium hydroxide and sodium hypochlorite to remove the cellulose.

This is because the lignin and hemicellulose that were attached to the cellulosic bagasse were successfully removed. Moreover, sulfuric acid combines with the amorphous portion of SCB during the chemical treatment, resulting in the hydrolytic breakage of the glycosidic linkages and the release of individual crystallites, [21]. In the case of sawdust, (Fig. 1b) showed that two intense peaks were observed at a diffraction angle $(2\theta) = 19.33^{\circ}$ and 24.88° which correspond to (110) and (200) lattice planes of cellulose. A further very small peak, which corresponds to (004) of crystalline cellulose, was seen at $2\theta = 31.53^{\circ}$. The same sawdust-related diffraction peaks were observed following acid hydrolysis, but at a reduced intensity. This indicates that cellulose makes up the obtained CNCs crystal structure.

Whereas the crystallinity index for sawdust and CNCs was determined by using Eq. (1), and the values were found to be 59.00% and 87.11% respectively in **Table (2)**. These values are slightly of those obtained from **[12]**. On the other hand, X-ray diffraction (XRD) for raw paper waste and the cellulose nanocrystals which produced from paper waste treated by mechanical and chemical processes were illustrated in **(Fig.1c)**.

The obtaineddata showed that paper waste exhibited three intense peaks were noted at a diffraction angle $(2\theta) = 17.65^{\circ}$ and 24.15° which correspond to (110) and (200) lattice planes of cellulose. A further very small peak, which corresponds to (004) of crystalline cellulose, was seen at $2\theta = 34.96^{\circ}$.

The paper waste diffraction peaks showed the same patterns following acid hydrolysis but at a reduced intensity. This indicates that cellulose makes up the obtained CNCs' crystal structure. However, the raw paper waste's crystallinity index, CNCs-chemical, and CNCs-mechanical methods was determined by using Eq. (1). The values of crystalline index in **Table** (2) for paper waste, CNCs- chemical and CNCs mechanical methods were found to be 47.66%, 90.00%, and 68.29%, respectively. These results are in harmony with those previously reported by [13].

Table (2): The crystallinity index of Sugarcane bagasse (SCB), Sawdust (SD) and Paper waste (PW).

Samples under investigation	2 heta (amorphous)		2θ (002) (o)		Crystallinity index	Size (A)°	D Average (nm)	
mvestigation	Degree	Intensity I _{am}	Degree Intensity I200		(CrI%)	(11)	Trerage (IIII)	
	Sug	arcane baga	asse (SCB)					
Cellulose	15.67	307	23.23	560	45.18%			
CNCs	15.57	200	23.14	890	77.53%	335.801	33.5801	
		Saw dust	(SD)					
Cellulose	19.27	738	24.83	1800	59.00%			
CNCs	19.33	290	24.88	2250	87.11%	368.560	36.8560	
		Paper waste	e (PW)					
Cellulose	18.55	785.1	24.22	1500	47.66%			
CNCs (chemical method)	18.25	230	24.00	2300	90.00%	260.748	26.0748	
CNCs								
(mechanical	19.88	666	24.00	2100	68.29%	256.258	25.6258	
method)								

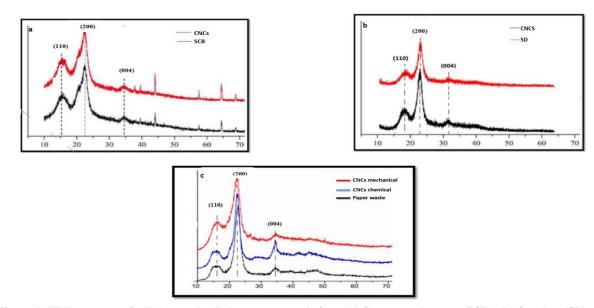


Figure 1: XRD patterns of cellulose and cellulose nanocrystals from (a) Sugarcane bagasse (SCB), (b) Sawdust (SD), and (c) paper waste (PW).

Scanning electron microscopy (SEM):

This process was used to described and change of fiber surface morphology, also determined the particle size of the raw materials, chemically and mechanical purified cellulose and cellulose nanocrystals from the wastes under investigation such as sugarcane bagasse (SCB), wood sawdust (SD), and paper industrial wastes (PW). The all images of (SEM) from these data are presented in Figs 2 (a,b,c,d,e,f and g).

From the obtained results, the original sugarcane bagasse (SCB) fiber had a significantly larger diameter, and each fiber looked to be made up of many micro fibrils. The compact structure of each elementary fiber is aligned along the fiber axis. Additionally, the SCB micrograph shows several non-fibrous elements dispersed across the fiber surface. The fiber's overall surface seems smooth due to the presence of wax and oil. Through complicated synthesis and de-polymerization, the lignin is eliminated after first treatment with sodium chlorite.

The hemicellulose is hydrolyzed and becomes water soluble after further alkali treatment. In contrast, the cellulose nanocrystal made from sugarcane bagasse (Fig. 2a and d) demonstrated a decrease in the size of the fibrillar structure as well as sporadic fragmentation of the fibrillar structure into individual fibrils. The essential characteristics of the nanocellulose micrograph are the occasional breakdown of the fibrillar structure in its axial direction and the refinement of the fibrillar structure linked to further diameter decrease. These outcomes concur with those of [9]. On the other hand, SEM images of SD as a raw material, and CNCs produced from SD were evaluated and the result are illustrated in Fig. 2 (b and e).

The data showed that different magnifications and extremely rigid cellulose crystals appeared as sticks in SEM micrographs. However, the presence of some non-cellulose components on the fiber's surface is responsible for the sawdust sample's smooth surface when it was left untreated. It was clear that lignin and hemicellulose had been eliminated following alkaline treatment and delignification with acidified sodium chlorite. After the raw fiber was chemically treated, the chemically purified cellulose was found to have a reticular and thin fibril structure.

This implies that the cellulose structure was not completely broken down and the amorphous area was not removed by the alkaline treatment and delignification procedures. The rupture of the amorphous domain may have caused the raw samples' surface morphology to shrink in size. However, the characteristics of the (CNCs) in the SD demonstrated that the size of the fibrillar structure had decreased, and the fibrillar structure had broken down intermittently into individual fibrils. The obtained data are close with those found by[12].

Also, the photos of morphology of PW samples were investigated using (SEM) and data are presented in Fig. 2 (c and f, c and g) for purified cellulose from PW and cellulose crystals- chemical and mechanical methods. These images, Fig. 2 (c and f) showed thatthe introduction of sulfuric acid in the CNCs-chemical samples caused the amorphous area to break down, which caused negatively charged sulfate groups to graft onto the CNC's surface [19]. While, Fig. 2 (c and g) it appears that isolations were only partially successful because the surface of the CNCs had both structured and amorphous patches.

Because of surface ionic change brought on by the acid hydrolysis process, the CNCs have a spherical form and little aggregation. By breaking down hydrogen bonds and cell wall structure with various stresses, fibrillation in a grinder is a mechanical process that individualizes pulp into nanoscalefiber[25].

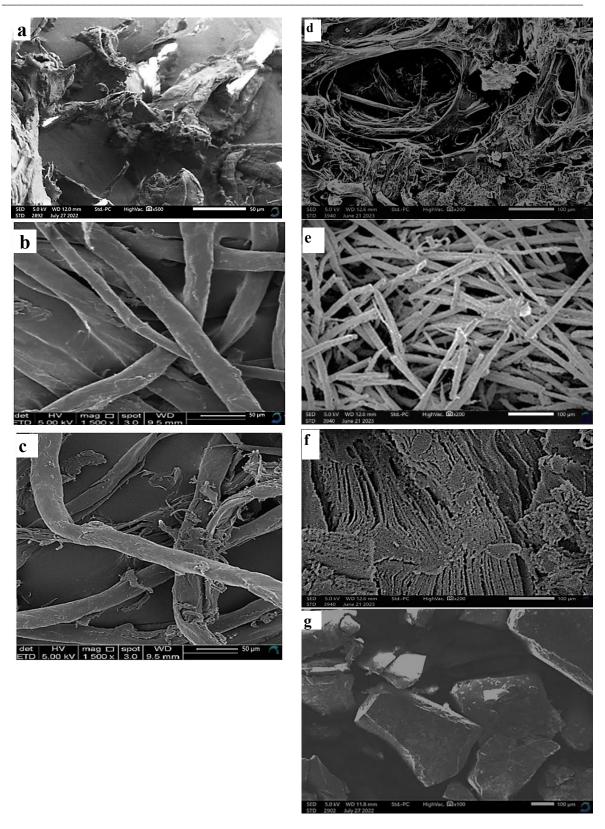


Figure 2: SEM images of cellulose (a) from Sugarcane bagasse (SCB), (b) from Sawdust (SD) (c) from paper waste (PW), (d) cellulose nanocrystals from (SCB), (e)cellulose nanocrystals from (SD) and (f) cellulose nanocrystals – chemical method from (PW), (g) cellulose nanocrystals – mechanical method from (PW).

Fourier transform infrared (FTIR) analysis:

The findings of this examination, which looks at how the chemical structure of cellulose fiber changed before and after chemical and mechanical treatments, it also verified the existence of bonds at 3330,2920,1630, and 896 cm⁻¹ that are indicatinal groups are shown in **Fig. 3** (a, band c). The comparable adsorption bonds that were produced indicate that the compositional integrity of cellulose was not harmed by the acid hydrolysis process utilized to create CNCs from SCB, **Fig. 3a.** The two primary absorbance zones seen in both samples are 800–1800 cm⁻¹ and 2700–3500 cm⁻¹. The bending vibration of the C-H bond in lignin is attributed to the peaks at 832 cm⁻¹. The C-O stretching in aromatic C-H deformation vibrations of cellulose and lignin is linked to the peak at 1034 cm⁻¹. The C=O anti-symmetric stretching vibration of a glucosidic ring in cellulose/hemicellulose is responsible for the peak at 1501 cm⁻¹. The pyranose ring C-O-C symmetric stretching of cellulose and hemicellulose is linked to the peak 1160 cm⁻¹. The results exhibit a high degree of agreement with those that have been previously published[9].

Fourier transform infrared of SD cellulose and cellulose nanocrystals showed in Fig. (3, b). According to these findings, cellulose was the primary component of sawdust; the original sawdust clearly displayed cellulose's distinctive beaks, O-H symmetric, aliphatic C-H stretching, C=O vibration, and C-O-C vibration were identified as the three peaks at wavenumbers of 3350 cm⁻¹, 2753 cm⁻¹, 1100 cm⁻¹, and 1470 cm⁻¹, respectively. Furthermore, maxima were detected at 890 cm⁻¹ for C-H scissoring and 1560 cm⁻¹ for C=O stretching bending. The infrared spectra of the bleached sawdust did not significantly alter. The same cellulose macromolecule beaks were seen in the extracted CNC. However, the OH peak's intensity (3350 cm⁻¹). Additionally, following acid hydrolysis, the strength of a little peak in SD crude fiber at 890 cm⁻¹ has grown. The achieved results are differing from those provided by [12].

On the other hand, the structural change of the PW, CNCs-mechanical, and CNCs-chemical samples were examined before and after treatments and the obtained results are illustrated in **Fig. 3c.** The peak at 3530 cm⁻¹ associated with the O-H bending vibration of absorbed water was evident in the spectra of all samples. On the subject of potential extraction effects on the transmittance bands, C-O-C found smoothed peaks in the range of 1100 to 1500 cm⁻¹, where specific vibrations cannot be assigned signatures. These tiny peaks may possibly originate from the hemicelluloses found in cellulose fiber. Presenting peaks at 630 cm⁻¹ for C-H which indicates, the purity of the crystalline band of cellulose, with characteristics of 1620 cm⁻¹ for C=O stretching vibration and elongation of cellulose typical pulp β -glycoside bonds, respectively, especially in nanocellulose spectra (CNCs-chemical and CNCs mechanical). It is important to note that the extracted nanocellulose shows transmittance signal at 1620, and 630 cm⁻¹ which show that the nanocellulose produced was primarily in the form of cellulose structure. These results are differ from those reported by [19].

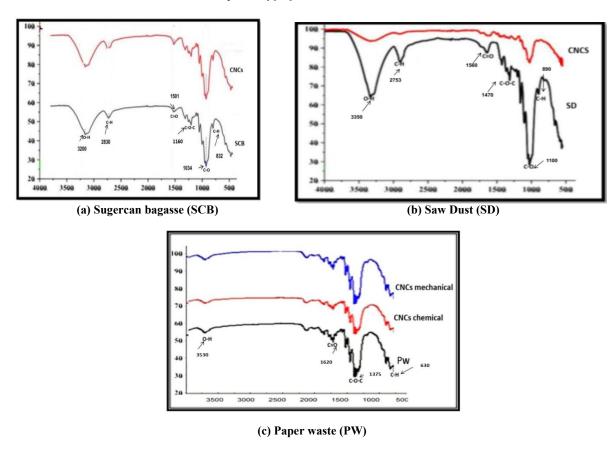


Figure 3: FTIR patterns of cellulose and cellulose nanocrystals from (a) Sugarcane bagasse (SCB), (b) Sawdust (SD), and (c) paper waste (PW).

Egypt. J. Chem. 68, SI: Z. M. Nofal (2025)

Transmission electron microscope (TEM):

A transmission electron microscope was used to study morphology and determine the particle size of the cellulose nanocrystals (CNCs). The acid hydrolysis of (SCB) to synthesis (CNCs), process is aimed to reduce the size of the cellulosic nanomaterials to the nanometer range while leaving the crystalline region, and the TEM analysis of CNCs are shown in Fig. 4 (a,b,c and d). From the data in Fig. 4 athe average diameter ranged between 18 nm to 58 nm, and the average length of the CNCs from sugarcane bagasse ranged between 193 nm to 378 nm, Also, the particles were fibril in shape. CNCs particles accumulate in some places while dispersing in others. These results are differ from those reported by [26].

While, the synthesis of (CNCs) from sawdust was investigated under a transmission microscope at different areas and the obtained results are presented in **Fig. 4b.** The image showed mostly individual nanocrystals and some aggregates. The CNCs dimensional values of diameter and length of the nanorods ranged from 47.41 nm to 55.77 nm, while the length ranged from 277.13 nm to 350.2 nm. These findings were caused by the hydroxyl groups' high density on the cellulose chain molecules surface. The outcomes showing a strong correlation with the previously published by **[12].**

On the other hand, the TEM images for PW are described in **Fig. 4** (c and d). The diameter of the CNCs ranged from 30 nm to 40 nm, and the length ranged from 250 nm to 577 nm. These results are in harmony with those previously reported by [14].

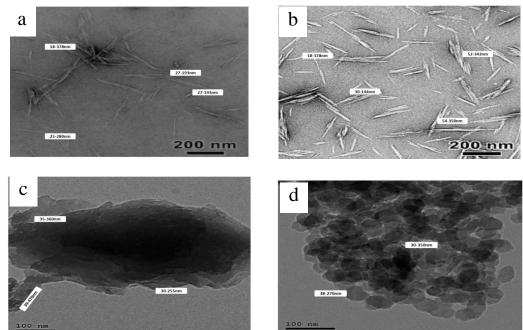


Figure 4: Apparent shape and TEM images of CNCs (a)SCB,(b) SD,(c) PW chemical method,(d) PW mechanical method.

3. Conclusions

The current work was proposed to synthesize worthy materialfrom useless agricultural and industrial wastes such as sugarcane bagasse, sawdust, and paper waste as industrial wastes. In this investigation, CNCs were extracted from cellulose and synthesized cellulose nanocrystals from SCB, SD, and PW. The cellulose that was extracted from the raw materials under implementation had a greater cellulose content and a lower hemicellulose and lignin content than the raw materials, according to the chemical analysis of the data. Along with FTIR and XRD, the morphological examination considered size and size distribution, while TEM showed the average diameter. Furthermore, XRD studies showed that the chemical composition and crystal lattice structure of CNCs and raw materials were the same. More significantly, it makes sense to take use of the functional characteristics of CNCs and their composites to develop novel and targeted applications in tissue engineering, energy, and sensors.

4. Conflicts of interest

The authors declare that they have no conflict of interest.

Ethical approval

Not required

Funding

None

Data availability

Not applicable

Egypt. J. Chem. 68, SI: Z. M. Nofal (2025)

Consent to Participate
Not applicable
Consent to Publish
Not applicable

5. Acknowledgments

The authors would like to thank Agricultural Analysis and Consulting Center for the help in chemical analysis, Prof. Dr. Mahmoud Hassan Mohamed for the help in statistical analysis, and special thanks are also extended to the Department of Biochemistry, Faculty of Agriculture, Benha University.

6. References and Bibliography

- [1]Li, S., and Chen, G. (2020). Agricultural waste-derived superabsorbent hydrogels: Preparation, performance, and socioeconomic impacts. J. of Cleaner Production, 251, 119669. https://doi.org/10.1016/j.jclepro.2019.119669
- [2]Debnath, B.; Haldar, D.; and Purkait, M. K. (2021). A critical review on the techniques used for the synthesis and applications of crystalline cellulose derived from agricultural wastes and forest residues. Carbohydrate polymers, 273, 118537. https://doi.org/10.1016/j.carbpol.2021.118537.
- [3]Rao,L.N.; Lakavat,M.; Gandi,S.; and Sri,G.K.(2023).Extraction and characterization of cellulose from agricultural waste materials. Materials Today: Proceeding, 80, 2740-2743. http://doi.org/10.1016/j.matpr.2021.07.030.
- [4]Bayda, S.; Adeel, M.; Tuccinardi, T.;Cordani, M.; and Rizzolio, F. (2020).The history of nanoscience and nanotechnology: from chemical–physical applications to nanomedicine. Molecules, 25(1), 112. DOI:10.3390/molecules25010112.
- [5]Liu, A.; Wu, H.; Naeem, A.; Du, Q.; Ni, B.; Liu, H.; and Ming, L. (2023). Cellulose nanocrystalline from biomass wastes: An overview of extraction, functionalization and applications in drug delivery. Int. J. of Biol. Macromolecules, 241, 124557. https://doi.org/10.1016/j.ijbiomac.2023.124557
- [6]Michelin, M.; Gomes, D. G.; Romaní, A.; Polizeli, M. D. L. T.; and Teixeira, J. A. (2020). Nanocellulose production: exploring the enzymatic route and residues of pulp and paper industry. Molecules, 25(15), 3411.; Doi:10.3390/molecules25153411
- [7]Zhang, X.; Wang, D.; Liu, S.; and Tang, J. (2022). Bacterial cellulose Nanofbril-based Pickering emulsions: Recent trends and applications in the food industry. In Foods (Vol. 11, issue 24) (Vol. 11).MDPI.https://doi.org/10.3390/foods11244064.
- [8]Kumar, A.; Kumar, V.; and Singh, B. (2021).Cellulosic and hemicellulosic fractions of sugarcane bagasse: Potential, challenges and future perspective. Int. J. of Biol. Macromolecules, 169, 564-582. https://doi.org/10.1016/j.ijbiomac.2020.12.175
- [9]Barakat, A.; Fahmy, A.; El-Moslamy, S. H.;El-Fakharany, E. M.; Kamoun, E. A.; and Bassyouni, M. (2021). Cellulose nanocrystals from sugarcane bagasse and its graft with GMA: Synthesis, characterization, and biocompatibility assessment. J. of Appl. Pharm. Sci, 11(2), 114-125. <u>DOI: 10.7324/JAPS.2021.110215</u>
- [10]Andalia, R.; Rahmi, R.; Julinawati, J.; and Helwati, H.(2020).Isolation and of cellulose from rice husk waste and sawdust with chemical method. J. natural, 20(1), 6-9. DOI: 10.24815/jn.v20i1.12016
- [11]Sultana, S.; Reja, M. M.; Hilary, L. N.; Islam, M. S.; Uddin, M. Z.; and Ajmotgir, W. M. (2021). Preparation and characterization of nanocellulose from *Albizialebbeck* sawdust and their application in nanocomposites using poly (vinyl chloride)(PVC). Bangladesh Council of Sci. and Industrial Res., https://orcid.org/000-0003-3671-8295
- [12]Fagbemi, O. D.; Andrew, J. E.; and Sithole, B. (2021). Beneficiation of wood sawdust into cellulose nanocrystals for application as a bio-binder in the manufacture of particleboard. Biomass Conversion and Biorefinery, 1-12.https://doi.org/10.1007/s13399-021-02015-6
- [13]Kumar, V.; Pathak, P.; and Bhardwaj, N. K. (2020). Waste paper: An underutilized but promising source for nanocellulose mining. *Waste management*, 102, 281-303.https://doi.org/10.1016/j.wasman.2019.10.041
- [14]Egamberdiev, E. A.; and Norboyev, S. K. (2022).Extraction of cellulose nanocrystals from secondary paper waste and their use in paper production. Tech. sci. and innovation, 2022(3), 215-222. : https://btstu.researchcommons.org/journal
- [15]Reddy, K. O.; Maheswari, C. U.; Reddy, D. J. P.; Guduri, B. R.; and Rajulu, A. V. (2010). Properties of ligno-cellulose ficusreligiosa leaf fibers. Int. J. of Polymers and technologies, 1.29-35. http://hdl.handle.net/10204/4639
- [16]Reddy, J. P.; and Rhim, J. W. (2014).Isolation and characterization of cellulose nanocrystals from garlic skin. Materials Letters, 129, 20-23. http://doi.org/10.1016/j.matlet.2014.05.019.
- [17]Plermjai, K.; Boonyarattanakalin, K.; Mekprasart, W.; Pavasupree, S.;Phoohinkong, W.; and Pecharapa, W. (2018). Extraction and characterization of nanocellulose from sugarcane bagasse by ball-milling-assisted acid hydrolysis.In AIP Conference Proceedings (Vol. 2010, No. 1). https://doi.org/10.1063/1.5053181
- [18] Kumar, A.; Negi, Y. S.; Choudhary, V.; and Bhardwaj, N. K. (2014). Characterization of cellulose nanocrystals produced by acid-hydrolysis from sugarcane bagasse as agro-waste. J. Mater. Phys. Chem., 2(1), 1-8. DOI:10.12691/jmpc-2-1-1.
- [19]Souza, A. G. D.; Kano, F. S.; Bonvent, J. J.; and Rosa, D. D. S. (2017). Cellulose nanostructures obtained from waste paper industry: a comparison of acid and mechanical isolation methods. Materials Research, 20, 209-214. https://doi.org/10.1590/1980-5373-MR-2016-0863

- _____
- [20]Park, S.; Baker, J. O.; Himmel, M. E.; Parilla, P. A.; and Johnson, D. K. (2010). Cellulose crystallinity index: measurement techniques and their impact on interpreting cellulase performance. Biotech.forbiofuels, 3, 1-10. <u>Http://www.boitechnology</u>
- [21]Evans, S. K.; Wesley, O. N.; Nathan, O.; and Moloto, M. J. (2019). Chemically purified cellulose and its nanocrystals from sugarcane baggase: isolation and characterization. Heliyon, 5(10). https://doi.org/10.1016/j.heliyon.2019.e02635
- [22]Oyohwose, U. A.; and Omoko, V. I. (2023). Synthesis, characterization and application of nanocrystalline cellulose: A review. Int. J. of Science and Research Archive, 8(2), 234-243.: https://doi.org/10.30574/ijsra.2023.8.2.0217
- [23]Mandal, A.; and Chakrabarty, D. (2011).Isolation of nanocellulose from waste sugarcane bagasse (SCB) and its characterization. Carbohydrate Polymers, 86(3), 1291-1299. http://doi.org/10.106/j.carbpo;.2011.06.030
- [24]Qin, Y.; Chang, R.; Ge, S.; Xiong, L.; and Sun, Q. (2017). Synergistic effect of glycerol and ionic strength on the rheological behavior of cellulose nanocrystals suspension system. Int. j. of boil.macromolecules, 102, 1073-1082. https://doi.org/10.1016/j.ijbiomac.2017.05.007
- [25]Khalil, H. A.; Davoudpour, Y.; Islam, M. N.; Mustapha, A.; Sudesh, K.; Dungani, R.; and Jawaid, M. (2014). Production and modification of nanofibrillated cellulose using various mechanical processes: a review. *Carbohydrate polymers*, 99, 649-665. https://doi.org/10.1016/j.carbpol.2013.08.069.
- [26] Vu, A. N.; Nguyen, L. H.; Yoshimura, K.; Tran, T. D.; and Van Le, H. (2024). Cellulose nanocrystals isolated from sugarcane bagasse using the formic/peroxyformic acid process: Structural, chemical, and thermal properties. Arabian J. of Chem, 105841. https://doi.org/10.1016/j.arabjc.2024.105841