



## EFFECT OF SULFURIC ACID TREATMENT IN CELLULOSE NANOCRYSTALS EXTRACTION FROM *Sargassum* sp. SEAWEED

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### Abstract

*Sargassum* sp. is a type of brown seaweed often found in tropical waters, but it has not been optimally used. The high cellulose content of *Sargassum* sp. can be used to produce cellulose nanocrystals (CNC). CNC can act as a bionanocomposite-reinforced nanomaterial. This study aimed to determine the most effective sulfuric acid concentration for extracting cellulose nanocrystals from *Sargassum* sp. CNC was extracted from *Sargassum* sp. using acid hydrolysis and sonication. The sulfuric acid concentration was varied to 30, 40, 50, and 60%. CNC was characterized using Fourier-transform infrared spectroscopy (FTIR), X-ray diffraction (XRD), and thermogravimetric analysis (TGA). FTIR analysis confirmed the presence of characteristic CNC functional group peaks, including C–O–C (~1160 cm<sup>-1</sup>), C–O (~1050–1030 cm<sup>-1</sup>), and β-(1→4)-glycosidic C–H (~897 cm<sup>-1</sup>) as the CNC fingerprint. The FTIR findings indicated that the CNC extracted by sulfuric acid hydrolysis differed significantly from the raw *Sargassum* sp. material. Additionally, the XRD results showed that acid hydrolysis substantially affected the amorphous regions of cellulose. With 40% acid hydrolysis, the XRD analysis showed the highest CNC degree of 77.6%. Thermal analysis using TGA and DTG revealed that cellulose nanocrystals treated with 40% acid hydrolysis yielded CNC with enhanced thermal stability, exhibiting a maximum thermal decomposition temperature of 369.60°C. CNC isolated from *Sargassum* sp. cellulose has the potential to serve as a suitable source for manufacturing nanocomposites in various applications, such as pharmaceuticals, food packaging, and biomedical fields.

Keywords: brown algae, crystallinity, FTIR, hydrolysis, sonication

### Efek Perlakuan Asam Sulfat pada Ekstraksi Selulosa Nanokristal dari Rumput Laut *Sargassum* sp.

#### Abstrak

*Sargassum* sp. adalah jenis rumput laut coklat yang banyak ditemukan di perairan tropis, namun belum dimanfaatkan secara optimal. Kandungan selulosa yang tinggi dalam *Sargassum* sp. dapat digunakan untuk menghasilkan selulosa nanokristal (CNC). CNC dapat digunakan sebagai bahan nano penguat bionanokomposit. Penelitian ini bertujuan untuk menentukan konsentrasi asam sulfat yang optimum untuk mengekstraksi nanokristal selulosa dari *Sargassum* sp. CNC dari *Sargassum* sp. diekstraksi menggunakan

metode hidrolisis asam dan sonikasi. Variasi konsentrasi asam sulfat pada proses ekstraksi CNC, yaitu 30, 40, 50, dan 60%. Karakterisasi CNC dilakukan menggunakan spektroskopi inframerah (FTIR), difraksi sinar-X (XRD), dan analisis termogravimetri (TGA). Analisis FTIR mengonfirmasi keberadaan puncak gugus fungsi khas CNC, termasuk C–O–C ( $\sim 1160\text{ cm}^{-1}$ ), C–O ( $\sim 1050\text{--}1030\text{ cm}^{-1}$ ), dan C–H  $\beta$ -(1 $\rightarrow$ 4)-glikosidik ( $\sim 897\text{ cm}^{-1}$ ) sebagai sidik jari CNC. Hal ini menunjukkan bahwa CNC yang diekstraksi melalui hidrolisis asam sulfat memiliki perbedaan yang signifikan dibandingkan dengan bahan baku *Sargassum* sp. Selain itu, hasil XRD menunjukkan bahwa hidrolisis asam berpengaruh pada berkurangnya daerah amorf pada CNC. Analisis XRD menunjukkan tingkat CNC tertinggi sebesar 77,6% pada hidrolisis asam sulfat 40%. Analisis termal menggunakan TGA dan DTG menunjukkan bahwa CNC yang diproses dengan hidrolisis asam sulfat 40% menghasilkan CNC dengan stabilitas termal yang meningkat, menunjukkan suhu dekomposisi termal sebesar 369,60 °C. CNC yang diisolasi dari *Sargassum* sp. berpotensi sebagai bahan pembuatan nanokomposit dalam berbagai aplikasi, misalnya bidang farmasi, pengemasan makanan, dan biomedis.

Kata kunci: alga cokelat, FTIR, hidrolisis, kristalinitas, sonikasi

## INTRODUCTION

Indonesia, as an archipelagic country, has a large maritime area with a coastline of 81,290 km (Hikmawati *et al.*, 2023). This extensive coastline provides abundant marine resources, including seaweed. Among the diverse seaweed species, *Sargassum* sp., a type of brown macroalgae (*Phaeophyceae*), is commonly found along the coastal waters of Indonesia. However, its potential remains largely underutilized, and it is often regarded as waste that disrupts fishing activities and maritime navigation (Khansa *et al.*, 2024). *Sargassum* sp. has a rich chemical composition, containing valuable compounds such as alginic acid, fucoidan, fucoxanthin, phenolics (Erniati *et al.*, 2024; Ramlan *et al.*, 2024), and cellulose (Safitri *et al.*, 2021). The cellulose content of *Sargassum* sp. ranges from 23.97% to 35.22% (Taslim *et al.*, 2017), indicating significant potential for value-added applications.

Marine biomass, such as *Sargassum* sp., is a promising raw material for producing cellulose nanocrystals (CNC), which are renewable and biodegradable nanomaterials. CNC exhibit exceptional properties, including high mechanical strength, biocompatibility, high crystallinity, and large surface area, making them highly suitable for a wide array of applications, ranging from reinforcement in polymer composites and biodegradable packaging to drug delivery systems (Doh & Whiteside, 2020; George & Sabapathi, 2015). Although several studies have explored CNCs derived from marine algae biomass, such as *Dictyota bartayreiana* (Murugesan

*et al.*, 2024), *Gelidiella acerosa* (Singh *et al.*, 2017), and *Cladophora glomerata* (Plianwong & Sirirak, 2024), relatively few studies have investigated CNCs from marine sources such as *Sargassum*. Initial studies have indicated that CNC derived from *Sargassum* and other marine algae can enhance the mechanical and thermal properties of biopolymer films (Doh, 2020; El Achaby *et al.*, 2018). However, the extraction processes, particularly the optimization of acid hydrolysis parameters, remain inadequately explored.

CNC extraction typically involves acid hydrolysis to selectively remove the amorphous regions of cellulose, thereby isolating the crystalline domains of the cellulose (Muljani *et al.*, 2023). Mechanical treatment, such as ultrasonication, is often used afterward to reduce the particle size to the nanoscale (Saputri & Sukmawan, 2020). One critical factor affecting CNC yield and quality is the concentration of sulfuric acid used for hydrolysis. Several studies have reported the potential of brown seaweed (Doh *et al.*, 2020; Murugesan *et al.*, 2024) as a source of CNC for hydrolysis using sulfuric acid. However, the chemical composition and characteristics of *Sargassum* are highly dependent on its growing location, making it important to evaluate the potential of this species in Indonesian waters, particularly those around Yogyakarta.

Therefore, this study aimed to determine the most effective sulfuric acid concentration for the hydrolysis of cellulose nanocrystals from *Sargassum* sp. to optimize the yield and enhance the quality of the resulting nanomaterials for potential



applications. The findings are expected to support the sustainable utilization of marine biomass and contribute to the development of high-performance and environmentally friendly nanomaterials.

## MATERIALS AND METHODS

### Preparation of Raw Materials

*Sargassum* sp. was supplied by Gunung Kidul Regency, Yogyakarta, Indonesia. *Sargassum* sp. was washed and dried in the sun for two days. *Dry Sargassum* sp. (moisture content: 10.50%) was ground using a grinder, and stored at room temperature before use in the extraction process (Doh *et al.*, 2020). Commercial CNC was used as a positive control and obtained from CelluForce, Canada.

### Pretreatment

The chemical solutions used were hydrochloric acid (HCl), sodium hydroxide (NaOH), potassium hydroxide (KOH), sodium hypochlorite (NaOCl), glacial acetic acid, hydrogen peroxide ( $H_2O_2$ ), and sulfuric acid ( $H_2SO_4$ ). All chemical solutions used were of technical grade. *Sargassum* sp. (20 g) was dissolved in 200 mL 0.2 M HCl and heated in a *water bath* at 30°C for 2 h. The sample was centrifuged at 15,000  $\times g$  for 10 min. The residue was separated from the supernatant and re-suspended in 300 mL distilled water. The suspension was adjusted to pH 10 using 4% NaOH and heated in a *water bath* at 75°C for 3 h.

The heated sample was rinsed with water until a pH of 7 was reached, and then centrifuged at 15,000  $\times g$  for 10 min. The residue was separated from the supernatant, dried in an oven at 65°C for 20 h, and then added to 350 mL of 10% KOH. The mixture was heated in a *water bath* at 80°C for 3 h. The heated sample was rinsed with tap water until a pH of 7 was reached, and 50 mL of 6.5% NaOCl solution was added. The suspension was stirred and left to stand for 30 min until the color of the sample brightened.

The sample was rinsed with tap water 3-4 times, and 300 mL of distilled water was added. Glacial acetic acid was added to adjust the pH to 5, and the sample was heated in a

*water bath* at 75°C for 2 h. The heated sample was rinsed with distilled water until a pH of 7 was reached, and 20 mL of 10%  $H_2O_2$  solution was added. The suspension was heated in a *water bath* at 80°C for 70 minutes, followed by 3-4 flushes with distilled water (Doh *et al.*, 2020).

### Acid Hydrolysis

The wet pre-treated sample was added with 100 mL of  $H_2SO_4$  (30, 40, 50, and 60%), and then heated in a *water bath* at 45°C for 30 min. The heated sample was added to 200 mL of cold water and centrifuged at 15,000  $\times g$  for 10 min. The pellets were rinsed with tap water until pH 7 was reached, and then centrifuged at 15,000  $\times g$  for 10 min (centrifugation was carried out twice).

The acid-hydrolyzed samples were added to distilled water and then toned using an ultrasonic device (UCD 250, Biomaisen, China) at a frequency of 20 KHz and an amplitude of 30% of the total power of 500 W for 15 min. CNC was freeze dried using a freeze dryer, the liquid sample was frozen at -80°C for 24 h and then subjected to high vacuum (0.05 mbar) for another 24 h.

### Characterization

#### Yields

Yield is the ratio of the final weight of a product to the initial weight of a dry sample (Septiani *et al.*, 2017). The final weight of the sample was obtained by weighing the freeze-dried CNC, and the initial weight of the sample was obtained by weighing the *Sargassum* sp. powder.

### Fourier transform infrared spectroscopy (FTIR)

The FTIR spectrum was obtained using ATR-FTIR (Nicolet iS 5 ATR iD5, Thermo Scientific, USA) in the range of 500–4000  $cm^{-1}$ . Measurements were made at a scan resolution of 4  $cm^{-1}$  (Nurhayati *et al.*, 2025).

### X-ray diffraction (XRD)

The CNC crystallinity index was determined using XRD with Cu K $\alpha$  radiation ( $\lambda = 1.54060 \text{ \AA}$ ) in the range of  $2\theta$  4° to 60°. The operating conditions were set at 40

kV and 25 mA. The crystallinity index was calculated using the Segal method, following the defined equation. The CNC crystallinity index was determined using XRD, using Cu K $\alpha$  radiation ( $\lambda = 0.15406$  nm) in the range of  $2\theta$   $4^\circ$  to  $60^\circ$  with operating conditions set at 40 kV and 25 mA. The crystallinity index is calculated using the Segal method, following the specified equation (Doh *et al.*, 2020).

$$\text{Crystallinity index (\%)} = \frac{I_{200} - I_{am}}{I_{200}} \times 100$$

Note:

$I_{200}$  = maximum intensity on the field (200) ( $2\theta = 22,4^\circ$ )

$I_{am}$  = minimum intensity in the valley between the fields (200) dan (110) ( $2\theta = 18,1^\circ$ )

### Thermogravimetric analysis (TGA)

The analysis was carried out under a nitrogen atmosphere (20 mL/min) with a heating rate of  $10^\circ\text{C}/\text{min}$ , covering a temperature range from  $25^\circ\text{C}$  to  $500^\circ\text{C}$  (Costa *et al.*, 2015).

### Crystallite size

The crystallite size of the cellulose nanocrystals (CNCs) was determined using X-ray diffraction (XRD) analysis. The average crystallite size (D) was calculated using the Scherrer equation (Alam *et al.*, 2024), based on the Full Width at Half Maximum (FWHM) of the most intense diffraction peak. The FWHM values were obtained by fitting the XRD peaks using Gaussian peak fitting in the Origin Learning for Student software.

$$D = \frac{K\lambda}{\beta \cos \theta}$$

Note:

D = average crystallite size (nm),

K = the shape factor (commonly taken as 0.9),

$\lambda$  = the wavelength of the X-ray (0.15406 nm),

$\beta$  = FWHM of the diffraction peak in radians,

$\theta$  = the Bragg angle corresponding to the peak.

### Data Analysis

The FTIR, XRD, and TGA analyses were processed and analyzed using Excel and Origin software. Yield data were collected in triplicate and analyzed using one-way ANOVA at a significance level of  $p < 0.05$ .

## RESULT AND DISCUSSION

### CNC Yield

Yield is defined as the ratio of the final weight of a product to the initial weight of the sample (Septiani *et al.*, 2017). The CNC of freeze-dried *Sargassum* sp. is shown in Figure 1. Based on Figure 1, it can be observed that the CNC extracted from *Sargassum* sp. is solid, white, odorless, and tasteless. These findings are in line with the statement that high-quality cellulose is usually powdered, white, and odorless (Pine *et al.*, 2021). While the CNC from *Sargassum* sp. does not show a powder shape but rather a dry solid form, which can be attributed to the freeze-drying process, causing the water to evaporate and the sample to become solid (Agustin & Wibowo, 2021). The white color of the CNC derived from *Sargassum* sp. is associated with the whitening process (Harpendi *et al.*, 2014).

The CNC yield for each treatment was calculated to determine the optimal  $\text{H}_2\text{SO}_4$  concentration for producing the highest amount of CNC from *Sargassum* sp. Yield is an important indicator of extraction efficiency (Fransiska *et al.*, 2020). A graph depicting the CNC results for *Sargassum* sp. is presented in Figure 2.

Based on Figure 2, the CNC yield of *Sargassum* sp. ranged from  $3.79 \pm 2.20\%$  to  $7.15 \pm 1.42\%$ . The highest yield was achieved using 40%  $\text{H}_2\text{SO}_4$ , whereas the lowest yield was observed using 60%  $\text{H}_2\text{SO}_4$ . This yield is relatively low compared to that of CNCs derived from other brown, red, and green seaweeds. Doh *et al.* (2020) reported CNC yields of  $25.8 \pm 0.9\%$  and  $26.1 \pm 1.2\%$  from *Sargassum fluitans* and *Laminaria japonica* (kombu), respectively. Similarly, Murugesan *et al.* (2024) found that *Dictyota bartayresiana*, a tropical brown seaweed species, yielded approximately 10% CNC under optimized acid hydrolysis conditions.



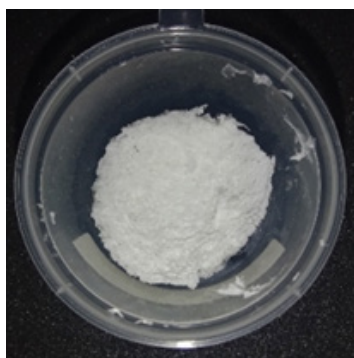


Figure 1 Freeze dried cellulose nano crystal from *Sargassum* sp.

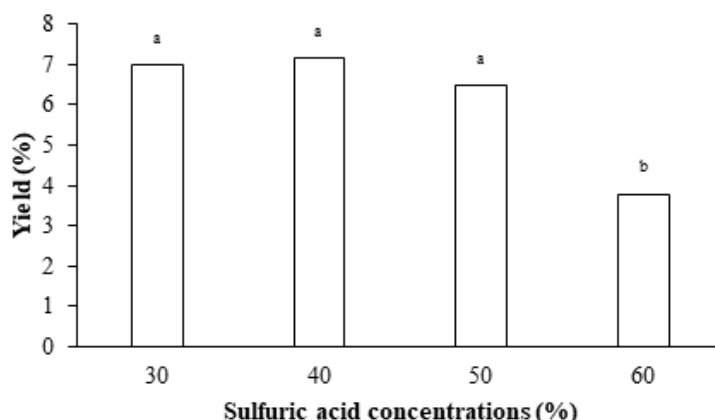


Figure 2 CNC yield from *Sargassum* sp. seaweed with varying  $\text{H}_2\text{SO}_4$  concentrations

Previous research by Harahap *et al.* (2019) found a similar trend, with CNC corn cobs extracted using 45%  $\text{H}_2\text{SO}_4$  yielding 16.98%, lower than the 36.48% yield obtained with 55%  $\text{H}_2\text{SO}_4$ . This can occur because low-concentration  $\text{H}_2\text{SO}_4$  is less effective in breaking down all the amorphous parts of cellulose, causing many cellulose crystals to remain attached to the cellulose chain, resulting in a lower number of cellulose crystals.

Sulfuric acid ( $\text{H}_2\text{SO}_4$ ) at a concentration of 30-50% is effective in removing amorphous cellulose, thus enabling the isolation of high amounts of crystalline cellulose. Higher yield values correlate with greater efficacy in producing large quantities of the desired product (Moranda *et al.*, 2018).

The extraction of *Sargassum* sp. using less than 60%  $\text{H}_2\text{SO}_4$  can be attributed to an increase in the hydrogen bonds between the hydroxyl cellulose (OH) groups and water molecules. Hydrolysis using  $\text{H}_2\text{SO}_4$  can split the hydrogen bonds in cellulose, resulting in

a free OH group formation. The OH group in cellulose can bind to water molecules (Gian *et al.*, 2017). The binding of water molecules by OH groups increases the weight of the CNC, thereby increasing the percentage of results (Simarmata *et al.*, 2024).

The CNC yields extracted using 60%  $\text{H}_2\text{SO}_4$  decreased because of the removal of the amorphous regions in the cellulose chain by  $\text{H}_2\text{SO}_4$ , leaving only crystalline regions (Effendi *et al.*, 2015). Sunardi *et al.* (2019) also reported a decrease in microcrystalline cellulose (MCC) yield with an increase in hydrochloric acid (HCl) concentration.

### Fourier Transform Infrared Spectroscopy (FTIR) of CNC

FTIR spectroscopy was used to identify the functional groups present in the compounds. Each functional cluster has a unique infrared absorption frequency. Figure 3 presents the FTIR spectra of CNCs obtained at different hydrolysis concentrations are presented in Figure 3.

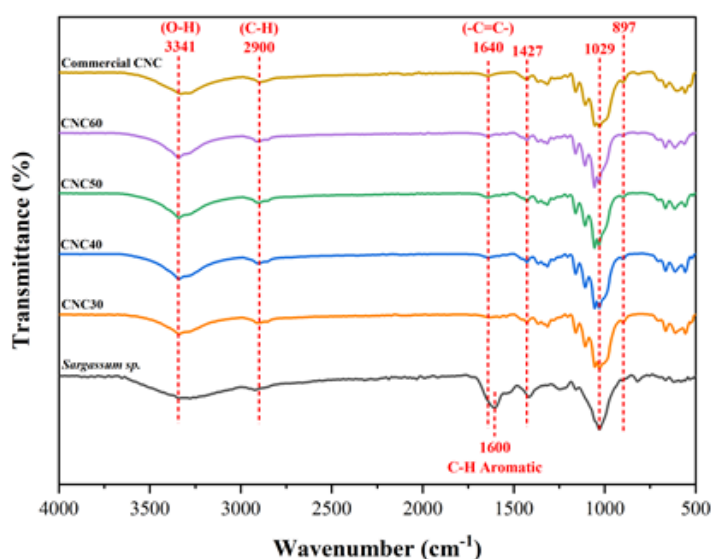


Figure 3 FTIR spectra of CNC commercial and CNC extracted from *Sargassum* sp. under different sulfuric acid hydrolysis concentration

The peak observed at  $3341\text{ cm}^{-1}$  may be attributed to the O-H stretching of the hydroxyl group in cellulose. This aligns with the findings that OH stretching vibrations typically appear within the  $3500\text{--}3200\text{ cm}^{-1}$  range (Theivandran *et al.*, 2015). Additionally, the peaks around  $2900\text{ cm}^{-1}$  are attributed to the stretching vibrations of the CH groups (alkanes). This observation aligns with the fact that peaks in the  $2900\text{--}2800\text{ cm}^{-1}$  range are characteristic of CH stretching, particularly from  $\text{CH}_3$  and  $\text{CH}_2$  groups (Topală *et al.*, 2017).

The peak at  $1640\text{ cm}^{-1}$  shows a cluster vibration of  $\text{-C=C-}$ , which is in accordance with the statement by Theivandran *et al.* (2015) that peaks in the range of  $1680\text{--}1640\text{ cm}^{-1}$  are associated with the stretching vibration of the  $\text{-C=C-}$  group in alkenes. The  $\text{C=C}$  group is a structural component of the aromatic ring in lignin (Pambudi *et al.*, 2017). The peak at  $1600\text{ cm}^{-1}$  in the *Sargassum* sp. spectrum indicates the absorption of infrared radiation by the lignin aromatic CH group. This is in accordance with the statement of Natsir *et al.* (2022) that the vibration of the stretching of the CH group aromatic lignin occurs at approximately  $1600\text{ cm}^{-1}$ .

The peaks at  $1427\text{ cm}^{-1}$  and  $1415\text{ cm}^{-1}$  correspond to the aromatic CC group vibrations. This is in accordance with the

statement by Theivandran *et al.* (2015), who reported that the vibration of the CC group stretch in the aromatic ring structure occurs at  $1500\text{--}1400\text{ cm}^{-1}$ . The peaks at  $1366\text{ cm}^{-1}$  and  $1314\text{ cm}^{-1}$  indicate CH cluster vibrations. Pangau *et al.* (2017) stated that peaks at  $1369\text{ cm}^{-1}$  and  $1327\text{ cm}^{-1}$  indicate CH group vibration of cellulose.

Peaks at  $1159$ ,  $1108$ ,  $1059$ , and  $1029\text{ cm}^{-1}$  show the stretching vibrations of the cellulose C-O-C group. This is in accordance with the statement of Bashar *et al.* (2019), who indicated that the peaks at  $1160$ ,  $1110$ ,  $1060$ , and  $1028\text{ cm}^{-1}$  indicate the vibration of the pyranos ring skeleton and glucose. Peaks at  $897\text{ cm}^{-1}$  indicate CH group vibrations associated with  $\beta$ -glycosidic bonds, which is consistent with the statement by Abu-Thabit *et al.* (2020) that absorption at a peak of  $895\text{ cm}^{-1}$  is associated with CH vibrations (specific anomeric vibrations of  $\beta$ -glycosidic bonds).

Lower transmission values correspond to higher absorption of infrared radiation by the functional groups in the compounds (Raturandang *et al.*, 2022). The transmission values of the functional groups are presented in Table 1.

The transmission values of CNC30, CNC40, CNC50, and CNC60 were lower than those of *Sargassum* sp. at  $3341$ ,  $2900$ ,  $1366$ ,  $1314$ ,  $1159$ ,  $1108$ ,  $1056$ ,  $1029$ , and  $897\text{ cm}^{-1}$ .

Table 1 Transmittance of CNC commercial and CNC extracted from *Sargassum* sp. under different sulfuric acid hydrolysis conditions

Wavenumber (cm <sup>-1</sup> )	Transmittance (T)					Functional group	Ref.
	CNC30	CNC40	CNC50	CNC60	Commercial CNC	<i>Sargassum</i> sp.	
3341	0.90	0.79	0.81	0.80	0.92	0.96	(Theivandran <i>et al.</i> , 2015)
2900	0.96	0.92	0.93	0.93	0.96	0.98	(Topalà <i>et al.</i> , 2017)
1640	0.99	0.97	0.96	0.97	0.99	0.95	(Theivandran <i>et al.</i> , 2015)
1600	0.98	0.98	0.98	0.98	0.99	0.94	(Natsir <i>et al.</i> , 2022)
1427	0.96	0.93	0.94	0.94	0.97	0.96	(Theivandran <i>et al.</i> , 2015)
1415	0.97	0.95	0.96	0.96	0.98	0.96	(Theivandran <i>et al.</i> , 2015)
1366	0.95	0.92	0.93	0.93	0.96	0.98	(Umpuch, 2015)
1314	0.94	0.89	0.91	0.92	0.95	0.98	(Pangau <i>et al.</i> , 2017)
1203	0.99	0.97	0.97	0.96	0.98	0.98	(Kusumaningsih <i>et al.</i> , 2022) (finger print)
1159	0.91	0.83	0.84	0.84	0.92	0.98	(Bashar <i>et al.</i> , 2019) (finger print)
1108	0.84	0.71	0.74	0.74	0.87	0.96	(Bashar <i>et al.</i> , 2019) (finger print)
1056	0.74	0.54	0.56	0.57	0.79	0.92	(Bashar <i>et al.</i> , 2019) (finger print)
1029	0.73	0.53	0.59	0.61	0.79	0.90	(Bashar <i>et al.</i> , 2019) (finger print)
897	0.96	0.96	0.97	0.98	0.96	0.99	(Abu-Thabit <i>et al.</i> , 2020) (finger print)

This indicates a higher cellulose content in the 30%, 40%, 50%, and 60% samples. The high cellulose content indicates that the non-cellulose components (lignin, hemicellulose, alginate, etc.) were successfully reduced during the pretreatment stage, and cellulose was successfully extracted from *Sargassum* sp. using the acid hydrolysis method.

The O–H stretching band at  $3341\text{ cm}^{-1}$  showed reduced transmittance for CNC40, indicating stronger hydrogen bond cleavage during acid hydrolysis. The C–O–C ( $1159\text{ cm}^{-1}$ ) and C–O ( $1203\text{ cm}^{-1}$ ) bands also displayed noticeable fluctuations, suggesting the partial degradation of glycosidic linkages, particularly at moderate acid concentrations. In contrast, the C–H ( $2900\text{ cm}^{-1}$ ) and aromatic C–C bands ( $1427$  and  $1415\text{ cm}^{-1}$ ) remained relatively stable, indicating resistance to acid-induced structural damage.

So *et al.* (2021) also reported similar results in FTIR analysis of cellulose extracted from *Gelidium amansii* using CNC extraction, where cellulose functional groups were found

at wavenumbers  $2892$ – $2930$ ,  $1369$ ,  $1315$ ,  $1160$ , and  $1054\text{ cm}^{-1}$ . The discovery of this wavenumber indicates the successful isolation of CNC from *Gelidium amansii*.

CNC30, CNC40, CNC50, and CNC60 showed lower transmission values than *Sargassum* sp. at wave numbers  $1427$ ,  $1415$ , and  $1203\text{ cm}^{-1}$ , which correspond to the aromatic CC and C–O functional groups that make up the lignin structure. Lower transmission values at this number of waves indicate a higher content of the aromatic functional groups CC and C in the sample. This phenomenon can occur due to changes in the structure of lignin that remain during sulfuric acid treatment, where the reactive aromatic ring of lignin is condensed to form a C–C bond, making lignin denser and less soluble in acid (Naufala & Pandebsie, 2015)

### Cristallinity of CNC

XRD analysis was performed to determine the CNC crystallinity index. Peaks with maximum intensity are associated

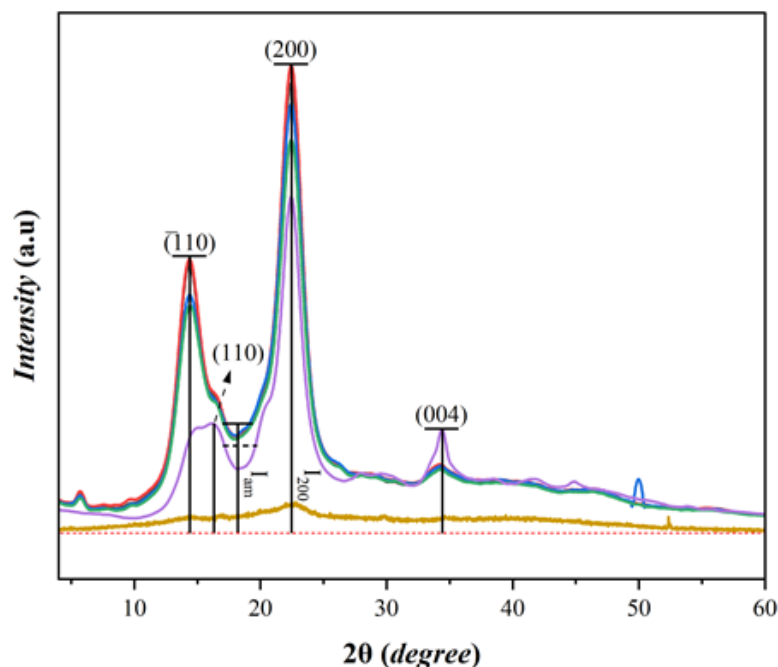


Figure 4 Diffractogram of CNC commercial and CNC extracted from *Sargassum* sp. under different sulfuric acid hydrolysis conditions.

Note: (—)CNC30, (—)CNC40, (—)CNC50, (—)CNC60 are cellulose nanocrystals obtained by 30%, 40%, 50%; 60% sulfuric acid hydrolysis; (—) commercial CNC and (—) *S. polycystum*





with crystalline peaks, whereas those with minimum intensity are associated with the presence of amorphous regions (Suciati *et al.*, 2022).

Based on Figure 4, *Sargassum* sp. has a wider peak in X-ray diffraction than the other samples. This is due to the high lignin and hemicellulose contents of the sample (Doh *et al.*, 2020; Holilah *et al.*, 2021; Xu & Wang, 2015).

Commercial CNC exhibited amorphous regions between the peaks at  $22.4^\circ$  (200) and  $16^\circ$  (110), whereas all CNC samples from *Sargassum* sp. indicated the presence of amorphous regions between the peaks at  $22.4^\circ$  (200) and  $14^\circ$  ( $\bar{1}\bar{1}0$ ). The amorphous region of all samples was observed at  $2\theta = 18^\circ$ . The sample crystallinity index was calculated using the Segal method, and the results are presented in Table 2.

Based on Table 2, the highest crystallinity index was obtained for the commercial CNC at 79.1%. However, the peak intensity of the commercial CNC diffraction at  $2\theta = 22.4^\circ$  was lower than that of CNC30, CNC40, CNC50, and CNC60. This phenomenon can occur because of the smaller size of commercial CNCs, resulting in a wider X-ray diffraction peak with a lower peak intensity. This is in line with the statement by Sehe *et al.* (2017), who stated that smaller crystals produce a wider peak in X-ray diffraction, while larger crystals with a single orientation result in a peak of X-ray diffraction closer to the vertical line.

Commercial CNC produced lower peaks at  $2\theta = 22.4^\circ$ , but the peaks were

still relatively sharp with lower amorphous intensities compared to CNC30, CNC40, CNC50, and CNC60. This indicates the high content of cellulose crystals in commercial CNC. This is in accordance with Septriani & Muldarisnur (2022), who stated that low noise (amorphous) and sharp crystal peaks are indicators of the high crystallinity of nanoparticles.

CNC from *Sargassum* sp. showed a fairly high crystallinity index, and the highest crystallinity index was obtained after 40%  $\text{H}_2\text{SO}_4$  treatment. A high crystallinity index indicates the success of the extraction process in removing the amorphous part of the cellulose chain (El Achaby *et al.*, 2018). CNC was extracted from *Sargassum* sp. using 30%  $\text{H}_2\text{SO}_4$  exhibited a crystallinity index lower than 40%. These results are in line with the research of Rahmi *et al.* (2023), who showed that the crystallinity index of cellulose from *I. cylindrica* treated with 20% and 30%  $\text{H}_2\text{SO}_4$  was lower than that of cellulose treated with 40%  $\text{H}_2\text{SO}_4$ . A lower crystallinity index occurs because the low concentration of  $\text{H}_2\text{SO}_4$  ( $\leq 30\%$ ) could not be able to degrade the amorphous part of the cellulose crystalline.

The crystallinity index that decreased in the treatment with 50%  $\text{H}_2\text{SO}_4$  and 60%  $\text{H}_2\text{SO}_4$  indicates the degradation of crystalline cellulose due to excessively high  $\text{H}_2\text{SO}_4$  concentrations. Rahmi *et al.* (2023) also showed that the cellulose crystallinity index of *Imperata cylindrica* decreased by 50% after  $\text{H}_2\text{SO}_4$  treatment. This decrease in the crystallinity index occurs because high

Table 2 Crystallinity index of CNC commercial and CNC extracted from *Sargassum* sp. under different sulfuric acid hydrolysis conditions

Sample	2 $\theta$ (200) ( $^\circ$ )		2 $\theta$ (amorphous) ( $^\circ$ )		Crystallinity index (%)
	Degree	$I_{200}$	Degree	$I_{am}$	
CNC30	22.43	12.163	18.12	2.789	77.0
CNC40	22.43	12.638	18.10	2.826	77.6
CNC50	22.43	11.686	18.12	2.868	75.4
CNC60	22.45	10.592	18.14	2.753	74.0
Commercial CNC	22.45	9.192	18.28	1.919	79.1

Note: CNC 30, 40, 50, and 60 are cellulose nanocrystals obtained by 30%, 40%, 50% dan 60% sulfuric acid hydrolysis

concentrations of  $\text{H}_2\text{SO}_4$  ( $\geq 50\%$ ) can degrade cellulose crystalline and cellulose interchain hydrogen bonds, resulting in low cellulose crystallinity.

The crystallinity of the samples indicates that they are suitable for nanomaterials, as they are in the range of 54-88%. This is in accordance with the statement of Gorbunova *et al.* (2023), who stated that the range of CNC crystallinity index as a nanomaterial amplifier for biocomposites is 54-88%.  $\text{H}_2\text{SO}_4$  40% was the most effective concentration for extracting CNC from *Sargassum* sp. because it produced the highest crystallinity index. Nanocellulose with a high crystallinity index has a more regular and dense crystal arrangement, resulting in a strong and rigid structure. This structure allows the use of CNC as a reinforcing nanomaterial (Fadly *et al.* 2019).

### Thermal Analysis of CNC

TGA was performed to determine the thermal stability of the sample by measuring the changes in the mass of the sample over time and temperature (Apriyanti *et al.*, 2024). An increase in temperature triggers physical and chemical changes in the sample, resulting in mass changes over time (Hu *et al.*, 2014). The thermogravimetric analysis of commercial CNC and CNC extracted from *Sargassum* sp. under different acid hydrolysis conditions is shown in Figure 5.

Based on Figure 5, two stages of decomposition were found in all the analyzed samples, where the first stage of decomposition occurred at temperatures below  $100^\circ\text{C}$ , while the second stage of decomposition occurred in the temperature range of  $250\text{-}390^\circ\text{C}$ . The decomposition of samples below  $100^\circ\text{C}$  indicates the evaporation of absorbed water and low-molecular-weight compounds adsorbed on the surface of the material (Nurazzi *et al.*, 2021). Cellulose decomposes at  $300^\circ\text{C}$ , whereas lignin decomposes at  $400^\circ\text{C}$  (Henrique *et al.*, 2015). Hemicellulose decomposes at  $220^\circ\text{C}$  (Escalante *et al.*, 2022). The details of the initial decomposition temperature (Tonset), maximum decomposition temperature (Tmax), and residue CNC commercial and CNC extracted from *Sargassum* sp. under

different acid hydrolysis conditions are presented in Table 3.

Based on Table 3, *Sargassum* sp. had the lowest initial decomposition temperature (Tonset) among the other samples, at approximately  $254^\circ\text{C}$ . This is due to the presence of hemicelluloses. Hemicellulose decomposes at  $220^\circ\text{C}$  (Escalante *et al.*, 2022). The acetyl group in hemicellulose can weaken the intermolecular bonds, making hemicellulose decompose more easily at lower temperatures, which contributes to the weight loss of the sample (Hu *et al.*, 2014). Commercial CNC, CNC30, CNC40, CNC50, and CNC60 had a higher Tonset than *Sargassum* sp., at approximately  $280\text{-}300^\circ\text{C}$ . This indicates the occurrence of a cellulose decomposition process consisting of several simultaneous processes, such as dehydration, depolymerization, and decomposition of glycosidic units (Henrique *et al.*, 2015).

CNC40, CNC50, and CNC60 had lower Tonset values than CNC30. This is related to the concentration of sulfuric acid used during hydrolysis. Higher acid concentrations cause cellulose to be hydrolyzed into smaller sizes (Muljani *et al.*, 2023). Smaller particle sizes have a high surface area and lower heat resistance (Marwanto *et al.*, 2021).

*Sargassum* sp. has two peaks of maximum decomposition temperature (Tmax) at  $270^\circ\text{C}$  and  $336^\circ\text{C}$ , which indicate the decomposition of hemicellulose, crystalline cellulose, and cellulose pyrolysis. Commercial CNC, CNC30, CNC40, CNC50, and CNC60 exhibited a single Tmax (sharp peak) at  $319\text{-}369^\circ\text{C}$ , indicating the acquisition of crystalline cellulose with high purity. This is supported by Bashar *et al.* (2019) research on thermal analysis of burlap fibers which showed three peaks of maximum temperature at  $285$ ,  $342$ , and  $451^\circ\text{C}$ , which correlated with the decomposition of hemicellulose, crystalline cellulose, cellulose pyrolysis, and lignin, while the CNC sample showed only one peak at a maximum decomposition temperature of  $305\text{-}318^\circ\text{C}$ , proving the acquisition of crystalline cellulose with high purity.

Commercial CNC exhibited sharp DTG peaks at a maximum decomposition

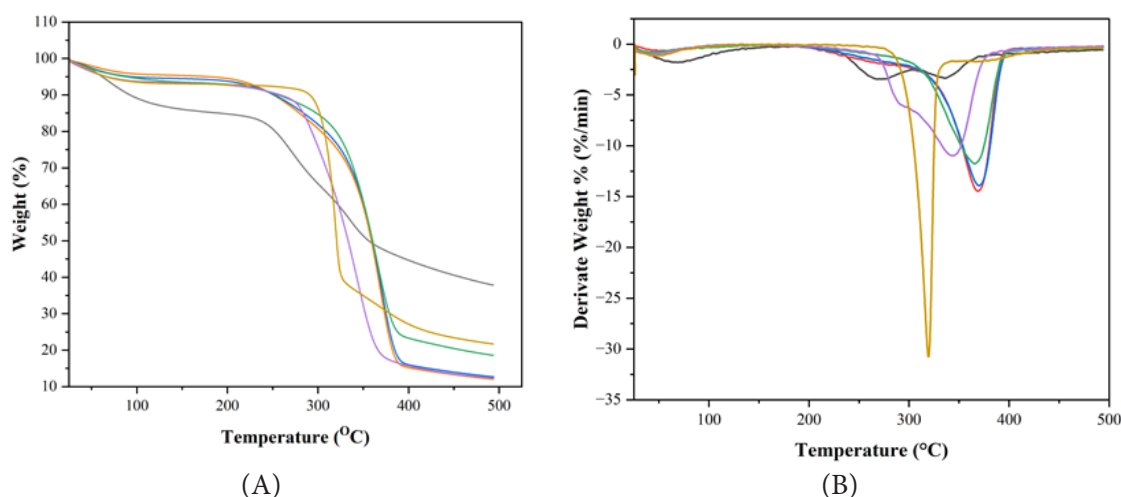


Figure 5 Thermogravimetric analysis of CNC commercial and CNC extracted from *Sargassum* sp. under different sulfuric acid hydrolysis conditions: (A) Thermogravimetric (TG) % weight; (B) Derivative thermogravimetric (DTG). Note: (—) *S. polycystum*, (—) CNC30, (—) CNC40, (—) CNC50, (—) CNC60 are cellulose nanocrystals obtained by 30%, 40%, 50%; 60% sulfuric acid hydrolysis; and (—) commercial CNC

Table 3 Tonset, Tmax, and percentage of residue of CNC commercial and CNC extracted from *Sargassum* sp. under different sulfuric acid hydrolysis conditions

Sample	Tonset (°C)	Tmax (°C)	Residue (%)
<i>Sargassum</i> sp.	254.18	270.23 336.99	39.43
CNC30	336.30	367.85	15.95
CNC40	332.76	369.60	15.86
CNC50	332.87	366.45	23.19
CNC60	283.28	343.30	16.67
Commercial CNC	307.00	319.16	37.00

Note: CNC 30, 40, 50, and 60 are cellulose nanocrystals obtained by 30%, 40%, 50% dan 60% sulfuric acid hydrolysis

temperature of approximately 319°C, which was lower than those of the *Sargassum* sp. samples, CNC30, CNC40, CNC50, and CNC60. El Achaby *et al.* (2018) also showed that the maximum decomposition temperature of CNC was lower than that of *Gelidium sesquipedale*. This may be due to the presence of sulfate groups on the CNC surface, which can reduce the thermal stability of the CNC. Bashar *et al.* (2019) stated that the smaller particle size and higher specific surface area of CNC, as well as the presence of more active surface groups, led to lower heat resistance.

The CNC30, CNC50, and CNC60 samples exhibited Tmax values lower than that of CNC40. This can occur due to the lower crystallinity index and the possibility of replacing the hydroxyl group with a sulfuric acid group (O-SO<sub>3</sub>H) during the acid hydrolysis process. The replacement of these groups lowers the activation energy for CNC decomposition, making the sample less resistant to the pyrolysis. This results in a dehydration reaction that accelerates decomposition at lower temperatures (Costa *et al.*, 2015).

The CNC40 sample had the highest maximum decomposition temperature compared to the others because it had a higher crystallinity index. A higher crystallinity index indicates greater heat resistance and increases the maximum decomposition temperature (Kazharska *et al.*, 2019). A 40% sulfuric acid concentration is likely the most effective for extracting CNC from *Sargassum* sp. because it has a higher maximum decomposition temperature than other concentrations.

The highest residue percentage was obtained for *S. polycystum*, which was approximately 39%. Singh *et al.* (2017) also showed that *Gelidiella acerosa* has a higher residue than CNC, which is 30%.

### Cristallin Size of CNC

X-ray diffraction (XRD) analysis revealed a clear trend in the Full Width at Half Maximum (FWHM) and crystallite size of the CNC samples hydrolyzed using different concentrations of sulfuric acid (30%, 40%, 50%, and 60%). The CNC 30% sample exhibited the narrowest FWHM value (8.57°), corresponding to the highest crystallite size among the synthesized samples (approximately 0.94 nm). In contrast, the CNC 40%, 50%, and 60% samples showed broader diffraction peaks with FWHM values exceeding 8.85° and slightly reduced crystallite sizes in the range of 0.90–0.91 nm. This trend suggests that higher concentrations of sulfuric acid induce more extensive hydrolysis, leading to the fragmentation of the crystalline regions of cellulose.

As a result, the crystallites formed were smaller and more structurally disordered, consistent with previous studies indicating that intense acid hydrolysis increases the amorphous fraction of cellulose nanocrystals (Beck-Candanedo *et al.*, 2005; Gray, 2013).

In contrast, the commercial CNC sample exhibited a remarkably narrower FWHM of 1.22° and a significantly larger crystallite size of 6.61 nm than the synthesized CNCs. This indicates a higher degree of crystallinity, likely due to differences in raw material purity, process control, and post-treatment techniques used in industrial production. Commercially produced CNCs are typically derived from highly purified cellulose sources and may undergo optimized hydrolysis and stabilization procedures, which help preserve and promote the development of larger crystalline domains. This high crystallinity enhances the mechanical strength, thermal stability, and optical clarity of CNC, making them ideal for advanced applications such as nanocomposites, coatings, and biomedical materials (Habibi *et al.*, 2010; Moon *et al.*, 2011; Wang *et al.*, 2007).

The crystallinity index achieved by CNC from *Sargassum* sp. (up to 77.6%) and its high thermal stability (Tmax up to 369.60°C) demonstrate characteristics that are in line with the criteria for such applications (Habibi *et al.*, 2010; Moon *et al.*, 2011). Although the crystallite size was relatively small (0.90–0.94 nm), it still fell within the acceptable range used in nanocomposite material formulations. However, further characterization, including

Table 4 Crystalline size of CNC commercial and CNC extracted from *Sargassum* sp. under different sulfuric acid hydrolysis conditions

Sample	FWHM (°)	Crystalline size(nm)
<i>Sargassum</i> sp.	-	-
CNC30	8.5731	0.94
CNC40	8.9609	0.90
CNC50	8.8589	0.91
CNC60	8.9813	0.90
Commercial CNC	1.2247	6.61

Note: CNC 30, 40, 50, and 60 are cellulose nanocrystals obtained by 30%, 40%, 50% dan 60% sulfuric acid hydrolysis



morphological analysis, surface charge, and dispersion stability, is required to confirm its suitability for specific applications.

## CONCLUSION

FTIR analysis confirmed the reduction of lignin and hemicellulose in the CNC of *Sargassum* sp. after delignification and bleaching treatment. XRD analysis showed that CNC had higher crystallinity than the raw materials, while TGA showed better thermal stability. Hydrolysis with 30% sulfuric acid was effective in producing CNC with a high crystallinity index (77.6%) and significant thermal stability ( $T_{max} = 369.60^{\circ}\text{C}$ ). Treatment with varying sulfuric acid concentrations had no significant effect on the crystallite size of the CNC. The crystallite size remained within the narrow range of 0.90–0.94 nm. These findings highlight the potential of *Sargassum* sp. as a valuable resource for CNC production, contributing to its utilization as a nanomaterial in a wide range of applications.

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