

Fabrication of Microcrystalline Cellulose From Waste Paper as Potential Eco-Friendly Cellulose Aerogel Material

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ABSTRACT

Waste paper was a raw material with a relatively high cellulose content, ranging from 40–80%. It was also an abundant feedstock that had already undergone partial processing to remove lignin, which provided advantages for cellulose extraction. In this study, microcrystalline cellulose was successfully extracted through a chemical route using a NaOH/H₂O₂ reaction system followed by hydrolysis with H₂SO₄. Scanning electron microscopy (SEM) results revealed that, after chemical treatment, the surface of the obtained microcrystalline cellulose became smooth, and no impurities were observed. Fourier-transform infrared spectroscopy (FTIR) spectra showed absorption peaks at wavenumbers corresponding to the vibrational modes of O–H, C–H, and C–O bonds, which were characteristic of the chemical structure of cellulose. X-ray diffraction (XRD) patterns of the microcrystalline cellulose exhibited higher diffraction intensity compared to those of the raw material. Thermogravimetric analysis (TGA) demonstrated that the extracted microcrystalline cellulose possessed greater thermal stability than the original waste paper. Finally, Brunauer–Emmett–Teller (BET) analysis showed that the cellulose aerogel fabricated from microcrystalline cellulose had a specific surface area of 0.711 m²/g, a BJH pore volume of 0.003424 cm³/g, and an average pore diameter of 34.99 nm. These results demonstrate that microcrystalline cellulose obtained from waste paper is a promising raw material for the fabrication of environmentally friendly cellulose aerogels applicable in diverse fields.

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1. Introduction

In the context of the circular economy and emission reduction targets, the recycling of cellulose-rich waste streams, particularly wastepaper, has emerged as a sustainable strategy for generating value-added materials [1]. The global volume of municipal solid waste is increasing rapidly, with paper and paperboard products accounting for approximately 17–25% of total household waste in many developed and emerging economies, thereby placing substantial pressure on landfills and waste management systems [2]. Wastepaper typically contains 40–80% cellulose, 5–15% hemicellulose, together with smaller amounts of lignin and protein [3]. This makes it an abundant feedstock that has already undergone partial processing (remove lignin), which facilitates the subsequent extraction of cellulose.

Cellulose is the most abundant natural polymer, with an annual global production estimated at 10¹¹–10¹² tons [4]. It is a polysaccharide composed of repeating β-D-glucose units linked via β-1,4-glycosidic bonds. Structurally, cellulose is a linear semi-crystalline polymer consisting of long chains of D-glucose units connected through β-1,4-glycosidic linkages between D-glucopyranosyl residues [5]. Moreover, cellulose exhibits notable physicochemical properties, including high crystallinity, large specific surface area, non-toxicity, high mechanical strength, hydrophilicity, low density, flexibility, and the ability to form networks [6]. Consequently, cellulose holds significant potential for applications in diverse fields such as energy, environmental remediation, and biomedicine [7].

Several methods for the isolation of cellulose from biomass are widely employed, including chemical (acid or alkali pretreatment), biological (bacterial or fungal), and mechanical approaches (ultrasonication, microwave irradiation, steam explosion), to eliminate hemicellulose, lignin, pectin, and other amorphous components [5], [8]-[10]. Subsequently, acid hydrolysis is carried out using inorganic acids (e.g., H_2SO_4 , HNO_3) or organic acids (e.g., acetic acid, formic acid) to remove the paracrystalline regions of cellulose, thereby obtaining microcrystalline cellulose (MCC) [5]. The obtained MCC is then used to fabricate cellulose aerogels through a multistep process, beginning with the dissolution of cellulose in an appropriate solvent to achieve uniform dispersion of the cellulose fibers. This is followed by a gelation process, during which a three-dimensional porous network is formed. Solvent exchange is subsequently performed to remove the original solvent and simultaneously adjust the pore size and distribution, thereby optimizing the specific surface area and adsorption capacity of the material. In addition, the thawing cycle is a crucial step in forming the porous structure of the cellulose aerogel. Finally, the gel is dried to stabilize the porous network [11]. The morphology and structure of the resulting aerogel strongly depend on the drying method. When conventional drying techniques such as oven drying are employed, pore collapse may occur, leading to reduced porosity, specific surface area, and adsorption efficiency [12]. Therefore, freeze-drying is commonly applied, as this method preserves the three-dimensional network structure of the gel, resulting in a material with high porosity, low density, and a large specific surface area, as well as enhanced adsorption performance.

In recent years, researchers have demonstrated the feasibility of converting wastepaper into cellulose aerogels at a lower cost than commercial aerogels, thereby opening promising opportunities for multidisciplinary applications. For instance, a superhydrophobic cellulose aerogel derived from paper waste and combined with candelilla wax exhibited an adsorption capacity for oils and organic solvents of up to 200 g/g, along with outstanding oil/water separation efficiency for environmental remediation [13]. In addition, cellulose aerogels synthesized from wastepaper were functionalized with graphene oxide and aminosilane to enhance CO_2 adsorption capacity, offering a promising solution for environmental management [14]. In the biomedical field, biocompatible cellulose aerogels have been applied in drug delivery systems and as hemostatic materials [15]. In the construction sector, hybrid composites of recycled cellulose and silica aerogels have exhibited high thermal stability, low thermal conductivity (0.025–0.035 W/m·K), and, after surface modification, improved flame resistance, underscoring their potential to replace conventional thermal insulation materials [16]. Therefore, the valorization of wastepaper to produce microcrystalline cellulose (MCC) through chemical methods, followed by its utilization in cellulose aerogels fabrication, not only contributes to addressing the challenges of solid waste management but also enables the development of next-generation bio-based materials with advanced, sustainable, and environmentally friendly applications.

In this study, microcrystalline cellulose was successfully extracted from waste paper pulp using a $NaOH/H_2O_2$ system followed by acid hydrolysis. The obtained microcrystalline cellulose was subsequently used to prepare cellulose aerogel using a $NaOH/urea$ system, involving a thawing process and freeze-drying to generate a three-dimensional porous structure. This work effectively valorized cellulose-rich waste paper - an abundant, low-cost, and commonly discarded material - into a high-value microcrystalline cellulose material with potential application in the fabrication of environmentally friendly cellulose aerogels applicable in diverse fields.

2. Materials and Methods

2.1. Materials

Waste paper was collected from a photocopy office. Hydrogen peroxide (H_2O_2), Sodium hydroxide ($NaOH$), Sulfuric acid (H_2SO_4), Ethanol (C_2H_5OH), Chloroform ($CHCl_3$), Urea (NH_2CONH_2) were purchased from Sigma-Aldrich.

2.2. Extraction of Cellulose from Waste Paper

The raw waste paper was first sorted and shredded, then washed repeatedly with water and soaked for 12 h to remove impurities. The suspension was subsequently heated for 2 h, filtered, and allowed to cool to room temperature. The material was then dried at 45 °C for 48 h and ground using a mechanical grinder. Five grams of shredded waste paper were treated with 200 mL of a mixed solution of 4% $NaOH$

and 30% H₂O₂ (1:1, v/v), and the suspension was magnetically stirred at 70 °C for 3 h. The resulting mixture was filtered and washed with distilled water until neutral pH = 7, followed by drying in an oven at 45 °C to constant weight.

2.3. Preparation of Microcrystalline Cellulose by Acid Hydrolysis

First, 1 g of cellulose extracted from waste paper was dispersed into a 5.5 M H₂SO₄ solution at a solid-to-liquid ratio of 1:20 (g/mL), with continuous stirring at 500 rpm at 45 °C for 1 h. After completion of the cellulose hydrolysis, the resulting suspension was washed several times with distilled water, followed by centrifugation at 5000 rpm for 10 min; this step was repeated three times. Finally, the product was neutralized with 1% NaOH solution until neutral pH.

2.4. Preparation of Cellulose Aerogel

Cellulose aerogel was prepared by dispersing 1 g of microcrystalline cellulose in 50 mL of a mixed aqueous solvent containing 1 M NaOH, urea, and distilled water at a volumetric ratio of 1:1:1. The suspension was stirred and ultrasonicated for 15 min at 40 °C, then frozen for 12 h. After thawing to room temperature, ethanol was added under continuous stirring, and the mixture was refrozen for an additional 12 h. The sample was subsequently thawed to room temperature and washed repeatedly with distilled water. Finally, the product was freeze-dried for 48 h to obtain cellulose aerogel. Figure 1 illustrated the extraction process of microcrystalline cellulose and the fabrication of cellulose aerogel.

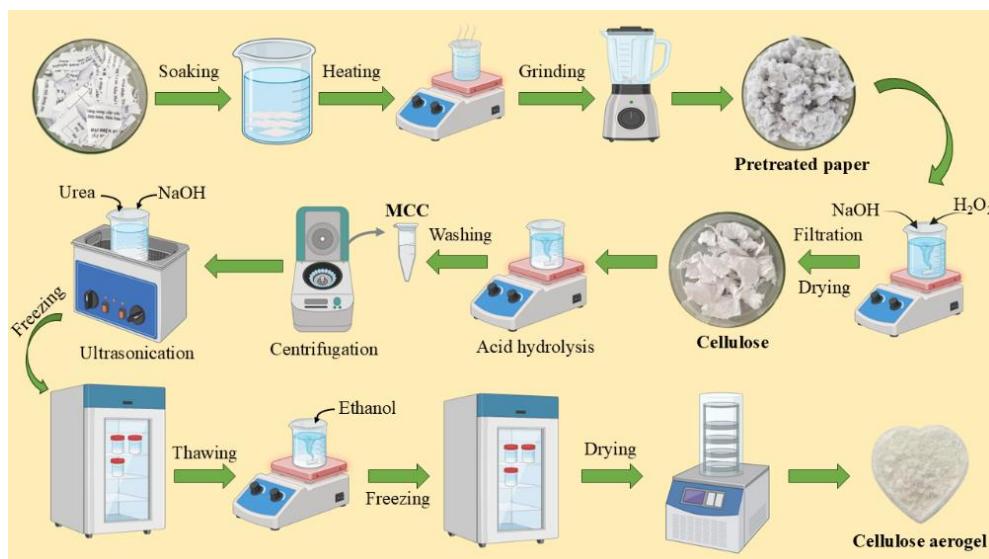


Figure 1. The fabrication process of cellulose aerogel based on microcrystalline cellulose derived from waste paper.

2.5. Characterization

2.5.1. Yield of Microcrystalline Cellulose

The yield of microcrystalline cellulose was calculated according to Equation (1) [17]:

$$H (\%) = \frac{m}{m_o} \times 100 \quad (1)$$

Where H (%) represents the yield, m is the dry mass of the obtained microcrystalline cellulose, and m_o is the mass of the initial raw material.

2.5.2. Surface Morphology of Waste Paper and Microcrystalline Cellulose

Scanning electron microscopy (SEM) was employed to investigate the surface morphology of waste paper and the extracted microcrystalline cellulose. Prior to imaging, the samples were sputter-coated with platinum and dried, and subsequently analyzed using an S-4800 SEM (Hitachi, Japan) operated at 10 kV accelerating voltage.

2.5.3. Fourier Transform Infrared Spectroscopy (FTIR)

FTIR spectroscopy was used to characterize the chemical structure of the samples before and after treatment. Spectra were recorded using a NIR MIR Frontier spectrometer (PerkinElmer) over a wavenumber range of 4000–600 cm^{-1} , with a resolution of 4 cm^{-1} and 20 scans per sample.

2.5.4. X-ray Diffraction (XRD)

X-ray diffraction (XRD) was employed to analyze the crystalline structure and crystallinity index (CI) of the samples before and after treatment. The measurements were carried out on an EMPYREAN diffractometer (PANalytical) using Cu K α radiation ($\lambda = 0.1542 \text{ nm}$), within a 2θ range of 10° – 40° , at a scanning rate of $2^\circ/\text{min}$, under operating conditions of 40 kV and 40 mA.

The crystallinity index (CI) of microcrystalline cellulose was calculated using Equation (2) [18]:

$$CI(\%) = \frac{I_{200} - I_{am}}{I_{200}} \times 100 \quad (2)$$

Where I_{200} is the maximum diffraction intensity at $2\theta \approx 22.8^\circ$, corresponding to both crystalline and amorphous regions, and I_{am} is the minimum intensity at $2\theta \approx 18.6^\circ$, corresponding to the amorphous regions of cellulose.

2.5.5. Brunauer–Emmett–Teller (BET)

The Brunauer–Emmett–Teller (BET) method was employed to determine the characters of porous structure and specific surface area of the fabricated cellulose aerogel material. The pore volume and pore size distribution of the fabricated cellulose aerogel were determined based on the N_2 adsorption characteristics using the Barrett–Joyner–Halenda (BJH) method [19]. The parameters such as specific surface area, BJH pore volume, and average pore diameter were automatically calculated by the instrument software (BET 201A, Porous Materials Inc., USA).

3. Results and Discussion

3.1. Extraction Yield of Microcrystalline Cellulose

The extraction yield of microcrystalline cellulose from waste paper, obtained using the alkaline hydrogen peroxide method followed by acid hydrolysis, was 38.12%, as calculated according to Equation 1. This extraction yield was relatively higher than that reported in another study (35.55%) [20]. The extraction yield is influenced by various factors such as the type of used waste paper (different cellulose content), extraction method, extraction conditions (treatment parameters), and the skill of researcher conducting experiments. Another published study indicated that the extraction yield of cellulose nanocrystals from office paper waste reached as high as 87.10% under optimal treatment conditions determined using response surface methodology (RSM) [21].

3.2. SEM Results

The SEM images of the waste paper and the extracted microcrystalline cellulose were shown in Figure 2. The results indicated that the surface of the waste paper was rough, non-uniform, and exhibited significant agglomeration. In addition, the surface morphology of the waste paper revealed a considerable amount of impurities with micrometer-size impurities [22].

In contrast, after chemical treatment and acid hydrolysis, the obtained microcrystalline cellulose exhibited a smoother and more homogeneous surface. This observation demonstrated that the treatment effectively removed hemicellulose and lignin, disrupted agglomerated domains, and facilitated the separation of cellulose fibers, led to the formation of a three-dimensional network structure [23]. However, after drying, the cellulose fibrils tended to reaggregate into larger bundles, most likely due to hydrogen bond formation between the hydroxyl groups of adjacent cellulose molecules.

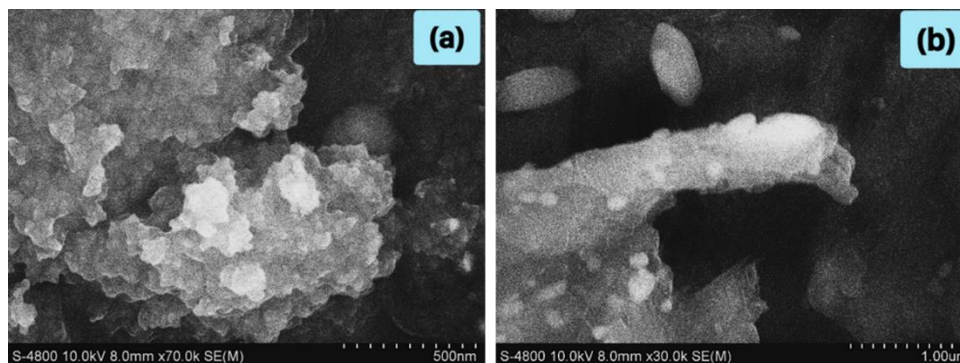


Figure 2. SEM images of (a) waste paper (WP) and (b) microcrystalline cellulose (MCC).

3.3. FTIR Analysis

FTIR spectroscopy were recorded to identify the vibrational modes of functional groups present in the samples during the preparation of microcrystalline cellulose. Figure 3 showed the FTIR spectra of raw paper and extracted microcrystalline cellulose.

The FTIR spectra of all samples exhibited characteristic vibrations corresponding to the functional groups in cellulose molecules. A broad absorption band at $3200\text{--}3500\text{ cm}^{-1}$ was attributed to the stretching vibrations of free hydroxyl (O-H) groups located on the surface of cellulose chains [24]. The peak observed at 2900 cm^{-1} corresponded to the (C-H) stretching vibrations of methylene CH_2 groups present in the chemical structures of cellulose, hemicellulose, and lignin [25]. Moreover, the FTIR spectra of raw paper and microcrystalline cellulose did not show any significant differences. However, several absorption peaks characteristic of vibrational modes associated with lignin and hemicellulose appeared only in the raw paper spectrum and were absent in the microcrystalline cellulose spectrum. Specifically, the band at $1730\text{--}1750\text{ cm}^{-1}$ was assigned to the stretching vibration of acetyl carbonyl (C=O) groups in hemicellulose or ester ($-\text{COO}-$) groups of ferulic acid moieties in lignin. In addition, the absorption peak at 1640 cm^{-1} was attributed to the bending vibration of (O-H) bonds from adsorbed water molecules [26]. The deformation vibrations of (C-O-H) groups were observed in the region of $1200\text{--}1400\text{ cm}^{-1}$, often coupled with other vibrational modes, appearing as relatively weak peaks. The band at $1000\text{--}1100\text{ cm}^{-1}$ was assigned to (C-O) stretching vibrations and (C-H) deformation within the pyranose ring of cellulose [26]. Finally, the peak at 899 cm^{-1} was associated with the β -glycosidic linkages between glucose units in the cellulose backbone [27]. These findings were consistent with those in the previously published study [26].

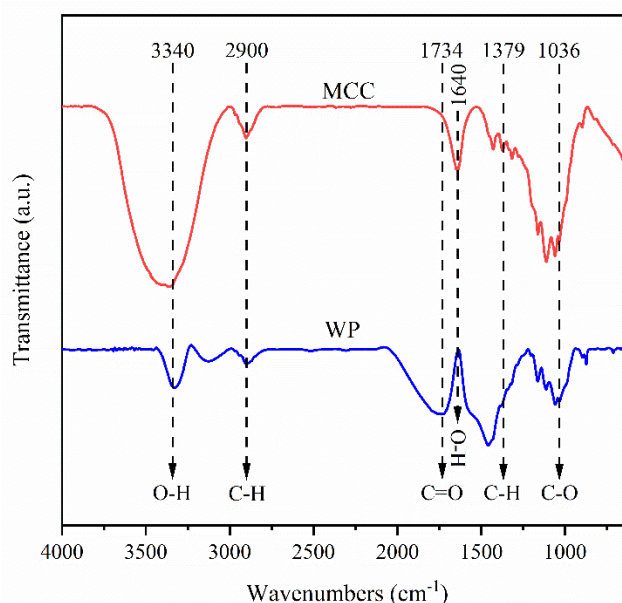


Figure 3. FTIR spectra of waste paper (WP) and microcrystalline cellulose (MCC).

3.4. XRD Characterization

The XRD patterns of the raw material and extracted microcrystalline cellulose were presented in Figure 4. Both samples exhibited characteristic diffraction peaks at 2θ values of 16.01° , 22.6° , and 34.1° , corresponding to the (110), (200), and (004) planes, which indicated the cellulose I crystal structure. In addition, compared with the microcrystalline cellulose, several less distinct peaks observed at 25.6° , 28.2° , 29.2° , 34.1° , 37.9° , and 38.7° in the raw material sample were attributed to the presence of calcite [28]. This indicates that the microcrystalline cellulose, after chemical treatment, has effectively removed calcite. Furthermore, the XRD pattern of the extracted microcrystalline cellulose showed higher diffraction intensities than that of the raw material. The crystallinity indices of the raw material and microcrystalline cellulose were 70.85% and 88.86%, respectively. The higher diffraction intensity and crystallinity of the extracted microcrystalline cellulose were attributed to chemical treatment and acid hydrolysis with H_2SO_4 , which effectively removed components such as hemicellulose and lignin from the chemical structure of the raw material. This process promoted the formation of hydrogen-bonded networks and enhanced the diffraction of the extracted cellulose [29]. These results were consistent with the FTIR findings reported above.

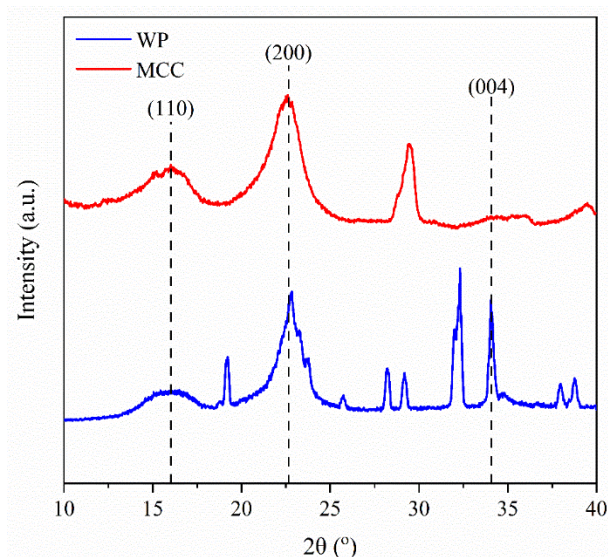


Figure 4. XRD patterns of waste paper (WP) and microcrystalline cellulose (MCC).

3.5. TGA Results

The comparison of the thermal stability of raw paper and the extracted microcrystalline cellulose samples was shown in Figure 5. The TGA curves of all samples exhibited three main stages of thermal decomposition: stage (I) below $130^\circ C$, stage (II) from 260 – $400^\circ C$, and stage (III) from 400 – $600^\circ C$. The thermal parameters were summarized in Table 1.

Table 1. Thermal parameters of raw waste paper (WP) and microcrystalline cellulose (MCC).

Sample	T_{max} ($^\circ C$)	Weight loss (%)	Residue (%) at $600^\circ C$
WP	358	67.14	23.95
MCC	356	55.33	37.48

In the first stage, the mass loss of both samples occurred between 35 – $130^\circ C$, corresponding to approximately 3% of the initial mass. This mass loss was attributed to the evaporation of water and moisture present in the samples [30]. Stage II represented the primary decomposition of the material, with mass losses of 67.14% and 55.33% for raw paper and microcrystalline cellulose, respectively. Additionally, the extracted microcrystalline cellulose exhibited higher thermal stability than the raw material. This was explained by the chemical treatment, which effectively removed low-thermal-decomposition amorphous component, and hemicellulose thereby enhancing the thermal resistance of

the microcrystalline cellulose [31]. The final stage corresponded to the carbonization of intermediate products formed during the previous stages, leaving residual masses of 23.95 % and 37.48 % for raw paper and microcrystalline cellulose, respectively.

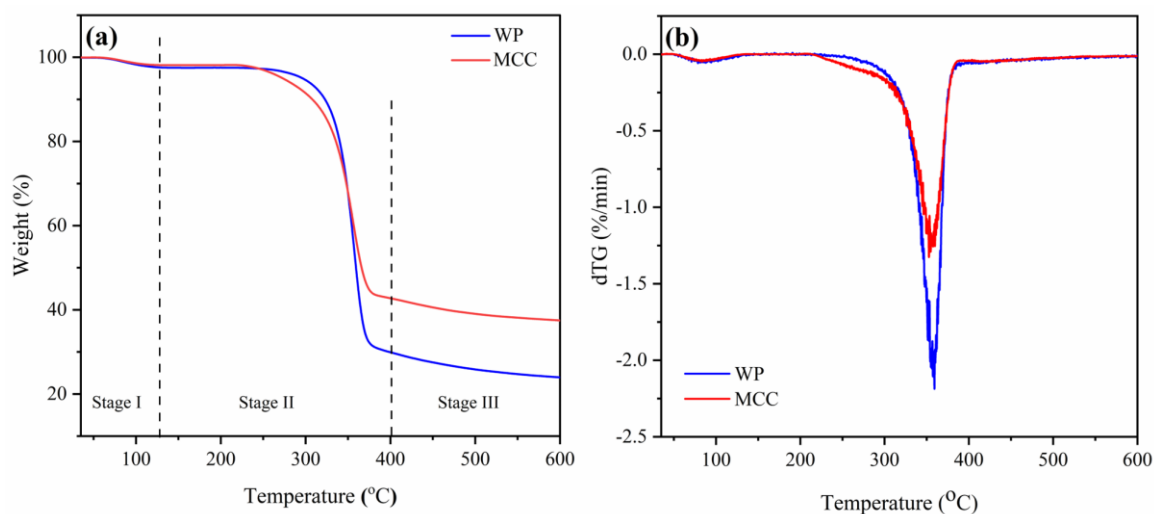


Figure 5. TGA curves of waste paper (WP) and microcrystalline cellulose (MCC).

3.6. Brunauer–Emmett–Teller Analysis

Figure 6 showed the nitrogen adsorption–desorption isotherms of the fabricated cellulose aerogel. The morphological and porosity characteristics of the cellulose aerogel were summarized in Table 2. The results indicated that the cellulose aerogel exhibited an average pore diameter of 34.99 nm and a BJH pore volume of 0.003424 cm³/g. The BJH pore volume value in Table 2 was determined using The Barrett-Joyner-Halenda method by analyzing N₂ adsorption/desorption isotherm result of the fabricated cellulose aerogel material (Figure 6). BJH pore volume indicates the cumulative volume of pores within a specific size range, while the pore size distribution presents the range of these volumes across different pore sizes [32].

Table 2. Textural parameters of cellulose aerogel.

No.	Parameter	Cellulose Aerogel
1	BET surface area (m ² /g)	0.711
2	BJH pore volume (cm ³ /g)	0.003424
3	Average pore diameter (nm)	34.99

Furthermore, Figure 6 showed that the N₂ adsorption–desorption isotherm at 77.35 K corresponded to a type IV isotherm according to the IUPAC classification, which was characteristic of mesoporous materials (pore sizes between 2 and 50 nm) [33]. The adsorption and desorption branches did not coincide, producing a hysteresis loop at relatively high relative pressures. This phenomenon was attributed to the “bottle-neck” core structure commonly observed in aerogel materials, in which the entrance was narrow but the internal cavity was wide. During adsorption, gas molecules easily entered the core, whereas during desorption, the gas was hindered from escaping, generating the difference between the adsorption and desorption branches [34]. Additionally, the BET specific surface area of the cellulose aerogel was measured to be 0.711 m²/g. The BET specific surface area of the fabricated cellulose aerogel was lower than that reported in other studies, as shown in Table 3. The relatively low BET surface area could be attributed to an insufficient number of thawing cycles to develop a well-defined porous structure. To enhance the specific surface area of the prepared cellulose aerogel, the number of thawing cycles should be increased and combined with the incorporation of other materials such as graphene oxide or activated carbon to enlarge the surface area, improve mechanical properties, enhance the reusability, and prolong the lifespan of the fabricated cellulose aerogel material.

Table 3. BET specific surface area of cellulose aerogel materials derived from various sources.

No.	Cellulose sources	BET surface area m ² /g	References
1	Waste paper	0.711	This study
2	Sunflower seed husk	0.3	[35]
3	Commercially microcrystalline cellulose	118	[36]
4	Commercially microcrystalline cellulose (MCC)	146-189	[37]
5	Bamboo powder	153.1	[38]

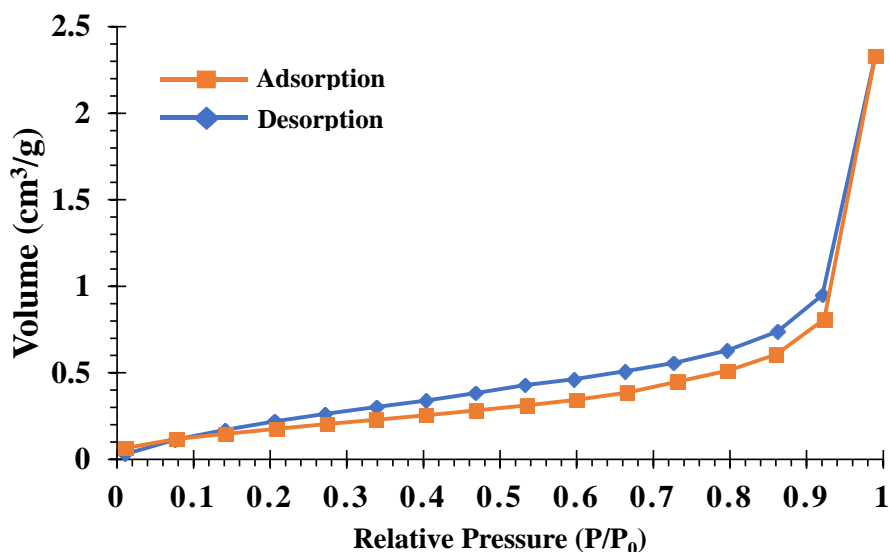


Figure 6. N₂ adsorption–desorption isotherm of cellulose aerogel at 77.35 K.

4. Conclusions

In this study, microcrystalline cellulose (MCC) was successfully extracted from waste paper (WP) with a yield of 38.12% using a NaOH/H₂O₂ reaction system followed by acid hydrolysis with H₂SO₄. SEM analysis revealed that the obtained MCC exhibited a smooth and homogeneous surface without detectable impurities. The FTIR spectra confirmed that the fundamental structure of cellulose remained intact after chemical treatments. Furthermore, the XRD patterns of the MCC indicated a cellulose I crystalline structure. The crystallinity index of the MCC and raw paper was 88.86% and 70.85%, respectively. In addition, TGA results demonstrated that the thermal stability of MCC was higher than that of the raw material. Finally, BET analysis showed that the cellulose aerogel fabricated from MCC had a specific surface area of 0.711 m²/g, a BJH pore volume of 0.003424 cm³/g, and an average pore diameter of 34.99 nm. These findings suggested that the extracted MCC possessed great potential for the fabrication of cellulose aerogels with applications in various fields.

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Conflict of Interest

The authors declare no conflict interest.

Data Availability Statement

The data that support the findings of this study are available from the corresponding author upon reasonable request.

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