



## RESEARCH ARTICLE

### VALORIZATION OF COCONUT COIR INTO NANOCELLULOSE: EXTRACTION AND PHYSICOCHEMICAL-NANOSTRUCTURAL CHARACTERIZATION TOWARD SLOW-RELEASE BIOFERTILIZER INNOVATION

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#### ABSTRACT

Agricultural waste valorization is a strategic approach for developing sustainable bio-based products. Coconut coir, abundantly generated in the Philippines, remains underutilized and poses environmental disposal challenges. Nanocellulose derived from such waste exhibits promising properties for agricultural applications, particularly as a carrier for biofertilizer encapsulation. This study aimed to develop and characterize nanocellulose extracted from coconut coir under a sustainable, low-cost framework, assessing its physicochemical, morphological, and chemical suitability as a carrier matrix for future nano-biofertilizer systems. Coconut coir dust underwent sequential alkaline pretreatment (5% NaOH), bleaching using commercial bleach, controlled acid hydrolysis (2M HCl or combined H<sub>2</sub>SO<sub>4</sub>/HCl), and ultrasonic dispersion to produce three nanocellulose samples. Colloidal properties were characterized using dynamic light scattering (DLS), zeta potential analysis, and optical tests (Tyndall effect). Morphology was evaluated via scanning electron microscopy (SEM), while chemical composition was determined by FTIR spectroscopy. The DA2-SA sample achieved nanoscale dimensions (83.4 ± 6.7 nm), with robust colloidal stability evidenced by high negative zeta potentials (−74 mV to −97 mV). SEM revealed entangled fibrillar networks (~10–20 μm widths), and FTIR confirmed high cellulose purity with dominant O–H, C–H, and C–O–C bands, minimal carbonyl signals indicating effective lignin removal. The process demonstrated feasibility using low-cost methods, supporting circular bioeconomy goals. Coconut coir can be sustainably transformed into cellulose nanomaterial with physicochemical and morphological attributes suitable for encapsulating beneficial microbes in nano-biofertilizer systems, contributing to climate-resilient and resource-efficient agriculture.

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## INTRODUCTION

Sustainable agriculture is crucial for meeting global food demands while preserving environmental integrity. Current intensive farming practices have led to soil degradation, biodiversity loss, and pollution<sup>1,2</sup>. To address these challenges, there is a growing emphasis on integrating ecological principles into crop production. Organic amendments and bio-fertilizers play a vital role in enhancing soil fertility, improving nutrient cycling, and reducing reliance on synthetic inputs<sup>3</sup>. Plant growth-promoting microorganisms and nanotechnology offer promising alternatives to harmful agrochemicals<sup>2</sup>. Innovative approaches such as climate-smart agriculture, organic farming, and precision agriculture can help meet increasing food demands while preserving the environment<sup>2</sup>. However, addressing socioeconomic issues like inequality in

resource access and population growth is also essential for achieving sustainability<sup>1</sup>. These strategies collectively contribute to a more sustainable agricultural system that balances productivity with environmental conservation. Biofertilizers, containing beneficial microorganisms, offer a sustainable alternative to chemical fertilizers in agriculture. They enhance soil fertility and crop productivity by promoting nutrient availability through mechanisms like nitrogen fixation, phosphate solubilization, and potassium mobilization<sup>4,5</sup>. These microbial formulations improve soil structure, increase organic matter content, and contribute to carbon sequestration<sup>4</sup>. Biofertilizers also enhance crop yields, reduce chemical fertilizer dependence, and foster sustainable farming practices<sup>6</sup>. Despite their potential, challenges such as limited awareness, inconsistent field performance, and quality control issues hinder widespread adoption<sup>4,6</sup>. Addressing these challenges

through advanced delivery systems, improved formulation techniques, and effective policy support is crucial for successful implementation of biofertilizer technology<sup>4</sup>. Overall, biofertilizers play a key role in promoting sustainable agriculture and long-term soil health<sup>5,6</sup>. Carriers and encapsulation techniques play a crucial role in improving the viability and effectiveness of biofertilizers. Traditional carriers like peat have limitations, prompting research into alternatives such as clay, vermicast, and biochar<sup>7</sup>. Biochar, in particular, shows promise as a microbial carrier due to its favorable properties, including high porosity, moisture-holding capacity, and nutrient content<sup>8</sup>. Encapsulation methods, such as spray drying, interfacial polymerization, and cross-linking, offer enhanced protection and controlled release of microorganisms<sup>9</sup>. Various bioencapsulation techniques have been explored, including fluidized bed, extrusion, and ionic gelation, with sodium alginate being a commonly used material<sup>10</sup>. These advanced formulations aim to overcome the drawbacks of conventional inoculants, extending shelf life and improving field efficacy. Ongoing research focuses on developing innovative carrier formulations to maintain microbial viability and effectiveness during storage and application<sup>7</sup>.

Coconut waste valorization aligns with circular economy goals, offering sustainable solutions for managing vast quantities of coir waste in the Philippines<sup>11</sup>. Nanocellulose, derived from lignocellulosic biomass, is a promising high-value product with unique properties such as biocompatibility, high surface area, and mechanical strength<sup>12</sup>. Agricultural and industrial waste sources, including coconut coir, show great potential as inexpensive and sustainable raw materials for nanocellulose production<sup>13</sup>. Nanocellulose extraction from various lignocellulosic biomass sources, including coir, involves chemical treatments like alkaline treatment, bleaching, and acid hydrolysis<sup>14</sup>. The resulting nanocellulose typically has an average diameter of 10–25 nm, high crystallinity, and thermal stability, making it suitable for diverse applications in packaging, electronics, and environmental remediation<sup>12,14</sup>.

This study explores the production and applications of nanocellulose derived from coconut coir, a abundant agricultural waste. Coir pith, composed of cellulose, hemicellulose, and lignin, can be transformed into nanocellulose through chemical and mechanical treatments<sup>15</sup>. The resulting cellulose nanofibers, typically 30–90 nm wide, exhibit crystalline properties and contain hydroxyl, C–H, and C–O–C groups<sup>16</sup>. Advanced techniques like TEMPO-mediated oxidation can produce even finer nanofibrils, around 5.6 nm in diameter<sup>17</sup>. These nanocellulose materials show promise in various applications, including transparent films, filtration, water treatment, and as reinforcements in biodegradable composites<sup>15,17</sup>. The use of agricultural wastes like coconut coir for nanocellulose production aligns with sustainable practices, reducing environmental impact and fostering economic growth while closing the resource use circle<sup>18</sup>.

## MATERIALS AND METHODS

**Nanocellulose extraction from coconut coir:** Coconut coir dust was collected from local buko juice and tupig vendors in Brgy. San Felipe, City of Ilagan, Isabela. The material was air-dried, mechanically threshed, and sieved to two particle sizes:

- 60 mesh (250  $\mu\text{m}$ ) for Sample DA3-S1
- 120 mesh (125  $\mu\text{m}$ ) for Samples DA2-SA and DA1-MA.

### Each treatment began with:

- 5% NaOH at 80 °C for 2 h to remove lignin and hemicellulose.
- Bleaching using commercial bleach at 70 °C for 1 h.

Post-hydrolysis, samples were repeatedly washed with distilled water to neutral pH. Suspensions were then sonicated for 1–2 h in ice baths (15 min on / 5 min off cycles) for better dispersion.

### Physicochemical and colloidal analysis

#### Colloidal stability was first assessed by

- High-speed centrifugation (5000 rpm, 15 min) to observe biphasic separation.
- Tyndall effect test using a laser pointer to confirm light scattering by suspended nanoscale particles.

#### Samples were then submitted to the CLSU Nanotechnology R&D Facility for:

- Particle size and polydispersity index (PDI) via DLS (Horiba SZ-100) at 25 °C.
- Zeta potential measurement to evaluate electrostatic stability.

**Microstructural analysis:** Surface morphology was characterized by scanning electron microscopy (SEM). Dried samples were sputter-coated with gold and imaged at magnifications of 600 $\times$ , 1000 $\times$ , and 1500 $\times$  to assess fibril structure and porosity.

**Chemical analysis:** Functional groups were identified by Fourier Transform Infrared (FTIR) spectroscopy in the range 4000–500  $\text{cm}^{-1}$ , focusing on O–H, C–H, C–O, and carbonyl bands to verify cellulose purity.

## RESULTS

### Nanocellulose particle size and colloidal stability

- **DA2-SA:** Achieved nanoscale dimensions at  $83.4 \pm 6.7$  nm, with low polydispersity, meeting the ISO/TS 20477:2017 criterion for nanocellulose (<100 nm).
- **DA3-S1:** Larger mean size at  $428.6 \pm 37.2$  nm.
- **DA1-MA:** Reached  $1177.5 \pm 146.4$  nm, indicating partial defibrillation.

All samples had highly negative zeta potentials ranging –73.7 mV to –96.7 mV, surpassing the  $\pm 30$  mV benchmark for stable colloids. DA3-S1 showed the highest stability (–96.7 mV).

**Visual and Tyndall assessments:** All suspensions remained milky post-centrifugation with stable supernatants over time. Strong Tyndall scattering was observed in all, confirming dispersed nano- to sub-micron particles.

### Three nanocellulose extraction treatments were prepared

Sample	Post-bleach drying	Acid hydrolysis	Ratio & Conditions
DA3-S1	Sun-dried	2 M HCl, 60 °C, 2 h	1 g fibers : 10 mL acid
DA2-SA	Wet	Strong acid (10 mL 12.1N HCl + 30 mL 36N H <sub>2</sub> SO <sub>4</sub> + 60 mL H <sub>2</sub> O), RT, 1 h	1 g : 40 mL acid mix
DA1-MA	Wet	2 M HCl, 60 °C, 2 h	1 g : 40 mL acid

### Microstructural characteristics

- SEM images revealed porous, entangled fibrillar networks averaging ~10–20 µm in width. While larger than DLS values, this morphology provides extensive surface area ideal for future microbial encapsulation.

### FTIR analysis

#### All samples showed characteristic cellulose bands:

- O–H stretching (~3338 cm<sup>-1</sup>)
- C–H (~2898 cm<sup>-1</sup>)
- C–O–C and C–O stretching (1159–1034 cm<sup>-1</sup>)

Minimal carbonyl peaks near 1735 cm<sup>-1</sup> indicated effective removal of lignin and hemicellulose, confirming high cellulose purity.

## DISCUSSION

This study demonstrated that coconut coir can be sustainably converted into high-purity nanocellulose under mild chemical and energy-efficient conditions.

- DA2-SA, treated with strong acid on wet-bleached fibers, produced the smallest particles (<100 nm)<sup>19</sup> with excellent colloidal stability (−74 mV)<sup>20</sup>. This suggests avoiding drying before hydrolysis helps preserve accessible cellulose, enhancing nanoscale breakdown.
- DA3-S1 and DA1-MA, despite larger mean sizes, still maintained robust zeta potentials (up to −97 mV), supporting stable dispersions for applications like nano-biofertilizer matrices<sup>21</sup>.

SEM confirmed porous fibrillar networks, while partially micro-scale, provide channels beneficial for microbe encapsulation and gradual release into soil<sup>22</sup>. FTIR confirmed minimal lignin and hemicellulose, ensuring compatibility with microbial systems and alginate-based encapsulation polymers<sup>23</sup>. Economically, this process relies on local agro-waste (P0 raw material cost) and low-concentration NaOH/HCl solutions, offering a substantial savings over commercial nanocellulose. This positions it as a viable alternative for farmer cooperatives and local biofertilizer producers, advancing circular bioeconomy principles.

## CONCLUSION

Coconut coir, an abundant Philippine agro-waste, was successfully valorized into nanocellulose using a simple, low-cost extraction method. The resulting material:

- Exhibited nanoscale dimensions (83 nm), strong electrostatic stability (−74 to −97 mV), and porous fibrillar networks.

- Showed high chemical purity, supporting its suitability as a carrier for microbial biofertilizers.

This foundation enables next-phase development of nano-biofertilizers combining *Rhizobium tropici* and *Bacillus safensis* for improved soil fertility and climate-resilient agriculture. The process also promotes sustainable waste management, rural income opportunities, and reduced dependence on imported nanomaterials.

### Glossary of Abbreviations

**CNF:** Cellulose nanofibers

**CNC:** Cellulose nanocrystals

**DLS:** Dynamic Light Scattering

**FTIR:** Fourier Transform Infrared Spectroscopy

**SEM:** Scanning Electron Microscopy

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