Isolation and characterization of microcrystalline cellulose from tangkit pineapple leaves (ACT-MCC) as a pharmaceutical excipient

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Abstract

Background: Microcrystalline cellulose (MCC) is a widely used pharmaceutical excipient due to its high compressibility, stability, and inertness in tablet formulations. Tangkit pineapple leaves (Ananas comosus), an abundant agricultural waste in Jambi, Indonesia, offer a sustainable alternative source for cellulose. **Objective**: This study reports the preparation and characterization of microcrystalline cellulose (ACT-MCC) derived from Tangkit pineapple leaves, as well as its physicochemical characteristics in comparison to the commercial standard, Avicel PH 102. **Methods:** MCC was prepared from α-cellulose isolated from dried Tangkit pineapple leaves through sequential alkali treatment, bleaching, and acid hydrolysis. **Results:** From 200 g of dried raw material, 19.41% (w/w) α-cellulose was obtained, which was then converted to 75% (w/w) MCC, equivalent to 14.56% of the starting material. The resulting ACT-MCC had a melting point of 247-250°C, slightly lower than Avicel PH 102 (268-270 °C). Physicochemical analysis showed a pH of 7.51, total ash 0.318% (w/w), moisture 3.13% (w/w), and drying loss of 3.42% (w/w), all within pharmacopeial limits. ACT-MCC was comparatively fine, odourless, tasteless, and yellowish in appearance. **Conclusion:** ATC-MCC demonstrated comparable characteristics to commercial MCC, indicating its potential usage as a locally derived pharmaceutical excipient.

Keywords: Tangkit pineapple leaves; MCC, ATC-MCC; Physicochemical Characterization; Pharmaceutical excipient.

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INTRODUCTION

Microcrystalline cellulose (MCC) is one of the most widely used excipients in modern pharmaceutical manufacturing, especially for solid oral dosage forms, due to its advantageous properties such as high compressibility, inertness, biodegradability, and multifunctionality as a filler, binder, and direct compression material (1). MCC's physicochemical characteristics directly influence tablet hardness, friability, disintegration, and dissolution performance (1,2).

The quality of MCC is determined by its crystallinity, particle morphology, moisture and ash content, density, flowability, hydration capacity, and thermal stability (1–3). Commercial MCC is typically produced from wood pulp or cotton linters using controlled acid hydrolysis (3,4). Avicel PH-101 and PH-102 are the industry standards. However, reliance on these conventional raw materials leads to obstacles such as high cost, limited availability, environmental deterioration from deforestation, and import dependency in developing countries such as Indonesia (5–7). These restrictions emphasise the need for sustainable, locally produced alternatives derived from lignocellulosic agricultural waste (4,5,7).

Agricultural lignocellulosic biomass offers abundant, inexpensive, and renewable sources of cellulose (7,8). Prior research demonstrates that MCC can be successfully isolated from diverse wastes such as pineapple leaves, cocoa pod husks, ramie fibers, and palm nut fibers (3,6,8). Yields and characteristics differ by source for instance, MCC from cocoa pod husks yields 38.25% with 43.83% crystallinity, while MCC from pineapple leaves achieves ~75% crystallinity, comparable to commercial Avicel (81.25%) (3,6). Such variability emphasizes the need to optimize extraction processes to obtain high-quality MCC suitable for pharmaceutical applications. Among agricultural sources, pineapple leaves (Ananas comosus L.) are particularly promising. They contain high cellulose content (26.9%), exhibit good thermal stability (~356°C pyrolysis temperature), and are abundantly available (6,9–12).

Indonesia, one of the largest global pineapple producers, generates significant quantities of pineapple leaf waste up to 70% of total biomass per harvest most of which remains unutilized (9,11). Utilizing this biomass aligns with circular economy principles by reducing waste, providing economic value to farmers, and supporting sustainable industry development (7,11). Tangkit pineapple, an indigenous Indonesian cultivar, is a unique and underutilised resource for MCC production. Although there is less research on MCC from Tangkit pineapple leaves, other pineapple types have shown consistent cellulose output and crystallinity, indicating Tangkit's significant potential (6,9,10,12,13). Developing MCC from Tangkit variety supports raw material diversification, import substitution, and national pharmaceutical self-sufficiency.

The isolation process of microcrystalline cellulose (MCC) generally comprises several key stages, including raw material preparation, alkali delignification to remove lignin and hemicellulose, bleaching to enhance purity and whiteness, and acid hydrolysis to obtain microcrystalline fractions (3,8–10,12,14,15). Considering the abundance of pineapple leaves and the increasing requirement for sustainable pharmaceutical excipients, this study aims to isolate and characterise microcrystalline cellulose obtained from Tangkit pineapple leaves (ACT-MCC). Specifically, the aims are to isolate the MCC and assess its physicochemical features. The long-term objective is to establish ACT-MCC as a viable, environmentally friendly, and locally sourced excipient for Indonesia's pharmaceutical industry.

METHODS

Study design and setting

This experimental study was conducted at the Pharmaceutical Laboratory, Faculty of Medicine and Health Sciences, Universitas Jambi, from July to October 2025.

Materials

Pineapple leaves (Ananas comosus L. Merr., Tangkit variety) were collected from local farmers in Tangkit Baru Village, Sungai Gelam District, Muaro Jambi Regency, Jambi Province, Indonesia. Reagents, including sodium hydroxide (NaOH), sodium hypochlorite (NaClO), and hydrochloric acid (HCl), were obtained from Merck. Commercial microcrystalline cellulose (Avicel PH 102, FMC Biopolymer, USA) was used as a reference standard.

Procedure and data collection

The collected pineapple leaves were thoroughly cleansed with water to eliminate dust and contaminants before being air-dried for seven days in direct sunlight. Dried leaves were oven-dried at 60 °C for 48 h and subsequently ground to a fine powder passing through a 24-mesh sieve. The resulting powder, referred to as ACT powder, served as the starting material for cellulose isolation.

Each step was carefully managed to ensure that non-cellulosic components were effectively removed while cellulose's crystalline integrity was maintained, resulting in high-purity microcrystalline cellulose from Tangkit pineapple leaves. In the first stage, alkali delignification was carried out by treating powdered pineapple leaf cellulose with a 2% NaOH solution at 80°C for five hours. This method successfully dissolved lignin, hemicellulose, and other soluble components. The treated mixture was then filtered, washed repeatedly with distilled water until the filtrate achieved a neutral pH, and dried in an oven at 60°C overnight to yield the alkali-treated cellulose fraction (5,15–18). In the second stage, the alkali-treated cellulose was bleached with NaClO solutions (1:1 and 1:3) at 75 °C to remove remaining colours and organic impurities, resulting in purified α-cellulose.

The third stage was acid hydrolysis with HCl 2.5 N at 100 °C for 15 minutes to selectively break down the cellulose's amorphous areas and produce microcrystalline cellulose. The resultant suspension was diluted with cold water, left to stand overnight for complete precipitation, filtered, washed till neutral, and oven dried at 60 °C to achieve the final ACT-MCC product. (5,15–17,19–22). Pharmacopeial procedures were used to determine the physicochemical parameters, such as moisture content, total ash, pH, drying loss, and melting point.

Statistical analysis

Data were expressed as mean ± standard deviation (SD) from triplicate experiments to ensure statistical reliability. The physicochemical properties of the isolated ACT-MCC were compared to those of commercial microcrystalline cellulose (Avicel PH 102) used as a reference standard.

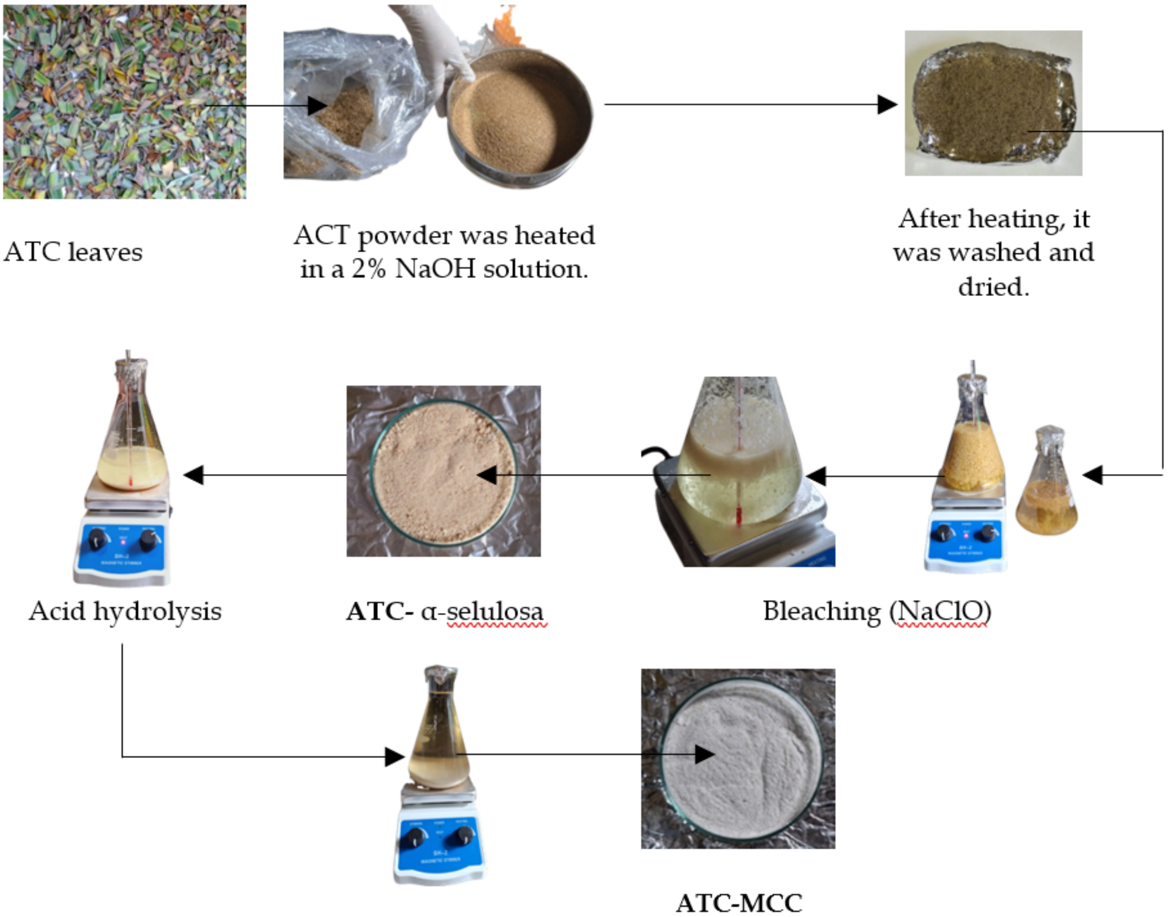

Figure 1. Isolation of ACT-MCC

RESULTS

Tangkit pineapple leaves yielded 19.41% (w/w) α-cellulose when isolated from the dried biomass. Acid hydrolysis transformed α-cellulose to microcrystalline cellulose (MCC) at a conversion efficiency of 75% (w/w), yielding 14.56% (w/w) more than the original dry leaves. These findings show that lignin, hemicellulose, and non-cellulosic components were effectively removed throughout the successive alkali delignification, bleaching, and acid hydrolysis procedures. The comparatively high cellulose recovery is due to Tangkit pineapple leaves' naturally high cellulose content (26.9%) and low lignin composition (about 4-5%), which ease chemical treatment and purification.

Table 1. Physicochemical properties of ACT-MCC compared with Avicel PH 102.

Parameter	ACT-MCC	Avicel PH 102	Specification	Evaluation
Appearance & Color	Fine, pale-yellow powder	White powder	–	Typical of natural MCC
Odor & Taste	Odorless, tasteless	Odorless, tasteless	–	Within standard
pH (1% dispersion)	7.51±0.14	7.42	5.0–7.5	Neutral and stable
Moisture Content (%w/w)	3.13±0.49	3.05	< 5.0	Acceptable
Total Ash (%w/w)	0.32±0.04	0.31	< 0.5	Acceptable

Parameter	ACT-MCC	Avicel PH 102	Specification	Evaluation
Loss on Drying (% w/w)	3.42±0.36	3.10	< 5.0	Acceptable
Melting Point (°C)	247–250±2.89	268–270	–	Slightly lower than Avicel

According to yield estimations, the method has good scalability. Assuming an average production of 2-3 tonnes of pineapple leaf waste per hectare per harvest, the potential output of MCC can be between 290 and 440 kg per hectare, demonstrating the viability of local biomass valorisation for industrial-scale MCC production. The isolated ACT-MCC appeared as a fine pale-yellow powder that was odourless and tasteless. Table 1 compares ACT-MCC's physicochemical parameters to commercial Avicel PH 102. All metrics were within the pharmacopeial range (USP-NF and Indonesian Pharmacopoeia). The neutral pH (7.51) allows for compatibility with a wide range of active medicinal substances, while the low moisture content (3.13%) and total ash (0.32%) guarantee high purity and storage stability. The melting point range (247-250 °C) is slightly lower than Avicel PH 102, however it is still suitable for pharmaceutical use.

DISCUSSION

The isolation procedure effectively produced high-purity microcrystalline cellulose (MCC) from Tangkit pineapple leaves (Figure 2), demonstrating both the efficacy of the chemical treatments used and the agricultural residue's promise as a renewable raw material. The combination of alkali delignification, graded bleaching, and acid hydrolysis effectively removed lignin and hemicellulose while preserving the crystalline domains of cellulose, resulting in an α-cellulose yield of 19.41% and a 75% conversion rate into MCC (14.56% of the original biomass) (23).

Figure 1. Tangkit pineapple

These findings are consistent with previous studies reporting MCC yields ranging from 10% to 22% from various lignocellulosic sources. Gunawan and Lestari (2020) obtained a 22% MCC yield from pineapple leaves (Ananas comosus L. Merr.) using a hydrogel-based isolation method (24), whereas Oluwasina et al. (2014) reported remarkably high yields exceeding 80% from banana species (Musa sapientum and Musa paradisiaca) through ethanol pulping (25). Other agricultural residues have also yielded variable amounts of MCC; ramie fiber produced 57.26%, carrot pomace yielded 36.62% under optimized autoclaving and ultrasonic pretreatment (26), and Albizia lebeck leaves produced a maximum cellulose yield of 50.2%.

Tangkit pineapple leaves have a comparatively high yield due to their naturally high cellulose content and low lignin proportion, which facilitate chemical delignification and cellulose recovery. Pineapple leaf fibres are well known as a rich source of cellulose with favourable chemical composition for MCC production (18,27). Reported crystallinity indices of pineapple leaf-derived MCC range from 73.48% to 83.16%, depending on processing parameters (22,28), which closely approximate the crystallinity of commercial Avicel PH 102 (81.25%) (29). This similarity confirms the structural integrity and quality of the MCC isolated from Tangkit pineapple leaves.

Beyond its technical performance, the production of ACT-MCC from Tangkit pineapple leaves represents a sustainable and economically valuable approach consistent with the principles of the circular economy (13). Indonesia serves as one of the world's largest pineapple cultivators, yet a large percentage of its agricultural biomass is underutilised and commonly thrown through open burning, which adds significantly to air pollution. Agricultural residues, such as pineapple leaves, are frequently viewed as garbage, emphasising the importance of valorisation initiatives that convert them into value-added goods. Converting this biomass into a high-value pharmaceutical excipient not only decreases environmental problems, but also boosts local industries, increases farmer income, and lessens national reliance on imported MCC. Several studies have showed that the transformation of agricultural leftovers such as pineapple leaves, banana stems, and carrot pomace into MCC produces environmentally and economically feasible products appropriate for pharmaceutical and industrial applications (25,26).

CONCLUSIONS

ACT-MCC had physicochemical parameters similar to commercial Avicel PH 102. The material's pH, ash content, moisture content, and thermal stability all met pharmacopeial criteria, indicating its possible use as a pharmaceutical excipient. Furthermore, considering Tangkit pineapple leaf waste encourages local innovation in the manufacture of pharmaceutical raw materials.

CONFLICT OF INTEREST

The authors declare that the research was conducted in the absence of any commercial or financial relationships that could be construed as a potential conflict of interest.

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DECLARATION OF ARTIFICIAL INTELLIGENCE USE

This manuscript was prepared with the assistance of QuillBot, a language refinement and paraphrasing tool, to improve clarity and readability. All edits and refinements were critically reviewed by the authors to ensure the accuracy, integrity, and reliability of the content. All interpretations, results, and conclusions were made solely by the authors.

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