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Modification of Microcrystalline Cellulose (MCC) Derived from Liberica Coffee Husk Using Saponins for Controlled Release of Dexamethasone

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ABSTRACT

Microcrystalline cellulose (MCC) is a partially depolymerized cellulose derivative obtained through the hydrolysis of α-cellulose. However, MCC has limitations in drug delivery systems, particularly for poorly water-soluble (hydrophobic) drugs; therefore, MCC modification is required to improve the loading and delivery efficiency of such compounds. This study aimed to modify MCC produced from the hydrolysis of Liberica coffee husk cellulose to enable its application as a carrier for hydrophobic drugs. The modification was carried out using saponins, followed by dexamethasone loading into MCC–saponin and unmodified MCC using ethanol as the solvent, with incubation for 24 h. The MCC–saponin system exhibited a substantially higher dexamethasone loading capacity (23.436 mg/g) than unmodified MCC (6.768 mg/g). In vitro drug release results further showed that dexamethasone release from MCC–saponin increased within 60–180 min.

Keywords: Liberica coffee husk Dexamethasone, Drug Delivery System, Microcrystalline Cellulose, Cellulose.

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INTRODUCTION

Cellulose $(C_6H_{10}O_5)_n$ is a renewable polysaccharide composed of linear polymer chains linked by β-1,4-glycosidic bonds. Cellulose is widely recognized as a renewable resource with biodegradable and biocompatible properties (Fitriana et al., 2018). Satriananda et al. (2022) reported that coffee husk typically contains a high proportion of cellulose, reaching approximately 63%, making it a promising lignocellulosic feedstock.

Jambi Province, particularly Jati Mulyo Village, Dendang Subdistrict, East Tanjung Jabung Regency, is one of the production areas of Liberica coffee. In this region, farmers commonly process harvested coffee into green beans and roasted coffee beans, which inevitably generate by-products such as coffee husk. Coffee husk consists of pulp, skin, mucilage, and parchment (Rebollo-Hernanz et al., 2021). The quantity of coffee husk is expected to increase in line with rising coffee productivity. Therefore, to mitigate

the accumulation of this agro-industrial residue, Liberica coffee husk was selected as a cellulose source for the production of cellulose derivatives, such as microcrystalline cellulose (MCC).

Microcrystalline cellulose (MCC) is a purified, partially depolymerized cellulose derivative obtained through the hydrolysis of α-cellulose. MCC has been widely applied in the food, cosmetic, and pharmaceutical industries as a stabilizer, anti-caking agent, emulsifier, adsorbent, binder, and emulsion stabilizer (Adeleye et al., 2022). One particularly promising application of MCC is as a drug-carrier matrix in drug delivery systems (DDS) designed for sustained and controlled release (Janvalkar et al., 2025).

In drug delivery applications, modification of microcrystalline cellulose is often intended to enhance its interaction with hydrophobic drugs by incorporating surfactants. One example is saponin, a natural surfactant derived from plants. Saponins are amphiphilic molecules, possessing hydrophilic moieties that can associate with the carrier matrix and hydrophobic moieties that can interact with poorly water-soluble drugs (Wijaya et al., 2020). This amphiphilic character makes saponins attractive for improving the loading and delivery performance of MCC toward hydrophobic compounds.

Dexamethasone is a synthetic corticosteroid with potent anti-inflammatory activity and is used in various clinical treatments. However, dexamethasone is also commonly considered a model drug with low water solubility. Developing a dexamethasone drug delivery system is important because, despite its broad medical use (e.g., reducing inflammation and suppressing immune responses), dexamethasone may cause systemic adverse effects such as hyperglycemia, adrenal suppression, osteoporosis, and hypertension. Accordingly, a controlled drug delivery system is expected to help minimize side effects while enabling more targeted and controllable drug release.

METHODS

Materials

Liberica coffee husk was collected from Jati Mulyo Village, Dendang Subdistrict, East Tanjung Jabung Regency, Jambi, Indonesia. Other materials included toluene, ethanol, 4% NaOH, 5% NaOCl, 58% H_2SO_4, phosphate buffer solution (pH 7.4), deionized water, saponin, and dexamethasone.

Instruments

The instruments used were an oven, analytical balance, volumetric pipettes, a 100-mesh sieve, mortar and pestle, standard glassware, a glass stirring rod, funnel, hammer mill, Soxhlet extraction apparatus, filter paper, centrifuge, ultrasonic bath/sonicator, magnetic stirrer, hot plate, shaking water bath, Fourier Transform Infrared (FTIR) spectrometer, Scanning Electron Microscope (SEM), Particle Size Analyzer (PSA), and a UV–Vis spectrophotometer.

Preparation of Liberica Coffee Husk

The collected Liberica coffee husk was separated from coffee beans, washed under running water, and sun-dried for 2 days. The dried husk was milled using a hammer mill and sieved through a 100-mesh sieve.

Cellulose Isolation from Liberica Coffee Husk

Cellulose isolation consisted of **dewaxing**, **delignification**, and **bleaching**.

Dewaxing (Soxhlet extraction)

A 50 g sample was extracted using a Soxhlet apparatus with a toluene: ethanol solvent mixture (2:1, v/v) at 85°C for 6 h. The residue was dried in an oven at 60°C for 4 h, weighed, and the yield was calculated.

Delignification

The dewaxed sample was treated with 4% NaOH at a solid-to-liquid ratio of 1:10 (w/v) and heated at 60–80°C for 2 h. The mixture was cooled, filtered, and washed with deionized water until the pH was neutral. The resulting cellulose extract was dried at 60°C for 2–3 h, then gently ground using a mortar and pestle to prevent agglomeration.

Bleaching

The delignified cellulose was treated with 5% NaOCl at a 1:10 (w/v) ratio in a water bath at 50°C for 2–3 h, with continuous stirring. After cooling, the sample was filtered and washed until the pH was neutral. The obtained solid was dried at 60°C for 3 h, and the yield was calculated.

Characterization of Isolated Cellulose

The functional groups of the isolated cellulose were analyzed by FTIR. Surface morphology was examined using SEM.

Hydrolysis to Produce Microcrystalline Cellulose (MCC)

One gram of Liberica coffee husk cellulose was hydrolyzed in 20 mL of 58% H_2SO_4 under constant stirring for 1 h. The reaction was quenched by adding deionized water at eight times the total solution volume, then left overnight to form a suspension. The mixture was centrifuged at 4000 rpm for 15 min and washed with deionized water until neutral pH. The MCC suspension was then sonicated for 60 min. Particle size was measured using PSA, and functional groups were analyzed by FTIR.

Saponin Modification of MCC

Saponin (5 mg) was dissolved in 10 mL deionized water and added to a beaker containing 50 mg MCC. The mixture was heated at 50°C for 1 h while continuously stirred at 200 rpm. MCC–saponin was separated by centrifugation, dried, and characterized by FTIR.

Preparation of Dexamethasone Standard Solution and Calibration Curve

A dexamethasone stock solution (1000 ppm) was prepared and diluted to obtain standard solutions at 20, 40, 60, 80, and 100 ppm. Absorbance was measured using UV–Vis spectrophotometry at 240 nm to construct the calibration curve.

Dexamethasone Drug Loading

Drug loading into MCC and MCC–saponin was performed using an initial dexamethasone concentration of 100 ppm. Then, 25 mg of MCC or MCC–saponin was added to the dexamethasone solution and sonicated for 2 h. The mixture was incubated in a water bath at 30°C for 24 h. Drug-loaded particles were separated by centrifugation and dried at room temperature. The remaining dexamethasone concentration in the supernatant was determined by UV–Vis at 240 nm.

Loading efficiency (ε): $\varepsilon = \left(\frac{Co-Ce}{Co}\right) \times 100\%$ (1)

Loading capacity (Q): $\left(\frac{Co-Ce}{m}\right) \times V$ ..... (2)

where:

C_0 = initial dexamethasone concentration (mg/L)
Ce = equilibrium/final dexamethasone concentration (mg/L)
V = solution volume (L)
m = mass of carrier (g)

In Vitro Drug Release Study

Dexamethasone release from drug-loaded MCC–saponin was evaluated by immersing the sample in 20 mL phosphate buffer (pH 7.4). The release medium was stirred using a magnetic stirrer at 37°C. Aliquots (5 mL) were withdrawn at 30, 60, 120, 180, and 240 min and replaced with 5 mL of fresh phosphate buffer (pH 7.4). The withdrawn samples were centrifuged to separate solids, and the supernatant was analyzed by UV–Vis at 240 nm.

% drug release: $\left(\frac{C_{release}}{C_{\infty}}\right)$ x 100% ..... (3)

where:

$C_{release}$ = drug concentration released at each time point (ppm)

$C\infty$ = concentration corresponding to the loaded drug (ppm)

RESULTS AND DISCUSSION

Isolation of Liberica Coffee Husk Cellulose

The cellulose isolation process from Liberica coffee husk consists of three stages. The first stage is **dewaxing**, which aims to remove extractive compounds from Liberica coffee husk—such as waxes, tannins, sugars, and fatty acids—using polar and nonpolar solvents. The second stage is **delignification**, intended to remove lignin and hemicellulose, which form the outer structural matrix surrounding cellulose. This step is carried out using an alkaline solution (NaOH). The final stage is **bleaching**, which aims to obtain cellulose with higher purity. Bleaching removes undesired colorants. Because the sample typically remains relatively dark after delignification, bleaching is required to eliminate residual lignin and hemicellulose, making the resulting powder lighter in color. The bleaching process uses NaOCl as an oxidizing bleaching agent that can degrade and remove color-causing components such as lignin.

Table 1. Yield of cellulose isolation from Liberica coffee husk

Step	Solvent/Reagent	Yield (%)
Dewaxing	Ethanol:toluene (v/v)	74.42
Delignification	NaOH	16.9
Bleaching	NaOCl	40.15

Acid Hydrolysis of Liberica Coffee Husk Cellulose

Acid hydrolysis is a commonly used method for breaking down cellulose and hemicellulose using mineral acids, such as sulfuric acid (H_2SO_4) and hydrochloric acid (HCl). Liu et al. (2016) reported that H_2SO_4- and HCl-based hydrolysis can produce different behaviors in terms of water dispersibility. Hydrolysis using H_2SO_4 tends to yield hydrolyzed products that are more readily dispersed in water, whereas hydrolysis using HCl results in materials that are less easily dispersed and form suspensions that are prone to flocculation.

Modification of Microcrystalline Cellulose (MCC)

Modification of microcrystalline cellulose (MCC) was conducted to enhance the loading efficiency of dexamethasone into the MCC-based carrier system. In this study, MCC was modified using saponin. Saponins were selected because of their amphiphilic nature (possessing both hydrophilic and hydrophobic moieties). The hydrophobic moiety (sapogenin) can promote the solubilization and dispersibility of

hydrophobic drugs within the MCC matrix, thereby improving drug–carrier interactions and potentially increasing drug loading.

Characterization (FTIR)

The functional groups of (i) cellulose isolated from Liberica coffee husk, (ii) hydrolyzed cellulose (MCC), and (iii) saponin-modified MCC were analyzed using Fourier Transform Infrared (FTIR) spectroscopy. The resulting IR spectra are presented in Figure 1.

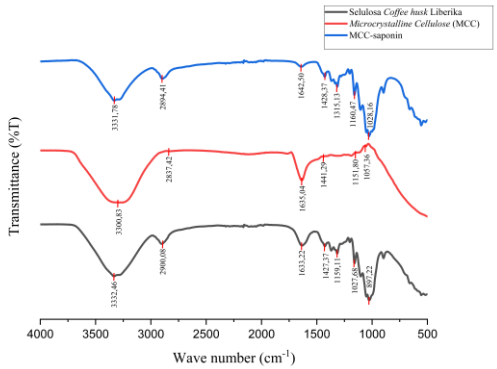

Figure 1. FTIR spectra of Liberica coffee husk cellulose, microcrystalline cellulose (MCC), and saponin-modified MCC (MCC–saponin).

FTIR Analysis

Based on the FTIR spectra, all samples exhibited an absorption band in the range of 3300–3332 cm^{-1}, which corresponds to the O–H stretching vibration. In addition, the bands observed at 2800–2900 cm^{-1} in all samples indicate C–H stretching vibrations of methyl and methylene groups within the cellulose structure. The region of 1000–1200 cm^{-1} is attributed to C–O stretching vibrations, which are characteristic of cellulose. Furthermore, a peak around ~1400 cm^{-1} corresponds to the CH_2 bending vibration of cellulose.

Overall, the FTIR results suggest that the isolation, hydrolysis, and modification processes did not significantly alter the chemical structure of cellulose. However, the MCC spectrum showed a shift to lower wavenumbers (and/or reduced intensity) in the O–H and C–H bands, which is associated with the reduction of amorphous regions due to acid hydrolysis, leaving a higher proportion of crystalline domains (Herawan & Rivani, 2013). The band at 1151.96 cm^{-1} indicates C–O–C ring stretching related to β-1,4-glycosidic linkages (Ratna et al., 2023). The successful modification of MCC with saponin was supported by the appearance of saponin-related functional groups. The oligosaccharide (hydrophilic) region was indicated by the band at 2898.41 cm^{-1} corresponding to C–H stretching, as well as bands at 1315.13 cm^{-1} (CH_2-related vibration) and 1028.16 cm^{-1} (C–O stretching). Meanwhile, the aglycone (hydrophobic) region was reflected in the MCC–saponin spectrum, as indicated by the presence of an olefin/alkene (C=C) band at 1642.50 cm^{-1}.

Scanning Electron Microscopy (SEM)

Scanning Electron Microscopy (SEM) analysis was performed to investigate the surface morphology of cellulose isolated from Liberica coffee husk. The morphology of Liberica coffee husk cellulose is presented in **Figure 2**.

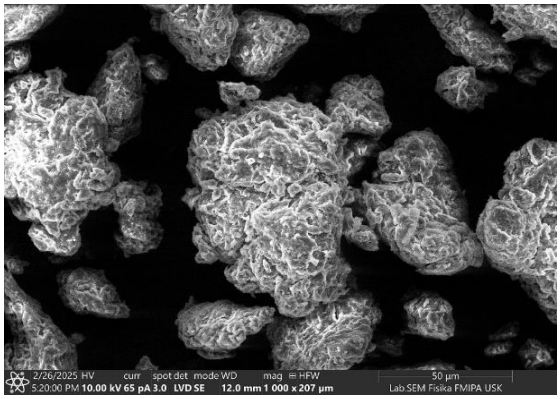

Figure 2. SEM micrographs showing the surface morphology of cellulose isolated from Liberica coffee husk.

SEM Morphology (Liberica Coffee Husk Cellulose)

The morphology of Liberica coffee husk cellulose after the bleaching process indicates the presence of fibrous structures characteristic of cellulose. In several areas, small aggregates were observed, which may represent residual non-cellulosic fractions such as lignin that were not completely

removed. However, these residues were relatively few and small, and thus unlikely to significantly affect the overall morphology. The extracted cellulose surface exhibited an irregular structure, appearing rough and porous.

Particle Size Analyzer (PSA)

Particle Size Analyzer (PSA) was used to determine the particle size distribution of the sample. Particle size is an important parameter in microcrystalline systems, as smaller particles can enhance drug release by increasing the surface area available for interaction with the carrier matrix. Conversely, larger particles generally require a longer diffusion path for the active compound to be released, which may slow the release process (Pathak & Deepak, 2009). Harwansh et al. (2010) reported that microparticle sizes typically range from 0.1–1.0 µm. Based on the PSA results, the produced microcrystalline cellulose had a particle size of 416.6 nm (0.4166 µm) and a polydispersity index (PDI) of 0.313. These results indicate that the MCC particles were relatively uniform and stable, with a low tendency to agglomerate.

Dexamethasone Drug Loading

Dexamethasone drug loading refers to the incorporation of dexamethasone into MCC and MCC–saponin matrices. The loading process was performed by incubating the system for 24 h to facilitate optimal drug–carrier association. The mixture was then centrifuged to separate drug-loaded MCC/MCC–saponin (pellet) from the unbound dexamethasone in the supernatant (filtrate). The resulting pellet was dried at room temperature to remove residual ethanol. The filtrate was analyzed using a UV–Vis spectrophotometer at 240 nm to determine the absorbance and quantify the remaining dexamethasone concentration.

Table 2. Dexamethasone drug loading results

Sample	Loading efficiency (%)	Loading capacity (mg/g)
MCC–saponin	58.59	23.436
MCC	16.91	6.768

Based on Table 2, the loading efficiency of dexamethasone in unmodified MCC was lower than that in saponin-modified MCC (MCC–saponin). This indicates that saponin modification enhances dexamethasone binding and loading within the carrier matrix. The presence of amphiphilic saponin is expected to improve the interfacial compatibility and increase the effective surface area available for drug–carrier interactions. Specifically, the hydrophilic moiety of saponin (glycone) can associate with the MCC matrix, while the hydrophobic moiety (aglycone) can interact with the hydrophobic dexamethasone molecules, thereby promoting higher drug incorporation. The loading capacity of MCC–saponin (23.436 mg/g) was comparable to that reported by Putro et al. (2022), who achieved a dexamethasone loading capacity of 27.54 mg/g using a rarasaponin-modified carrier system.

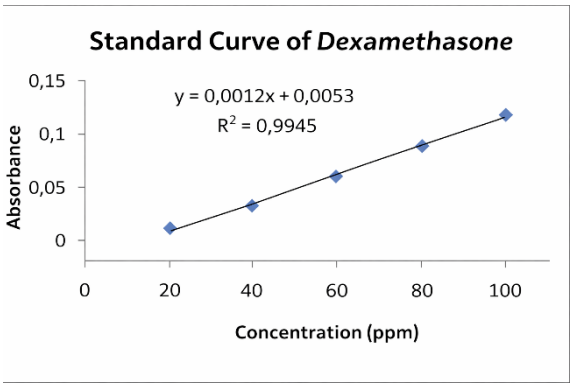

Figure 3. Dexamethasone standard calibration curve.

Dexamethasone Drug Release

The drug release study was conducted to determine the amount of dexamethasone released over time. Dexamethasone release was evaluated by immersing dexamethasone-loaded MCC–saponin and MCC in phosphate buffer solution (pH 7.4). Phosphate buffer at pH 7.4 was selected to simulate the gastrointestinal environment

(large intestine) following oral administration. Aliquots of 5 mL were withdrawn at predetermined time intervals (30, 60, 120, 180, and 240 min). After each sampling, an equal volume of fresh phosphate buffer (pH 7.4) was added to maintain sink conditions and minimize potential drug degradation or accumulation in the release medium. The collected samples were analyzed using a UV–Vis spectrophotometer at 240 nm.

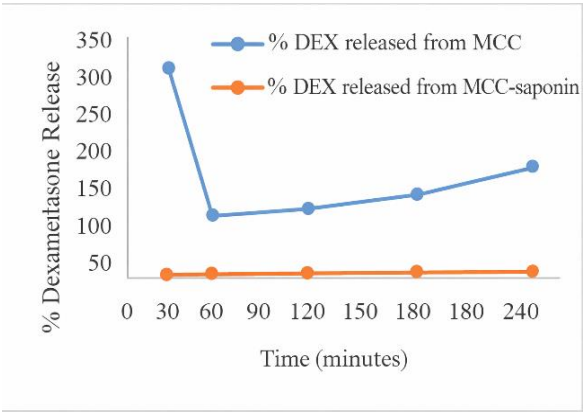

Figure 4. Dexamethasone drug release profile.

Based on the in vitro dexamethasone release results and Figure 4, the percentage of dexamethasone released from the MCC–saponin formulation gradually increased from 60 to 180 min. In contrast, dexamethasone release from unmodified MCC was substantially higher and exceeded 100%, indicating an uncontrolled release behavior. These findings suggest that MCC–saponin shows initial potential as a drug delivery system due to its ability to retain and regulate drug release while also improving dexamethasone loading efficiency. Although the overall release percentage from MCC–saponin was relatively low, its profile was more favorable because the release occurred in a controlled and sustained manner rather than being rapidly depleted at the early stage. A combined pattern of an initial release followed by sustained release is therapeutically important, as the early phase can quickly achieve a therapeutic dose, while the subsequent phase helps maintain therapeutic levels over an extended period (Komane et al., 2018).

CONCLUSION

Hydrolysis of MCC derived from Liberica coffee husk using sulfuric acid produced MCC particles with a size of 416.3 nm (0.4166 µm) and a polydispersity index (PDI) of 0.313. MCC modification using saponin was successfully performed to enhance the loading efficiency of dexamethasone. The in vitro dexamethasone release study indicated that MCC–saponin shows initial potential as a drug delivery system, as the release percentage gradually increased from 60 to 180 min.

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