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Penulis	:	Intan Martha Cahyani, Endang Diyah Ikasari, Novi Tadlkirotulladlat, Novita Sindy Anggraini

Tabel Tahapan Publikasi Artikel

No	Tahapan Publikasi	Tanggal	Halaman
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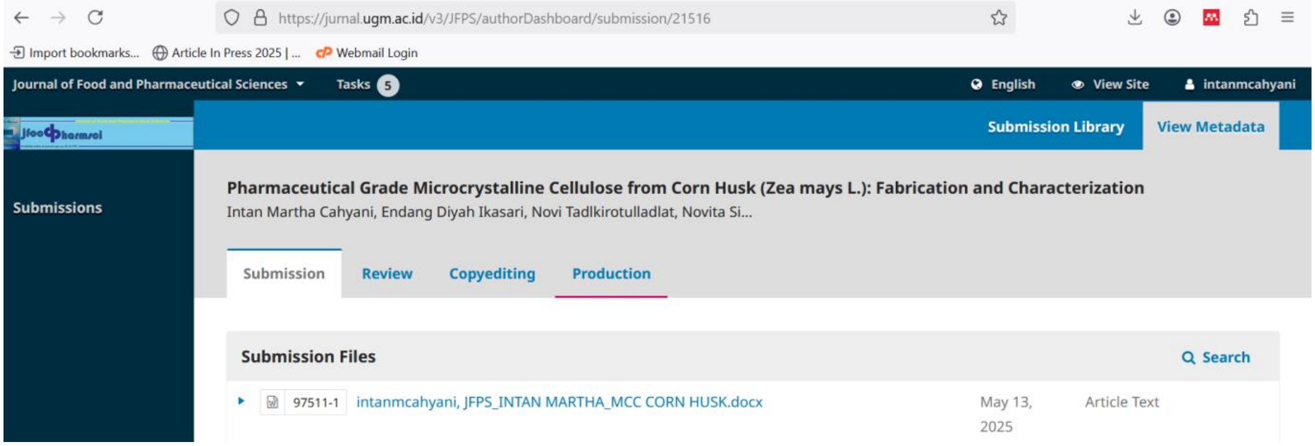
2	Editor Notification: Status Manuscript on Review	14 Mei 2025	Hal. 2
3	Review Discussions: Revisions Required and Comment of Reviewer 1 and 2	15 Mei 2025	Hal. 2
4	Submit Revised Manuscript	23 Mei 2025	Hal. 2
5	Editor Decision**:** Accept Submission (Article Acceptance)	2 Juni 2025	Hal. 3
6	Editor Decision: Notifications Editing is complete and sending it to production.	17 Juni 2025	Hal. 5
7	Check Galley Proof	17 Juni 2025	Hal. 6
8	Confirm Final Proofreading Approval	19 Juni 2025	Hal. 6
9	Published Information by email	30 Juni 2025	Hal. 6
10	Article Published Vol 13 issue 2 (30 Juni 2025) on Website https://jurnal.ugm.ac.id/v3/JFPS/article/view/21516 https://jurnal.ugm.ac.id/v3/JFPS/article/view/21516/5919	30 Juni 2025	Hal. 7
11	Lampiran Bukti Korespondensi	-	Hal 8 - 69

Pengusul

Dr. apt. Intan Martha Cahyani, M.Sc

1. Manuscript Submission

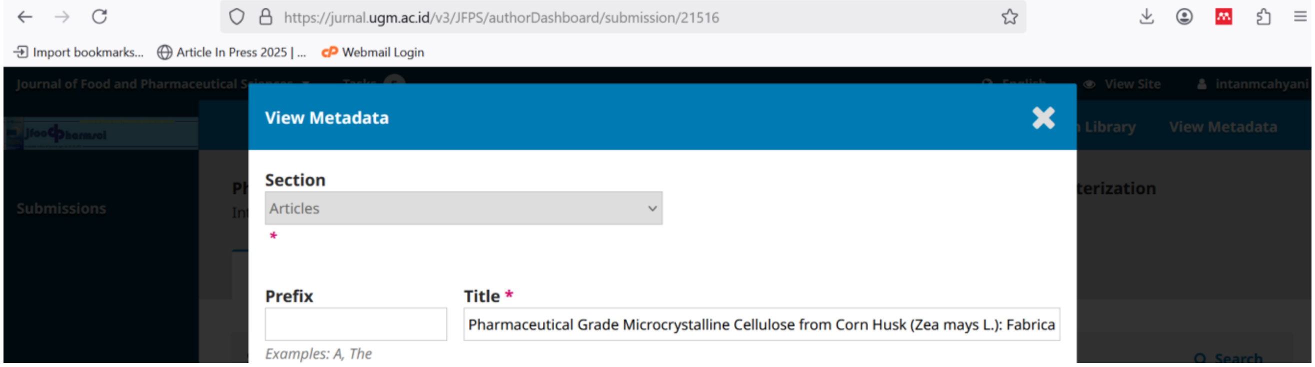


Pharmaceutical Grade Microcrystalline Cellulose from Corn Husk (Zea mays L.): Fabrication and Characterization

Intan Martha Cahyani, Endang Diyah Ikasari, Novi Tadlkirotulladlat, Novita Si...

Submission Files

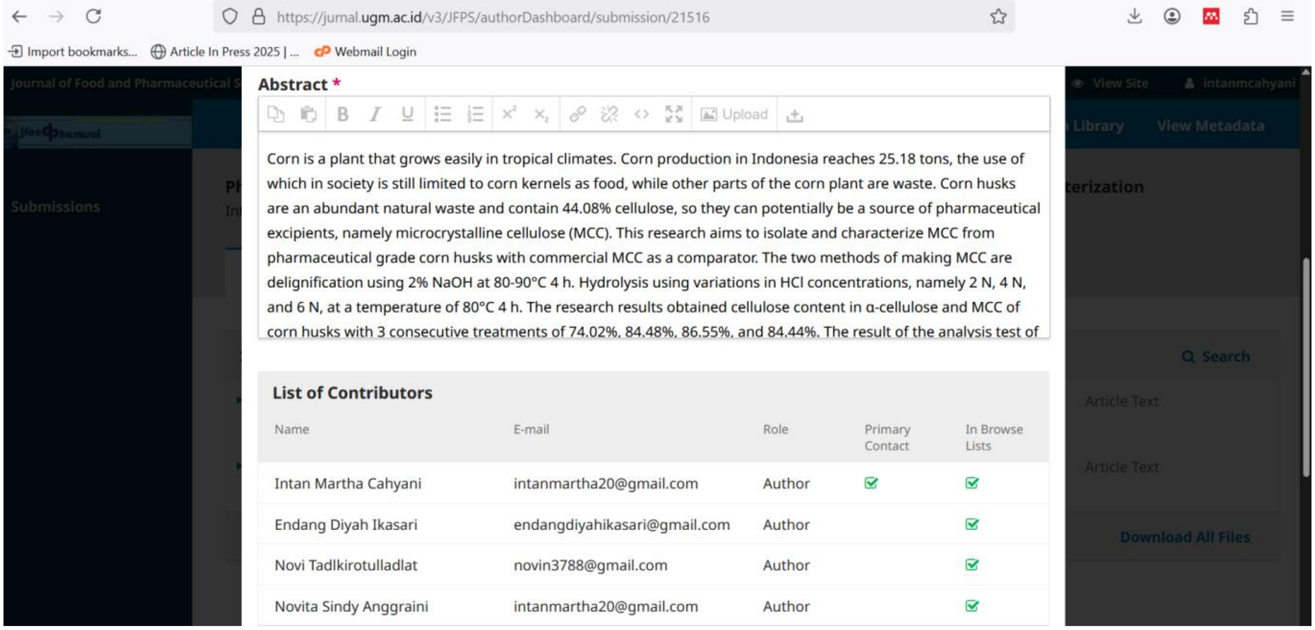
97511-1	intanmcahyani, JFPS_INTAN MARTHA_MCC CORN HUSK.docx	May 13, 2025	Article Text



Section: Articles

Prefix:

Title *: Pharmaceutical Grade Microcrystalline Cellulose from Corn Husk (Zea mays L.): Fabrica



Abstract *

Corn is a plant that grows easily in tropical climates. Corn production in Indonesia reaches 25.18 tons, the use of which in society is still limited to corn kernels as food, while other parts of the corn plant are waste. Corn husks are an abundant natural waste and contain 44.08% cellulose, so they can potentially be a source of pharmaceutical excipients, namely microcrystalline cellulose (MCC). This research aims to isolate and characterize MCC from pharmaceutical grade corn husks with commercial MCC as a comparator. The two methods of making MCC are delignification using 2% NaOH at 80-90°C 4 h. Hydrolysis using variations in HCl concentrations, namely 2 N, 4 N, and 6 N, at a temperature of 80°C 4 h. The research results obtained cellulose content in α-cellulose and MCC of corn husks with 3 consecutive treatments of 74.02%, 84.48%, 86.55%, and 84.44%. The result of the analysis test of

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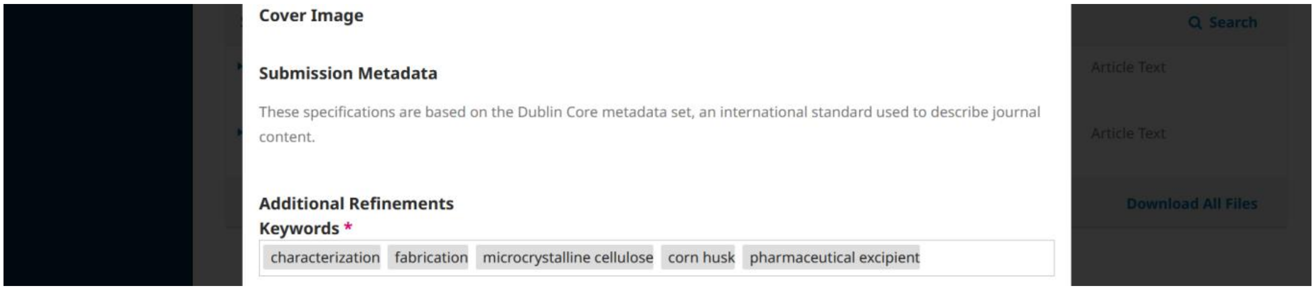

Cover Image

Submission Metadata

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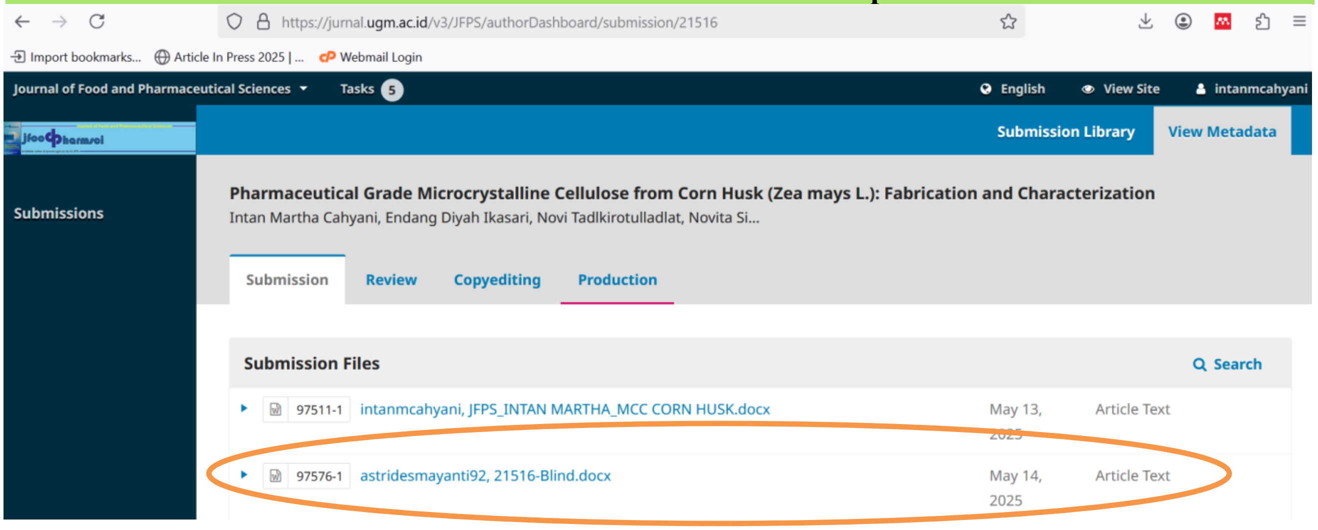
Additional Refinements

Keywords *

characterization | fabrication | microcrystalline cellulose | corn husk | pharmaceutical excipient

2. Editor Notification: Status Manuscript on Review



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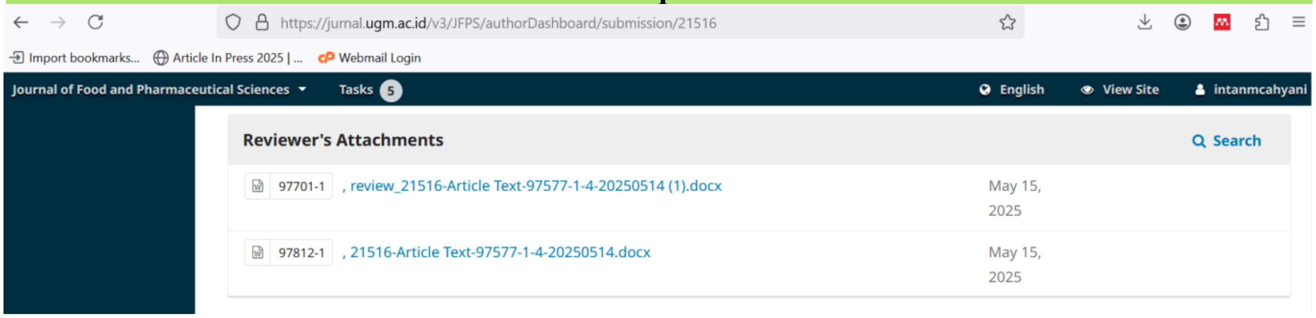
Pharmaceutical Grade Microcrystalline Cellulose from Corn Husk (Zea mays L.): Fabrication and Characterization

Intan Martha Cahyani, Endang Diyah Ikasari, Novi Tadlkirotulladlat, Novita Si...

Submission Files

97511-1	intanmcahyani, JFPS_INTAN MARTHA_MCC CORN HUSK.docx	May 13, 2025	Article Text
97576-1	astridesmayanti92, 21516-Blind.docx	May 14, 2025	Article Text

3. Review Discussions: Revisions Required and Comment of Reviewer 1 and 2



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Reviewer's Attachments

97701-1	, review_21516-Article Text-97577-1-4-20250514 (1).docx	May 15, 2025
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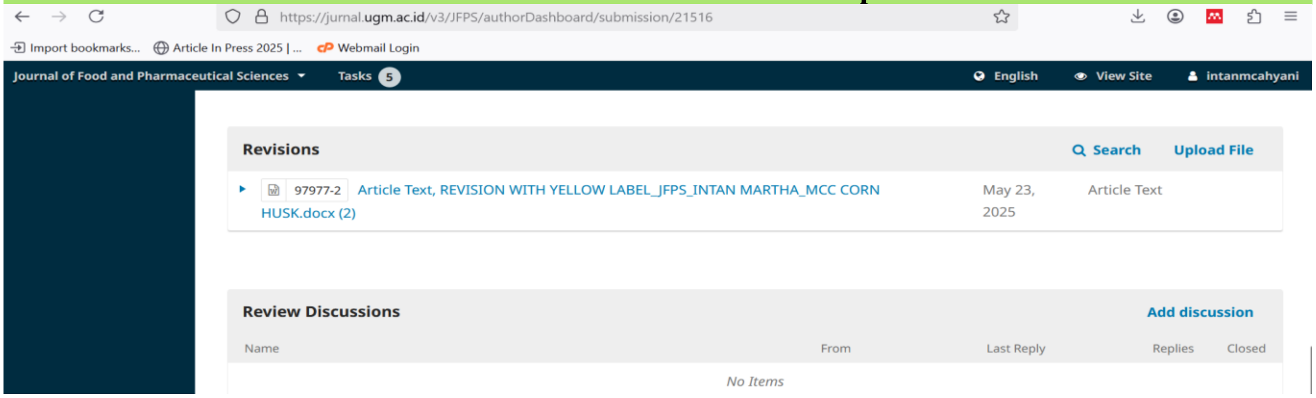
4. Submit Revised Manuscript



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Revisions

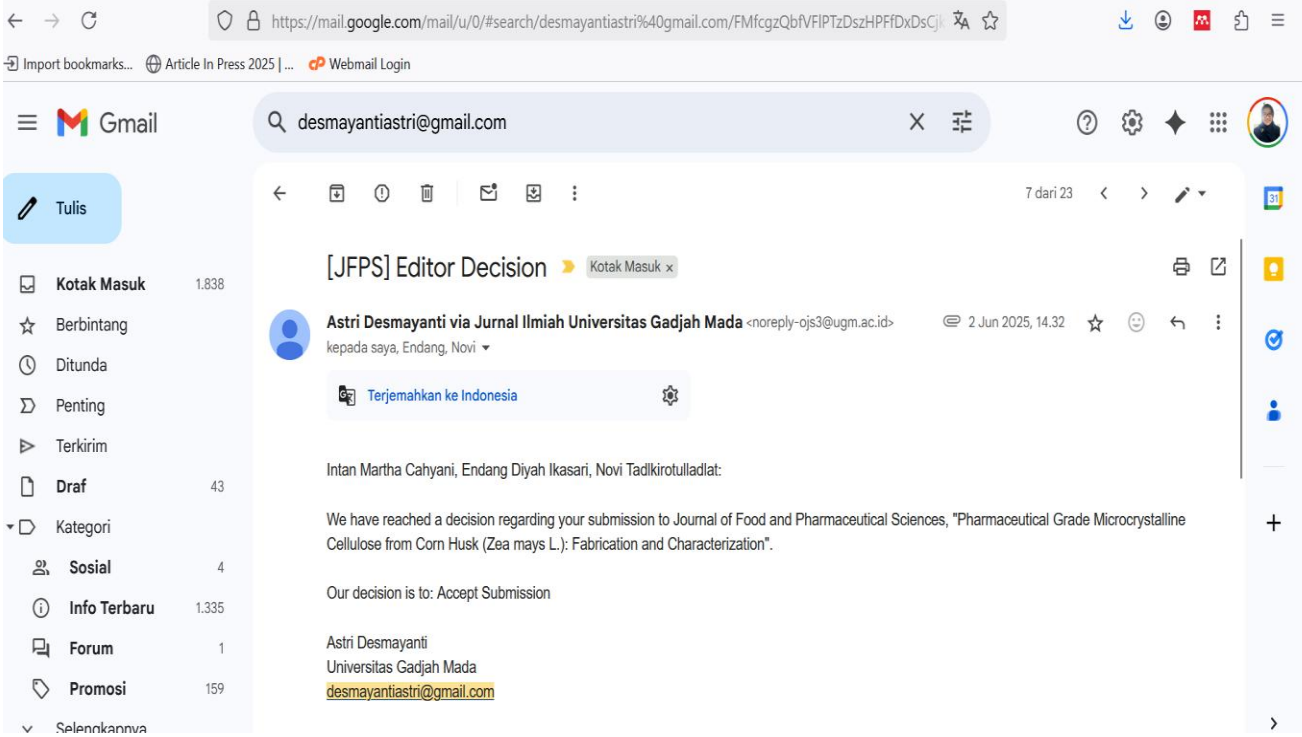
97977-2	Article Text, REVISION WITH YELLOW LABEL_JFPS_INTAN MARTHA_MCC CORN HUSK.docx (2)	May 23, 2025	Article Text

Review Discussions

Name	From	Last Reply	Replies	Closed

No Items

5. Editor Decision: Accept Submission (Article Acceptance)



[JFPS] Editor Decision

Astri Desmayanti via Jurnal Ilmiah Universitas Gadjah Mada <noreply-ojs3@ugm.ac.id> 2 Jun 2025, 14.32

kepada saya, Endang, Novi

Intan Martha Cahyani, Endang Diyah Ikasari, Novi Tadlkirotulladlat:

We have reached a decision regarding your submission to Journal of Food and Pharmaceutical Sciences, "Pharmaceutical Grade Microcrystalline Cellulose from Corn Husk (Zea mays L.): Fabrication and Characterization".

Our decision is to: Accept Submission

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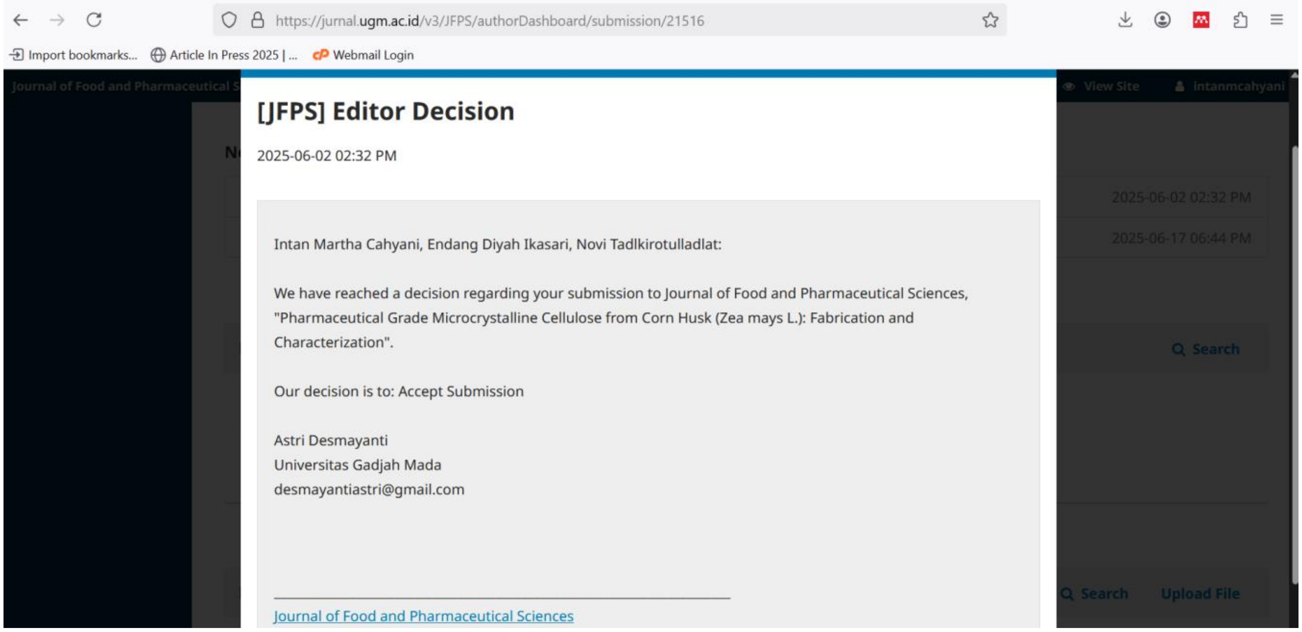

[JFPS] Editor Decision

2025-06-02 02:32 PM

Intan Martha Cahyani, Endang Diyah Ikasari, Novi Tadlkirotulladlat:

We have reached a decision regarding your submission to Journal of Food and Pharmaceutical Sciences, "Pharmaceutical Grade Microcrystalline Cellulose from Corn Husk (Zea mays L.): Fabrication and Characterization".

Our decision is to: Accept Submission

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Journal of Food and Pharmaceutical Sciences

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2 June 2025

Dear : Intan Martha Cahyani

ACCEPTANCE LETTER

Journal of Food and Pharmaceutical Research (JFPS), is pleased to inform you that the following manuscript has been accepted for publication in JFPS volume 13 issue 2 on June 2025.

Manuscript Title	: Pharmaceutical Grade Microcrystalline Cellulose from Corn Husk (Zea mays L.): Fabrication and Characterization
Authors	: Intan Martha Cahyani, Endang Diyah Ikasari, Novi Tadlkirotulladlat, and Novita Sindy Anggraini

We thank you for your fine contribution to the Journal of Food and Pharmaceutical Sciences and encourage you to submit other articles to the journal.

Your sincerely,

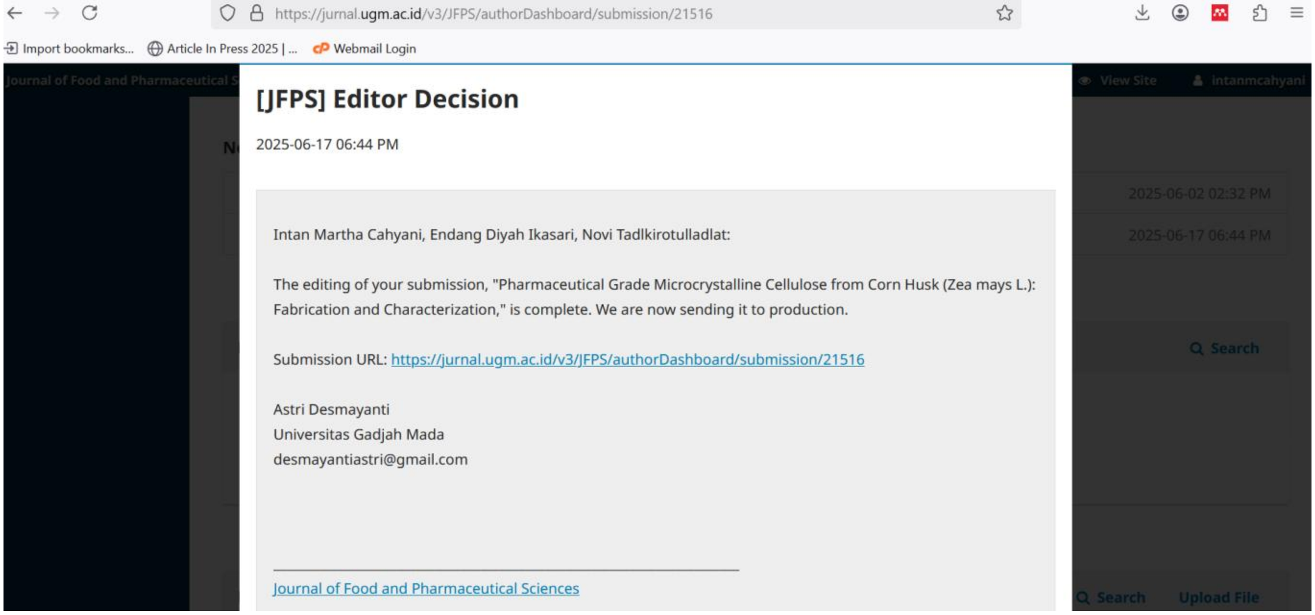
Prof. Dr. Abdul Rohman

Chief Editor

Journal of Food and Pharmaceutical Sciences

jfoodpharmsci.gama@gmail.com https://jurnal.ugm.ac.id/v3/JFPS

6. Editor Decision: Notifications Editing is Complete and Sending it to Production.



[JFPS] Editor Decision

2025-06-17 06:44 PM

Intan Martha Cahyani, Endang Diyah Ikasari, Novi Tadlkirotulladlat:

The editing of your submission, "Pharmaceutical Grade Microcrystalline Cellulose from Corn Husk (Zea mays L.): Fabrication and Characterization," is complete. We are now sending it to production.

Submission URL: https://jurnal.ugm.ac.id/v3/JFPS/authorDashboard/submission/21516

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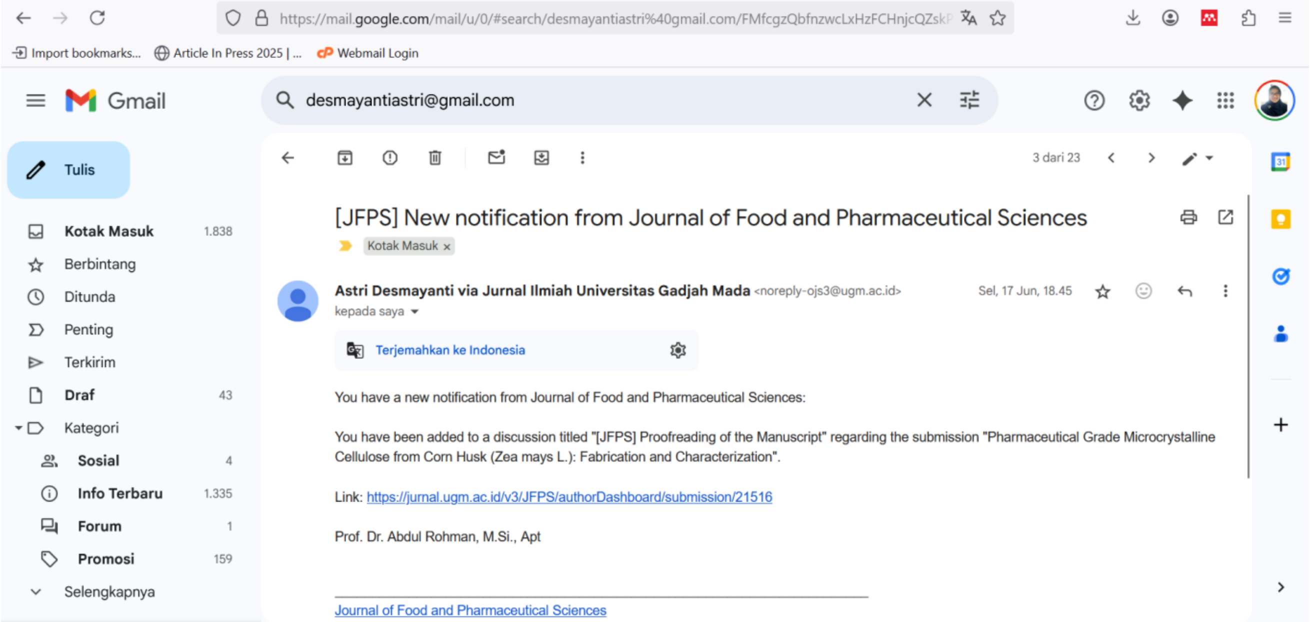
Journal of Food and Pharmaceutical Sciences



[JFPS] New notification from Journal of Food and Pharmaceutical Sciences

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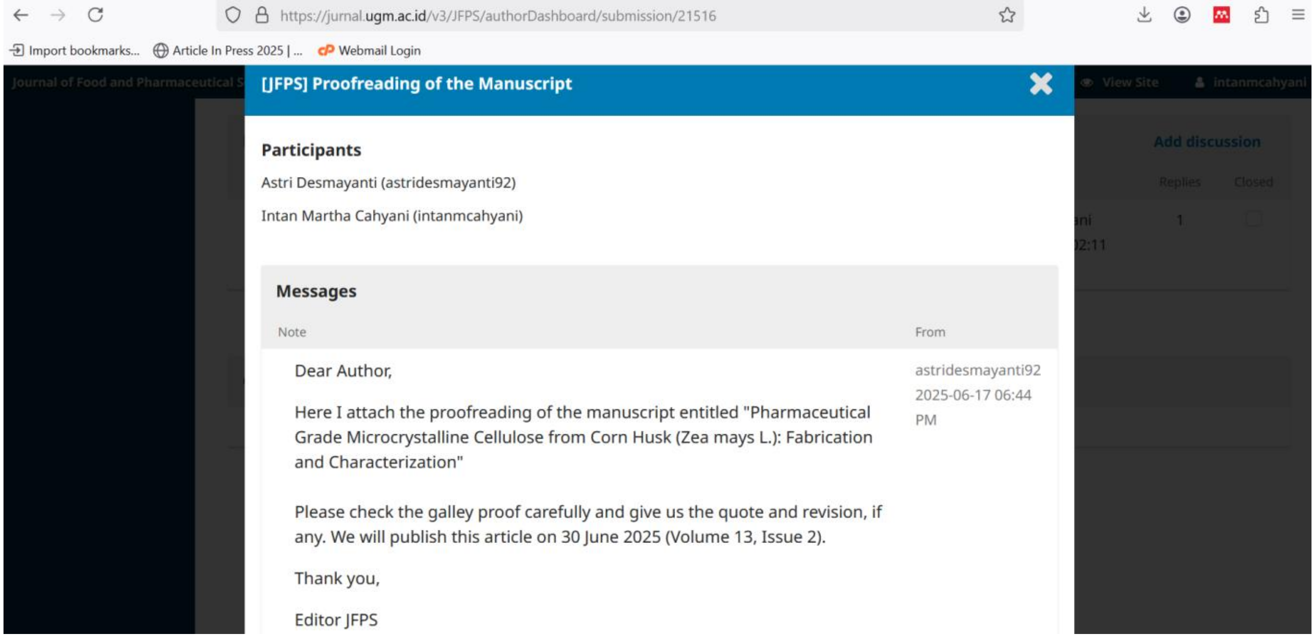
You have been added to a discussion titled "[JFPS] Proofreading of the Manuscript" regarding the submission "Pharmaceutical Grade Microcrystalline Cellulose from Corn Husk (Zea mays L.): Fabrication and Characterization".

Link: https://jurnal.ugm.ac.id/v3/JFPS/authorDashboard/submission/21516

Prof. Dr. Abdul Rohman, M.Si., Apt

Journal of Food and Pharmaceutical Sciences

7. Check Galley Proof



[JFPS] Proofreading of the Manuscript

Participants

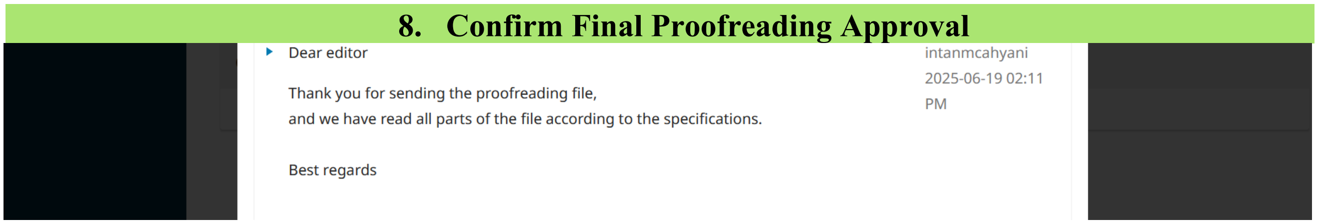
Astri Desmayanti (astridesmayanti92)

Intan Martha Cahyani (intanmcahyani)

Messages

Note	From
Dear Author, Here I attach the proofreading of the manuscript entitled "Pharmaceutical Grade Microcrystalline Cellulose from Corn Husk (Zea mays L.): Fabrication and Characterization" Please check the galley proof carefully and give us the quote and revision, if any. We will publish this article on 30 June 2025 (Volume 13, Issue 2). Thank you, Editor JFPS	astridesmayanti92 2025-06-17 06:44 PM

8. Confirm Final Proofreading Approval



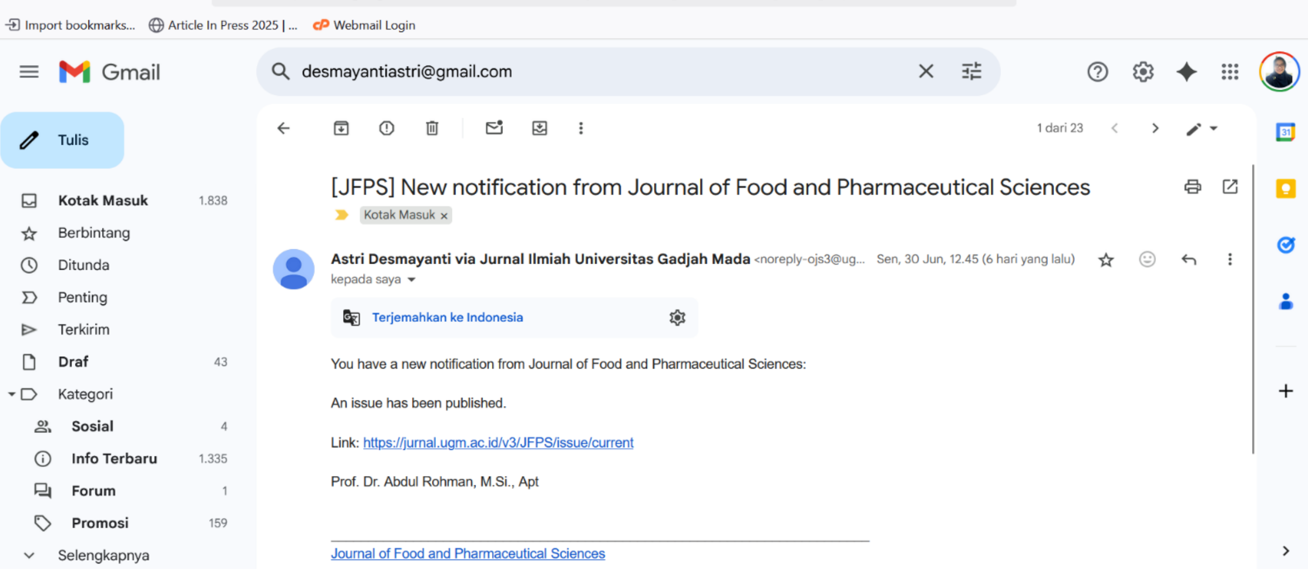
Dear editor

Thank you for sending the proofreading file,
and we have read all parts of the file according to the specifications.

Best regards

intanmcahyani
2025-06-19 02:11 PM

9. Published Information by email



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Journal of Food and Pharmaceutical Sciences

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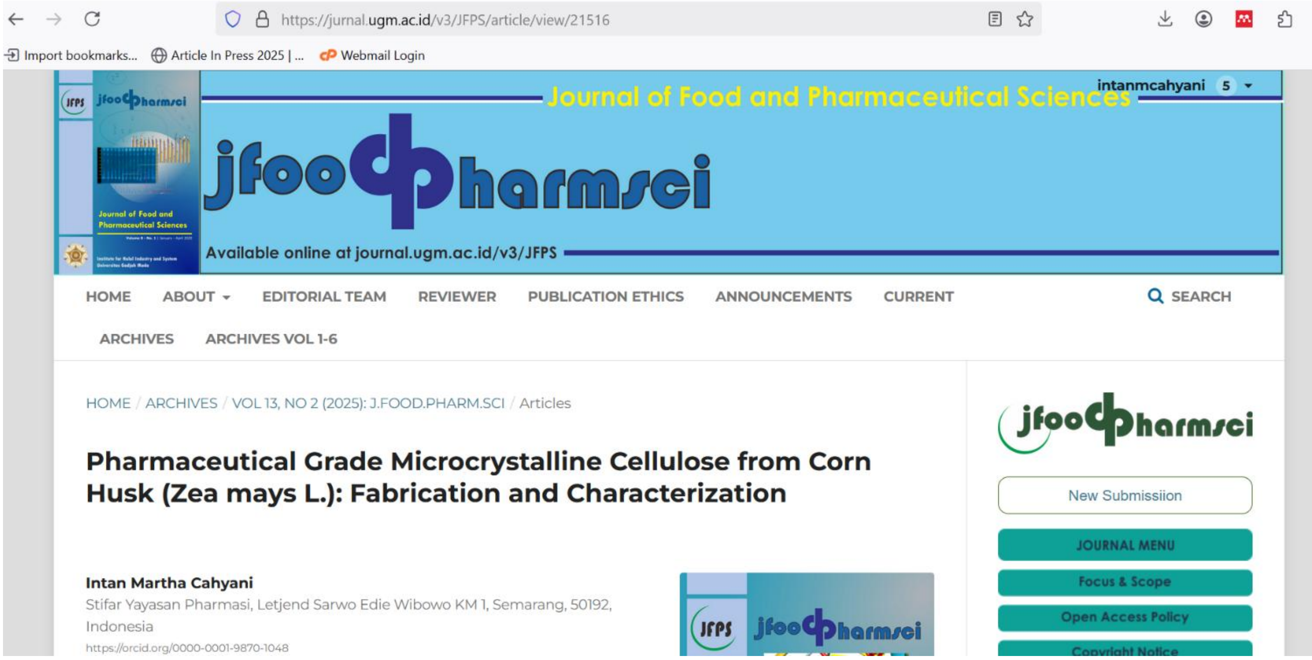

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Pharmaceutical Grade Microcrystalline Cellulose from Corn Husk (Zea mays L.): Fabrication and Characterization

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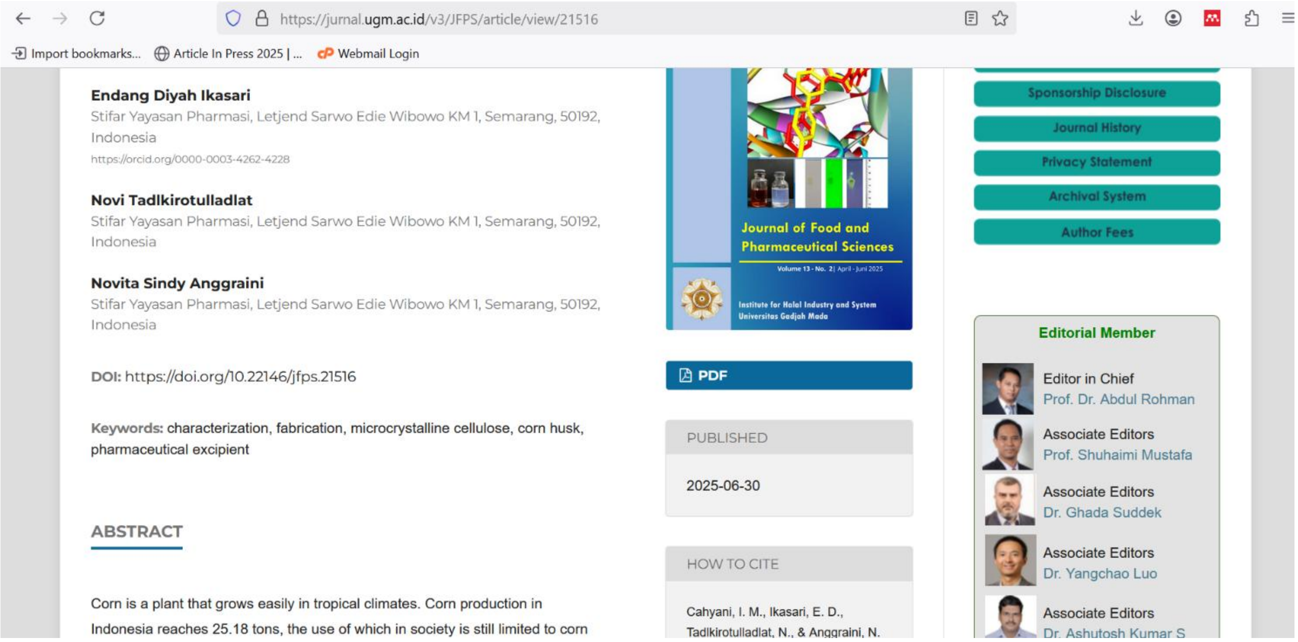

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Keywords: characterization, fabrication, microcrystalline cellulose, corn husk, pharmaceutical excipient

ABSTRACT

Corn is a plant that grows easily in tropical climates. Corn production in Indonesia reaches 25.18 tons, the use of which in society is still limited to corn

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Original Article

Pharmaceutical Grade Microcrystalline Cellulose from Corn Husk (*Zea mays* L.): Fabrication and Characterization

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Abstract: Corn is a plant that grows easily in tropical climates. Corn production in Indonesia reaches 25.18 tons, the use of which in society is still limited to corn kernels as food, while other parts of the corn plant are waste. Corn husks are an abundant natural waste and contain 44.08% cellulose, so they can potentially be a source of pharmaceutical excipients, namely microcrystalline cellulose (MCC). This research aims to isolate and characterize MCC from pharmaceutical grade corn husks with commercial MCC as a comparator. The two methods of making MCC are delignification using 2% NaOH at 80-90°C 4 h. Hydrolysis using variations in HCl concentrations, namely 2 N, 4 N, and 6 N, at a temperature of 80°C 4 h. The research results obtained cellulose content in α-cellulose and MCC of corn husks with 3 consecutive treatments of 74.02%, 84.48%, 86.55%, and 84.44%. The result of the analysis test of FTIR, SEM, XRD, and PSA instruments indicate that corn husk MCC has characteristics of commercial MCC as a standard. The resulting corn husk MCC has physicochemical characteristics according to standards that can be used as a pharmaceutical excipient.

Keywords: characterization, fabrication, microcrystalline cellulose, corn husk, pharmaceutical excipient.

1. INTRODUCTION

Corn plants are a staple food that is widely consumed after rice [1]. Corn kernels used in the food sector are only able to represent 5% of the total part of the corn plant; the remaining 95% of the corn plant is in the category of natural waste in the form of stalks, leaves, cobs, and corn husks [2]. The Pharmaceutical Industry in Indonesia still uses 95% of drug raw materials imported from abroad. Corn husks are part of the corn crop waste that has not been utilized optimally and contain quite high cellulose, which is 44.08% [3]. The high cellulose content in corn husks has the potential to be used as pharmaceutical excipient [4].

MCC is pure cellulose that has been isolated using mineral acids from α-cellulose fibrous plants. MCC is widely used as the best excipient in the manufacture of direct printed tablets. In the manufacture of tablets using of direct compression method, MCC is used as a dry binder, tablet disintegrant, filler, or thinner, absorbent, lubricant, and anti-adherent. MCC is widely used as an excipient in the manufacture of direct print tablets because it has good flow properties and compatibility [5,6].

MCC can be made by delignification and then hydrolysis. Delignification is carried out to change the structure of lignocellulose biomass, which aims to degrade lignin polymers bound to cellulose, then lignin will dissolve in water, and the result is α-cellulose. The deignified α-cellulose powder was subjected to controlled hydrolysis using an acidic solution. Acid hydrolysis can damage the amorphous region of the cellulose microfibrils, where the amorphous form will undergo disconnection and then leave a crystalline [7,8]. Several studies on the use of HCl in hydrolysis in the manufacture of MCC from natural materials have been able to increase the yield and crystallinity index [9–12].

Therefore, this study conducted further research with variations in HCl concentration in hydrolysis to produce corn husk MCC that have physicochemical characteristics in accordance with pharmaceutical grade standards and test the physicochemical characteristics of corn husk MCC compared to commercial MCC as a standard.

2. MATERIALS AND METHODS

2.1. Materials

Corn husks from plantation waste in the Semarang area, NaOH 2%, NaOCl 5%, HCl, and aquadest. Materials for testing the physicochemical characteristics of MCC: Avicel PH 102, H_2SO_4 1N, H_2SO_4 72%, ethanol, iodized zinc chloride solution (zinc chloride, potassium iodide and iodine) and iodine 0.05 M.

2.2. Methods

2.2.1. Fabrication of MCC Corn Husk

a. Alkaline Delignification

The corn husks are sorted wet, washed clean, and dried for 2 days in the sun. The dried corn husks are then mashed and sifted using a mesh no. 40. The corn husk powder is deignified with a 2% NaOH solution at 80-90°C for 4 h, the residue is filtered and washed down to a neutral pH of 6-7. The next stage is bleaching with a solution of NaOCl 5% at 70°C for 1 hour and NaOCl 5% for 24 h at room temperature. The residue is filtered and washed to a neutral pH of 6-7. Cellulose is produced, dried, and mashed [13].

b. Acid Hydrolysis

The α-cellulose sample was hydrolyzed with a variation in HCl concentrations of 2 N, 4 N, and 6 N for 80°C 4 hours and then filtered and washed until a neutral pH of 6-7. The next stage is bleached 2 times with a 5% NaOCl solution of 70°C for 1 hour and soaked in the same solution for 24 hours at room temperature. The residue is filtered and washed until a neutral pH of 6-7. MCC is dried and smoothed then sifted mesh no. 60 [14].

2.2.2 Physicochemical Characterization of MCC Corn Husk

a. Determination of Cellulose Concentration

The precentage of cellulose concentration was determinated using the Che.sson-datta method [15].

b. Moisture Content

Determined using a moisture content tool set at a temperature of 150 °C for automatic time to constant weight. The standar requirement for MCC moisture content was not more than 5% [16].

c. pH

MCC corn husks as much as 1 gram added 50 mL aquadest stirring for 5 minutes then measured the pH using a pH instrument [17].

d. Melting Point

MCC is inserted into a capillary pipe and then put into a melting point device (Mettler Toledo) with a temperature of 200°C when the device is switched on and the temperature is deformed when the solids begin to melt.

e. Flow Rate and Angle of Repose

The flow rate of MCC corn husks using a flowability tester (Erweka GT) with a funnel diameter of 15 mm. The cover at the bottom of the funnel is opened and the flow speed is calculated at the time the granule starts flowing until the granule stops flowing using a stopwatch and then the time obtained and the height and diameter of the cone are measured [18].

f. Density, Carr's Index and Hausner Ratio

40 grams of corn husk MCC is placed in a 100 mL measuring cup. The surface of the powder is carefully leveled without being compressed its volume (Vo) measurement is performed. A measuring cup is installed on the support of the tapped density tester, 10, 500, and 1250 taps are carried out and V10, V500, and V1250 are read on the nearest measuring cup unit. Volume measured to the last tap (Vt) [19]. The density of MCC corn husks was determined by dividing weight by Vo (bulk density) and Vt (tapped density). The true density of MCC is determined by determining the volume using a picnometre. Carr's index and hausner ratio indices were calculated from the results of the bulk and tapped density that had been calculated.

2.2.3. Fourier Transformed Infrared (FTIR)

Fourier Transform Infrared Spectroscopy (FTIR) testing of microcrystalline cellulose from corn husks was used to determine the functional groups of the corn husk MCC using Agilent Technologies Cary 630 FTIR.

2.2.4. Scanning Electrone Microscope (SEM)

Scanning Electron Microscope-Energy Dispersive X-Ray (SEM-EDX) MCC testing of corn husks was used to determine the morphological shape as well as to analyze the elements contained in the sample using the Scanning Electron Microscope-Energy Dispersive X-Ray microscope (JEOLJSM-6510LA).

2.2.5. X-Ray Diffraction (XRD)

X-Ray Diffraction (XRD) analysis of MCC from corn husks was used to determine the crystallinity index produced by corn husk MCC using X-Ray Diffraction (D8 ADVANC X-Ray Diffraction) [20].

2.2.6. Particle Size Analyzer (PSA)

Particle Size Analyzer (PSA) is used to determine the particle size distribution of corn husk MCC using the Particle Size Analyzer tool (Malvern® Mastersizer 3000 (Malvern instrument UK) ([21].

3. RESULTS AND DISCUSSION

3.1. Physicochemical Characterization of MCC Corn Husk

The results of determining cellulose content using the Chasson-datta method were obtained from the average cellulose content of corn husk powder of 42.90%, the results obtained were close to the literature that corn husks have a cellulose content of 44.08% [22]. The yield of cellulose content in corn husk α-cellulose increased by 74.02% due to alkalization treatment with NaOH which caused the loss of lignin, mainly due to the unstable ester bond between cellulose and lignin complex, so that lignin that loosely binds to alkali to form a water-soluble alkaline lignin complex. NaOH can break the bond between cellulose with hemicellulose and lignin, causing changes in cellulose levels to increase [13]. The result of MCC corn husks was 84.48% HCl 2 N and MCC 4 N was 86.55%. The result of the concentration of MCC 6 N of cellulose content decreased by 84.44% (Table 1). The decrease in cellulose levels that occur is caused by the higher concentration of HCl causing an increase in heat (heat) causing the cellulose structure to open up so that cellulose molecules are dispersed freely in the solution, this freely dispersed cellulose structure results in the presence of dissolved cellulose carried away in the solution when the filtration process is carried out [23].

Table 1. Physical Chemical Characterization Test of MCC Corn Husk

Type of Assay		**Result**				**Limit Requirements**
		MCC Corn Husk 2 N	**MCC Corn Husk 4 N**	**MCC Corn Husk 6 N**	**MCC Commercial**	
Determination of Cellulose Levels (%)		84.48±2.99	86.55±0.91	84.44±2.34	80.81±1.14	80.81
Moisture Content (%)		5.82±0.41	5.66±0.29	3.33±0.93	4.93±0.11	<5
pH		6±0	6±0	6±0	6±0	5-7,5
Melting Point (°C)		299.67±0.58	299.67±0.58	270.66±0.58	315.33±0.58	260-270
Flow rate (g/s)		19.87±3.16	27.66±3.30	31.20±5.12	29.104±3.32	1.41
Angle of Repose (°)		29.59±1.01	28.45±1.12	25.98±3.14	45.27±1.22	34.4-49
Bulk Density (g/mL)		0.341±0.02	0.397±0.01	0.617±0.13	0.371±0.01	0.337 g/cm^3
Tapped Density (g/mL)		0.460±0.005	0.532±0.03	0.751±0.13	0.457±0.002	0.478 g/cm^3
True density (g/mL)		1.401±0.05	1.399±0.03	1.512±0.08	1.466±0.04	1.512-1.668 g/cm^3
Hausner Ratio		1.35±0.07	1.34±0.14	1.22±0.06	1.23±0.02	1.00-1.11 = Very Good
Carr's Index (%)		25.98±3.57	25.15±7.79	17.99±3.74	18.67±1.53	1-10 = Very Good
Levels (%)	Chlor	0.15	0.25	0.35	0.10	0.10%; 0.36% & 0.24%
	Calcium	0.26	0.49	0.92	0.36	
	Natrium	0.13	0.09	0.13	0.24	
CrI (%)		34.1	34.7	34.3	34.5	34,5%
Particle size (μm)		362	362	395	332	20-200 μm

The results of the MCC moisture content test from corn husks (Table 1.). The MCC samples treated with 2 N and 4 N HCl showed moisture content values close to that of commercial MCC (pH 102), which has a reference moisture content of 5.37%. The moisture content of MCC treated with 6 N HCl aligns with literature values, which are typically below 5%. If the moisture content is relatively high, it can increase the cohesion between similar particles, causing the powder to lose its ability to flow properly [24]. pH MCC corn husk and the comparator, Avicel PH 102 (Table1.), also exhibited the same pH value of 6, which is consistent with the literature pH range is between 5 and 7.5 [16]. The results of the MCC melting point test from corn husks at each HCl concentration, as well as Avicel PH 102, showed that the powder did not melt but instead changed color to black within the temperature range of 270°C to 400°C. These results do not align with the literature, which states that the melting point should be around 260°C to 270°C [16].

The flow rate of MCC corn husk is better than Avicel PH 102 as a commercial standard (Table 1.), because a good flow rate is indicated by a value greater than 10 g/s. This shows that increasing the concentration of HCl can affect the density and particle size of MCC. Powders with smaller particle sizes tend to have poor flowability due to the larger surface area per unit mass, which increases contact between particles. This greater contact increases cohesive and frictional forces, thus inhibiting the flow of the powder [25]. The results of the angle of repose for MCC corn husk show that, on average, the faster the flow of MCC, the smaller the angle of repose formed. This is believed to be due to the larger particle size and low cohesiveness of the powder, which contribute to its good flow properties. Smaller particle size, higher cohesiveness, and greater frictional forces, thus inhibiting the flow of the powder [26].

The results of bulk and tapping density of MCC corn husks showed that samples treated with 2 N and 4 N HCl, as well as Avicel PH 102, produced values close to the limit requirements (Table 1). MCC corn husks treated with 6 N HCl produced higher values compared to Avicel PH 102 and the literature. The actual density of MCC corn husks treated with 6 N HCl was within the limit requirement range of 1.512–1.668 g/cm³ [16], while MCC treated with 2 N, 4 N HCl, and Avicel PH 102 showed lower actual density values than those reported. The hausner ratio value for MCC corn husks treated with 2 N, 4 N, and 6 N HCl concentrations was comparable to Avicel PH 102. The higher concentration of HCl used in the hydrolysis process had an effect on reducing the carr's index and hausner ratio values. MCC corn husks resulting from hydrolysis with 6 N HCl showed better flow properties and compressibility compared to Avicel PH 102.

3.2. Fourier Transformed Infrared (FTIR)

FTIR Spectra of MCC corn huks (Figure 1.) showed the presence of characteristic cellulose absorption bands. The absorption band at wavelengths of 3500–3250 cm^{-1} indicates the O–H stretching vibration of α-cellulose, while the band at 2970–2850 cm^{-1} corresponds to the C–H stretching vibration, further confirming the presence of α-cellulose [27]. Additionally, the absorption band at 900–800 cm^{-1} indicates the presence of β-glycosidic linkages, which are characteristic of microcrystalline cellulose (MCC) [28].

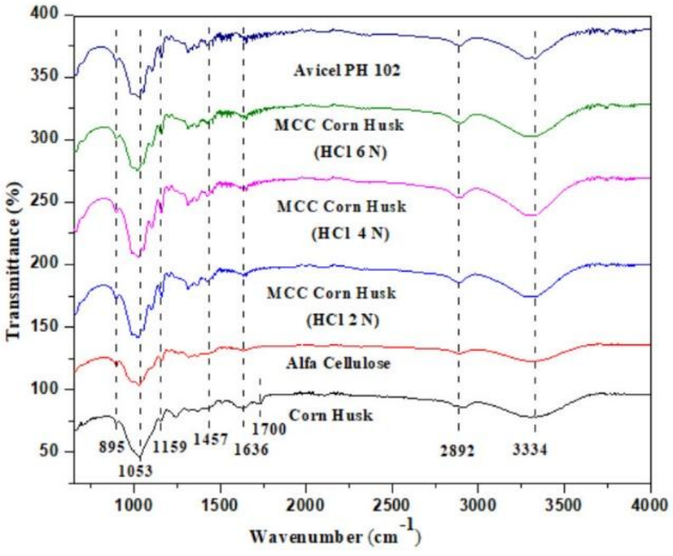

Figure 1. FTIR Spectra of Corn Husk, alfa cellulose, MCC Corn Husk and Avicel PH 102

FTIR analysis also revealed the presence of a lignin absorption band at around 1700 cm^{-1} in raw corn husk powder that had not been treated with NaOH, indicating the presence of lignin prior to the delignification process. In contrast, the FTIR spectra of MCC derived from corn husks treated with HCl concentrations of 2 N, 4 N, and 6 N showed similar spectral patterns to that of Avicel PH 102. These spectra confirmed the presence of cellulose, while the absorption bands associated with hemicellulose and lignin were no longer observed in the MCC samples and Avicel PH 102. This indicates that the non-cellulosic components were effectively removed during the delignification and purification processes, leaving behind primarily α-cellulose [13].

3.3. Scanning Electrone Microscopy (SEM)

The morphological observation of raw corn husk powder revealed a denser surface structure, which is attributed to the presence of lignin still embedded in the cell wall, serving to protect the cellulose. In contrast, the morphology of α-cellulose showed the initial stages of solid peeling, leading to the formation of irregular fibrous structures. MCC derived from corn husks treated with 2 N and 4 N HCl exhibited elongated, stem-like shapes with uneven surfaces, slightly hollow structures, and distinguishable blunt-angled edges (Figure 2.).

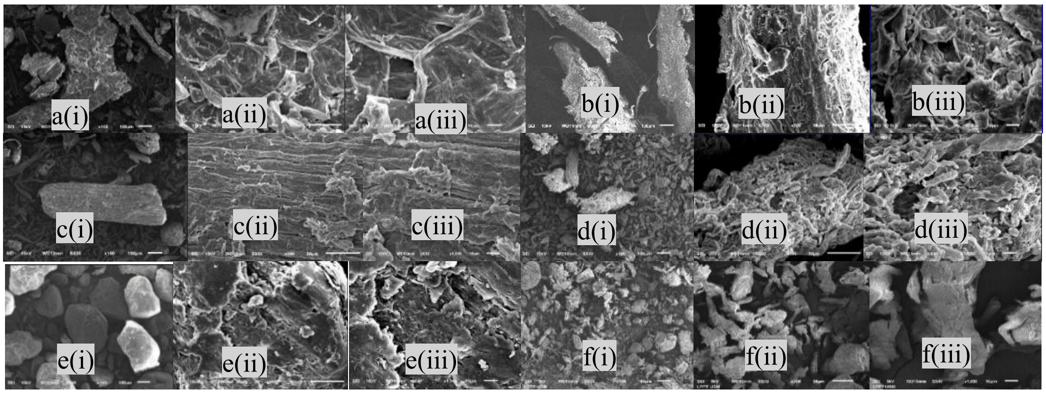

Figure 1. SEM Image of (a) Powder Corn Husk; (b) α-Cellulose; (c) MCC Corn Husk (HCl 2 N); (d) MCC Corn Husk (HCl 4 N); (e) MCC Corn Husk (HCl 6 N); (f) Avicel pH 102; (i) 100x; (ii) 500x; (iii) 1000x.

The comparator, Avicel PH 102, displayed irregularly shaped particles with varying lengths, uneven and slightly hollow surfaces, and both pointed and blunt edges [29]. MCC obtained using 6 N HCl showed a more compact, spherical, and granular morphology compared to MCC 2 N, 4 N, and Avicel PH 102. It also had a smoother surface and blunt angles. Morphology MCC plays an important role in influencing flow properties [9,21,25,30]. In addition to the morphological results of the Scanning Electron Microscope–Energy Dispersive X-Ray (SEM-EDX) analysis, it shows that the chlorine, calcium and sodium levels of corn husk MCC (Figure 3).

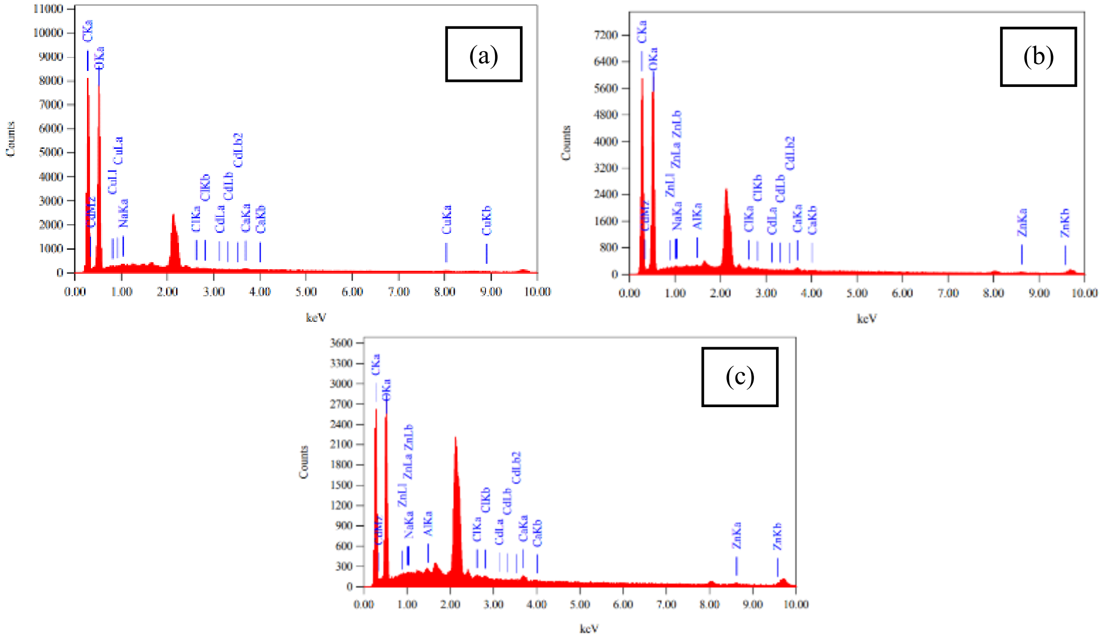

Figure 2. Test SEM-EDX Elements Klor, Calcium, Natrium (a) MCC Corn Husk (HCl 2 N); (b) MCC Corn Husk (HCl 4 N); (c) MCC Corn Husk (HCl 6 N)

3.4. X-Ray Diffraction (XRD)

X-Ray Diffraction (XRD) analysis of MCC corn husks showed that the crystallinity of the samples increased with higher HCl concentrations (Table 1). This increase in crystallinity is attributed to the removal of the amorphous lignin layer from the corn husk samples as evidenced by the absence of peaks at 2θ=24.2° (Figure 4.), which led to a higher cellulose content . However, at a 6 N HCl concentration, the crystallinity index decreased to 34.3%. This reduction is likely due to the high concentration of HCl, which, through the application of heat, caused the crystalline regions of the corn husk MCC to undergo hydrolysis, converting them into amorphous regions and thus reducing the overall crystallinity [31]

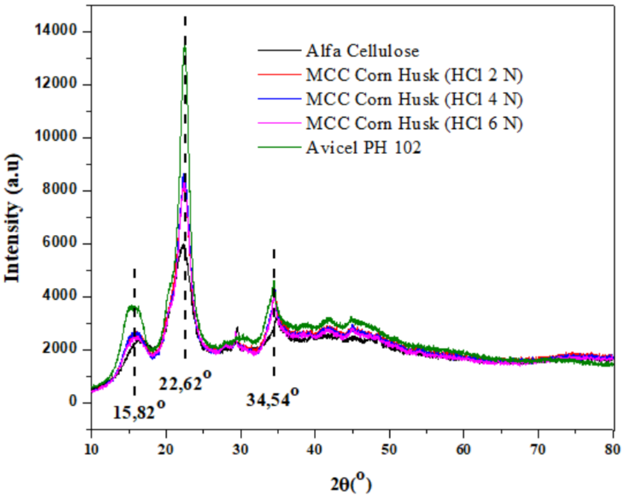

Figure 3. X-ray diffraction patterns of Corn Husk, alfa cellulose, MCC Corn Husk and Avicel PH 102

3.5. Particle Size Analyzer (PSA)

The results of the Particle Size Analysis (Table 2), it can be concluded that larger particle sizes result in better flowability of the MCC derived from corn husks. This improvement in flow

rate is attributed to the stronger gravitational force acting on the larger particles, which outweighs the tensile forces between the powder particles. Additionally, the reduced friction between particles makes it easier for the powder to flow [32].

Table 2. Average Particle Size Analyzer MCC Corn Husk

Sample	Dx (10) (μm)	Dx (50) (μm)	Dx (90) (μm)
Avicel pH 102	37.9	139	332
MCC 1	36.1	144	362
MCC 2	26.5	142	362
MCC 3	84.1	216	395
Mean	46.1	160	363
1xStd Dev	25.8	37.2	25.4
1xRSD (%)	55.8	23.3	7.01

4. CONCLUSION

The results of the physicochemical characterization tests conducted on the three MCC samples showed FTIR absorption patterns similar to that of Avicel PH 102. The surface morphology of the corn husk MCC particles varied with HCl concentration, with the highest concentration (6 N) resulting in round, dense, and more granular particles compared to those obtained at lower concentrations. The crystallinity index of α-cellulose for the three MCC samples was as follows: 30.7% for the raw corn husk, 34.1% for the 2 N HCl-treated sample, 34.7% for the 4 N HCl treated sample, and 34.3% for the 6 N HCl-treated sample. The particle size distribution for the three MCC samples at Dx 90 was 362 μm, 362 μm, 392 μm, and 332 μm, respectively. The difference in HCl concentration during the hydrolysis process contributed to the varying characteristics of the corn husk MCC, with the 6 N HCl concentration producing MCC that met the characteristics required for pharmaceutical-grade MCC.

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Conflicts of interest: The authors declare no conflict of interest.

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BUKTI KORESPONDENSI CATATAN REVIEWER 1

Original Article

Pharmaceutical Grade Microcrystalline Cellulose from Corn Husk (*Zea mays* L.): Fabrication and Characterization

Received: date; Accepted: date; Published: date

Abstract: Corn is a plant that grows easily in tropical climates. Corn production in Indonesia reaches 25.18 tons, the use of which in society is still limited to corn kernels as food, while other parts of the corn plant are waste. Corn husks are an abundant natural waste and contain 44.08% cellulose, so they can potentially be a source of pharmaceutical excipients, namely microcrystalline cellulose (MCC). This research aims to isolate and characterize MCC from pharmaceutical grade corn husks with commercial MCC as a comparator. The two methods of making MCC are delignification using 2% NaOH at 80-90°C 4 h. Hydrolysis using variations in HCl concentrations, namely 2 N, 4 N, and 6 N, at a temperature of 80°C 4 h. The research results obtained cellulose content in α-cellulose and MCC of corn husks with 3 consecutive treatments of 74.02%, 84.48%, 86.55%, and 84.44%. The result of the analysis test of FTIR, SEM, XRD, and PSA instruments indicate that corn husk MCC has characteristics of commercial MCC as a standard. The resulting corn husk MCC has physicochemical characteristics according to standards that can be used as a pharmaceutical excipient.

Keywords: characterization, fabrication, microcrystalline cellulose, corn husk, pharmaceutical excipient.

1. INTRODUCTION

Corn plants are a staple food that is widely consumed after rice [1]. Corn kernels used in the food sector are only able to represent 5% of the total part of the corn plant; the remaining 95% of the corn plant is in the category of natural waste in the form of stalks, leaves, cobs, and corn husks [2]. The Pharmaceutical Industry in Indonesia still uses 95% of drug raw materials imported from abroad. Corn husks are part of the corn crop waste that has not been utilized optimally and contain quite high cellulose, which is 44.08% [3]. The high cellulose content in corn husks has the potential to be used as pharmaceutical excipient [4].

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MCC is pure cellulose that has been isolated using mineral acids from α-cellulose fibrous plants. MCC is widely used as the best excipient in the manufacture of direct printed tablets. In the manufacture of tablets using of direct compression method, MCC is used as a dry binder, tablet disintegrant, filler, or thinner, absorbent, lubricant, and anti-adherent. MCC is widely used as an excipient in the manufacture of direct print tablets because it has good flow properties and compatibility [5,6].

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MCC can be made by delignification and then hydrolysis. Delignification is carried out to change the structure of lignocellulose biomass, which aims to degrade lignin polymers bound to cellulose, then lignin will dissolve in water, and the result is α-cellulose. The deignified α-cellulose powder was subjected to controlled hydrolysis using an acidic solution. Acid hydrolysis can damage the amorphous region of the cellulose microfibrils, where the amorphous form will undergo disconnection and then leave a crystalline [7,8]. Several studies on the use of HCl in hydrolysis in the manufacture of MCC from natural materials have been able to increase the yield and crystallinity index [9–12].

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Therefore, this study conducted further research with variations in HCl concentration in hydrolysis to produce corn husk MCC that have physicochemical characteristics in accordance with pharmaceutical grade standards and test the physicochemical characteristics of corn husk MCC compared to commercial MCC as a standard.

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2. MATERIALS AND METHODS

2.1. Materials

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Corn husks from plantation waste in the Semarang area, NaOH 2%, NaOCl 5%, HCl, and aquadest. Materials for testing the physicochemical characteristics of MCC: Avicel PH 102, H_2SO_4 1N, H_2SO_4 72%, ethanol, iodized zinc chloride solution (zinc chloride, potassium iodide and iodine) and iodine 0.05 M.

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2.2. Methods

2.2.1. Fabrication of MCC Corn Husk

a. Alkaline Delignification

The corn husks are sorted wet, washed clean, and dried for 2 days in the sun. The dried corn husks are then mashed and sifted using a mesh no. 40. The corn husk powder is deignified with a 2% NaOH solution at 80-90°C for 4 h, the residue is filtered and washed down to a neutral pH of 6-7. The next stage is bleaching with a solution of NaOCl 5% at 70°C for 1 hour and NaOCl 5% for 24 h at room temperature. The residue is filtered and washed to a neutral pH of 6-7. Cellulose is produced, dried, and mashed [13].

b. Acid Hydrolysis

The α-cellulose sample was hydrolyzed with a variation in HCl concentrations of 2 N, 4 N, and 6 N for 80°C 4 hours and then filtered and washed until a neutral pH of 6-7. The next stage is bleached 2 times with a 5% NaOCl solution of 70°C for 1 hour and soaked in the same solution for 24 hours at room temperature. The residue is filtered and washed until a neutral pH of 6-7. MCC is dried and smoothed then sifted mesh no. 60 [14].

2.2.2 Physicochemical Characterization of MCC Corn Husk

a. Determination of Cellulose Concentration

The precentage of cellulose concentration was determinated using the Che.sson-datta method [15].

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b. Moisture Content

Determined using a moisture content tool set at a temperature of 150 °C for automatic time to constant weight. The standar requirement for MCC moisture content was not more than 5% [16].

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c. pH

MCC corn husks as much as 1 gram added 50 mL aquadest stirring for 5 minutes then measured the pH using a pH instrument [17].

d. Melting Point

MCC is inserted into a capillary pipe and then put into a melting point device (Mettler Toledo) with a temperature of 200°C when the device is switched on and the temperature is deformed when the solids begin to melt.

e. Flow Rate and Angle of Repose

The flow rate of MCC corn husks using a flowability tester (Erweka GT) with a funnel diameter of 15 mm. The cover at the bottom of the funnel is opened and the flow speed is calculated at the time the granule starts flowing until the granule stops flowing using a stopwatch and then the time obtained and the height and diameter of the cone are measured [18].

f. Density, Carr's Index and Hausner Ratio

40 grams of corn husk MCC is placed in a 100 mL measuring cup. The surface of the powder is carefully leveled without being compressed its volume (Vo) measurement is performed. A measuring cup is installed on the support of the tapped density tester, 10, 500, and 1250 taps are carried out and V10, V500, and V1250 are read on the nearest measuring cup unit. Volume measured to the last tap (Vt) [19]. The density of MCC corn husks was determined by dividing weight by Vo (bulk density) and Vt (tapped density). The true density of MCC is determined by determining the volume using a picnometre. Carr's index and hausner ratio indices were calculated from the results of the bulk and tapped density that had been calculated.

2.2.3. Fourier Transformed Infrared (FTIR)

Fourier Transform Infrared Spectroscopy (FTIR) testing of microcrystalline cellulose from corn husks was used to determine the functional groups of the corn husk MCC using Agilent Technologies Cary 630 FTIR.

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2.2.4. Scanning Electrone Microscope (SEM)

Scanning Electron Microscope-Energy Dispersive X-Ray (SEM-EDX) MCC testing of corn husks was used to determine the morphological shape as well as to analyze the elements contained in the sample using the Scanning Electron Microscope-Energy Dispersive X-Ray microscope (JEOLJSM-6510LA).

2.2.5. X-Ray Diffraction (XRD)

X-Ray Diffraction (XRD) analysis of MCC from corn husks was used to determine the crystallinity index produced by corn husk MCC using X-Ray Diffraction (D8 ADVANC X-Ray Diffraction) [20].

2.2.6. Particle Size Analyzer (PSA)

Particle Size Analyzer (PSA) is used to determine the particle size distribution of corn husk MCC using the Particle Size Analyzer tool (Malvern® Mastersizer 3000 (Malvern instrument UK) ([21].

3. RESULTS AND DISCUSSION

3.1. Physicochemical Characterization of MCC Corn Husk

The results of determining cellulose content using the Chasson-datta method were obtained from the average cellulose content of corn husk powder of 42.90%, the results obtained were close to the literature that corn husks have a cellulose content of 44.08% [22]. The yield of cellulose content in corn husk α-cellulose increased by 74.02% due to alkalization treatment with NaOH which caused the loss of lignin, mainly due to the unstable ester bond between cellulose and lignin complex, so that lignin that loosely binds to alkali to form a water-soluble alkaline lignin complex. NaOH can break the bond between cellulose with hemicellulose and lignin, causing changes in cellulose levels to increase [13]. The result of MCC corn husks was 84.48% HCl 2 N and MCC 4 N was 86.55%. The result of the concentration of MCC 6 N of cellulose content decreased by 84.44% (Table 1). The decrease in cellulose levels that occur is caused by the higher concentration of HCl causing an increase

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in heat (heat) causing the cellulose structure to open up so that cellulose molecules are dispersed freely in the solution, this freely dispersed cellulose structure results in the presence of dissolved cellulose carried away in the solution when the filtration process is carried out [23].

Table 1. Physical Chemical Characterization Test of MCC Corn Husk

Type of Assay		**Result**				**Limit Requirements**
		MCC Corn Husk 2 N	**MCC Corn Husk 4 N**	**MCC Corn Husk 6 N**	**MCC Commercial**	
Determination of Cellulose Levels (%)		84.48±2.99	86.55±0.91	84.44±2.34	80.81±1.14	80.81
Moisture Content (%)		5.82±0.41	5.66±0.29	3.33±0.93	4.93±0.11	<5
pH		6±0	6±0	6±0	6±0	5-7,5
Melting Point (°C)		299.67±0.58	299.67±0.58	270.66±0.58	315.33±0.58	260-270
Flow rate (g/s)		19.87±3.16	27.66±3.30	31.20±5.12	29.104±3.32	1.41
Angle of Repose (°)		29.59±1.01	28.45±1.12	25.98±3.14	45.27±1.22	34.4-49
Bulk Density (g/mL)		0.341±0.02	0.397±0.01	0.617±0.13	0.371±0.01	0.337 g/cm^3
Tapped Density (g/mL)		0.460±0.005	0.532±0.03	0.751±0.13	0.457±0.002	0.478 g/cm^3
True density (g/mL)		1.401±0.05	1.399±0.03	1.512±0.08	1.466±0.04	1.512-1.668 g/cm^3
Hausner Ratio		1.35±0.07	1.34±0.14	1.22±0.06	1.23±0.02	1.00-1.11 = Very Good
Carr's Index (%)		25.98±3.57	25.15±7.79	17.99±3.74	18.67±1.53	1-10 = Very Good
Levels (%)	Chlor	0.15	0.25	0.35	0.10	0.10%; 0.36% & 0.24%
	Calcium	0.26	0.49	0.92	0.36	
	Natrium	0.13	0.09	0.13	0.24	
CrI (%)		34.1	34.7	34.3	34.5	34,5%
Particle size (µm)		362	362	395	332	20-200 µm

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The results of the MCC moisture content test from corn husks (Table 1.). The MCC samples treated with 2 N and 4 N HCl showed moisture content values close to that of commercial MCC (pH 102), which has a reference moisture content of 5.37%. The moisture content of MCC treated with 6 N HCl aligns with literature values, which are typically below 5%. If the moisture content is relatively high, it can increase the cohesion between similar particles, causing the powder to lose its ability to flow properly [24]. pH MCC corn husk and the comparator, Avicel PH 102 (Table1.), also exhibited the same pH value of 6, which is consistent with the literature pH range is between 5 and 7.5 [16]. The results of the MCC melting point test from corn husks at each HCl concentration, as well as Avicel PH 102, showed that the powder did not melt but instead changed color to black within the temperature range of 270°C to 400°C. These results do not align with the literature, which states that the melting point should be around 260°C to 270°C [16].

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The flow rate of MCC corn husk is better than Avicel PH 102 as a commercial standard (Table 1.), because a good flow rate is indicated by a value greater than 10 g/s. This shows that increasing the concentration of HCl can affect the density and particle size of MCC. Powders with smaller particle sizes tend to have poor flowability due to the larger surface area per unit mass, which increases contact between particles. This greater contact increases cohesive and frictional forces, thus inhibiting the flow of the powder [25]. The results of the angle of repose for MCC corn husk show that, on average, the faster the flow of MCC, the smaller the angle of repose formed. This is believed to be due to the larger particle size and low cohesiveness of the powder, which contribute to its good flow properties. Smaller particle size, higher cohesiveness, and greater frictional forces, thus inhibiting the flow of the powder [26].

The results of bulk and tapping density of MCC corn husks showed that samples treated with 2 N and 4 N HCl, as well as Avicel PH 102, produced values close to the limit requirements (Table 1). MCC corn husks treated with 6 N HCl produced higher values compared to Avicel PH 102 and the literature. The actual density of MCC corn husks treated with 6 N HCl was within the limit requirement range of 1.512–1.668 g/cm³ [16], while MCC treated with 2 N, 4 N HCl, and Avicel PH 102 showed lower actual density values than those reported. The hausner ratio value for MCC corn husks treated with 2 N, 4 N, and 6 N HCl concentrations was comparable to Avicel PH 102. The higher concentration of HCl used in the hydrolysis process had an effect on reducing the carr's index and hausner ratio values. MCC corn husks resulting from hydrolysis with 6 N HCl showed better flow properties and compressibility compared to Avicel PH 102.

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3.2. Fourier Transformed Infrared (FTIR)

FTIR Spectra of MCC corn huks (Figure 1.) showed the presence of characteristic cellulose absorption bands. The absorption band at wavelengths of 3500–3250 cm⁻¹ indicates the O–H stretching vibration of α-cellulose, while the band at 2970–2850 cm⁻¹ corresponds to the C–H stretching vibration, further confirming the presence of α-cellulose [27]. Additionally, the absorption band at 900–800 cm⁻¹ indicates the presence of β-glycosidic linkages, which are characteristic of microcrystalline cellulose (MCC) [28].

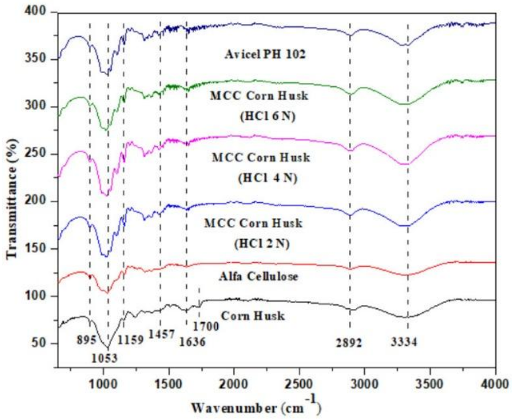

Figure 1. FTIR Spectra of Corn Husk, alfa cellulose, MCC Corn Husk and Avicel PH 102

FTIR analysis also revealed the presence of a lignin absorption band at around 1700 cm⁻¹ in raw corn husk powder that had not been treated with NaOH, indicating the presence of lignin prior to the delignification process. In contrast, the FTIR spectra of MCC derived from corn husks treated with HCl concentrations of 2 N, 4 N, and 6 N showed similar spectral patterns to that of Avicel PH 102. These spectra confirmed the presence of cellulose, while the absorption bands associated with hemicellulose and lignin were no longer observed in the MCC samples and Avicel PH 102. This indicates that the non-cellulosic components were effectively removed during the delignification and purification processes, leaving behind primarily α-cellulose [13].

3.3. Scanning Electrone Microscopy (SEM)

The morphological observation of raw corn husk powder revealed a denser surface structure, which is attributed to the presence of lignin still embedded in the cell wall, serving to protect the cellulose. In contrast, the morphology of α-cellulose showed the initial stages of solid peeling, leading to the formation of irregular fibrous structures. MCC derived from corn husks treated with 2 N and 4 N HCl exhibited elongated, stem-like shapes with uneven surfaces, slightly hollow structures, and distinguishable blunt-angled edges (Figure 2.).

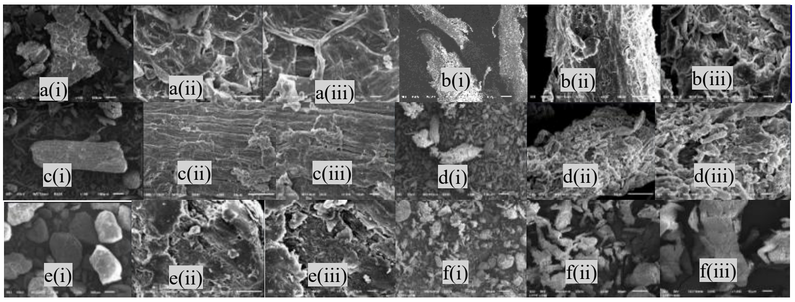

Figure 1. SEM Image of (a) Powder Corn Husk; (b) α-Cellulose; (c) MCC Corn Husk (HCl 2 N); (d) MCC Corn Husk (HCl 4 N); (e) MCC Corn Husk (HCl 6 N); (f) Avicel pH 102; (i) 100x; (ii) 500x; (iii) 1000x.

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The comparator, Avicel PH 102, displayed irregularly shaped particles with varying lengths, uneven and slightly hollow surfaces, and both pointed and blunt edges [29]. MCC obtained using 6 N HCl showed a more compact, spherical, and granular morphology compared to MCC 2 N, 4 N, and Avicel PH 102. It also had a smoother surface and blunt angles. Morphology MCC plays an important role in influencing flow properties [9,21,25,30]. In addition to the morphological results of the Scanning Electron Microscope–Energy Dispersive X-Ray (SEM-EDX) analysis, it shows that the chlorine, calcium and sodium levels of corn husk MCC (Figure 3).

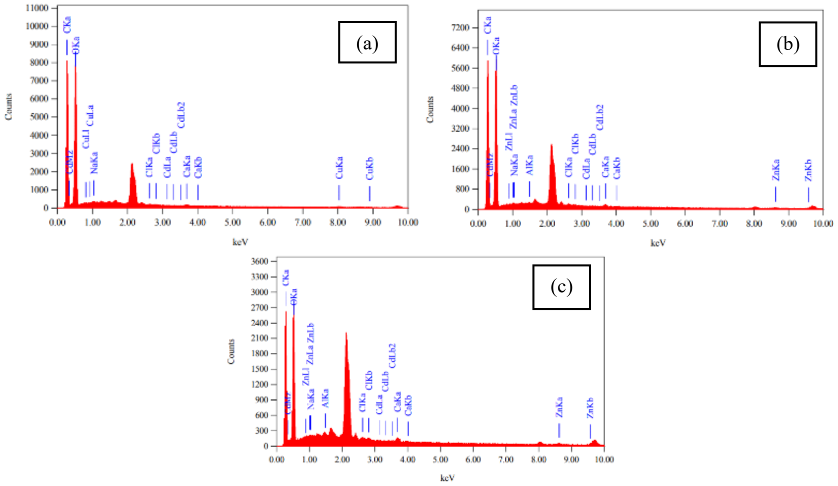

Figure 2. Test SEM-EDX Elements Klor, Calcium, Natrium (a) MCC Corn Husk (HCl 2 N); (b) MCC Corn Husk (HCl 4 N); (c) MCC Corn Husk (HCl 6 N)

3.4. *X-Ray Diffraction (XRD)*

X-Ray Diffraction (XRD) analysis of MCC corn husks showed that the crystallinity of the samples increased with higher HCl concentrations (Table 1). This increase in crystallinity is attributed to the removal of the amorphous lignin layer from the corn husk samples as evidenced by the absence of peaks at 2θ=24.2° (Figure 4.), which led to a higher cellulose content. However, at a 6 N HCl concentration, the crystallinity index decreased to 34.3%. This reduction is likely due to the high concentration of HCl, which, through the application of heat, caused the crystalline regions of the corn husk MCC to undergo hydrolysis, converting them into amorphous regions and thus reducing the overall crystallinity [31]

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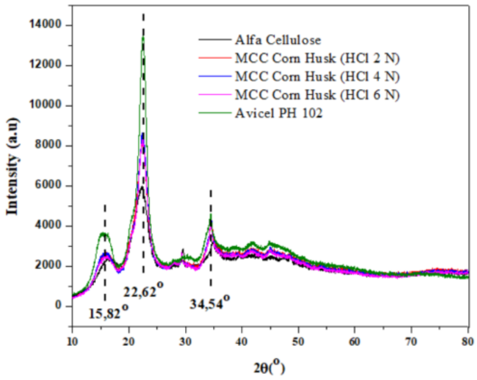

Figure 3. X-ray diffraction patterns of Corn Husk, alfa cellulose, MCC Corn Husk and Avicel PH 102

3.5. *Particle Size Analyzer (PSA)*

The results of the Particle Size Analysis (Table 2), it can be concluded that larger particle sizes result in better flowability of the MCC derived from corn husks. This improvement in flow

rate is attributed to the stronger gravitational force acting on the larger particles, which outweighs the tensile forces between the powder particles. Additionally, the reduced friction between particles makes it easier for the powder to flow [32].

Table 2. Average Particle Size Analyzer MCC Corn Husk

Sample	Dx (10) (µm)	Dx (50) (µm)	Dx (90) (µm)
Avicel pH 102	37.9	139	332
MCC 1	36.1	144	362
MCC 2	26.5	142	362
MCC 3	84.1	216	395
Mean	46.1	160	363
1xStd Dev	25.8	37.2	25.4
1xRSD (%)	55.8	23.3	7.01

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4. CONCLUSION

The results of the physicochemical characterization tests conducted on the three MCC samples showed FTIR absorption patterns similar to that of Avicel PH 102. The surface morphology of the corn husk MCC particles varied with HCl concentration, with the highest concentration (6 N) resulting in round, dense, and more granular particles compared to those obtained at lower concentrations. The crystallinity index of α-cellulose for the three MCC samples was as follows: 30.7% for the raw corn husk, 34.1% for the 2 N HCl-treated sample, 34.7% for the 4 N HCl treated sample, and 34.3% for the 6 N HCl-treated sample. The particle size distribution for the three MCC samples at Dx 90 was 362 µm, 362 µm, 392 µm, and 332 µm, respectively. The difference in HCl concentration during the hydrolysis process contributed to the varying characteristics of the corn husk MCC, with the 6 N HCl concentration producing MCC that met the characteristics required for pharmaceutical-grade MCC.

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Conflicts of interest: The authors declare no conflict of interest.

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BUKTI KORESPONDENSI CATATAN REVIEWER 2

Original Article

Pharmaceutical Grade Microcrystalline Cellulose from Corn Husk (*Zea mays* L.): Fabrication and Characterization

Received: date; Accepted: date; Published: date

Abstract: Corn is a plant that grows easily in tropical climates. Corn production in Indonesia reaches 25.18 tons, the use of which in society is still limited to corn kernels as food, while other parts of the corn plant are waste. Corn husks are an abundant natural waste and contain 44.08% cellulose, so they can potentially be a source of pharmaceutical excipients, namely microcrystalline cellulose (MCC). This research aims to isolate and characterize MCC from pharmaceutical grade corn husks with commercial MCC as a comparator. The two methods of making MCC are delignification using 2% NaOH at 80-90°C 4 h. Hydrolysis using variations in HCl concentrations, namely 2 N, 4 N, and 6 N, at a temperature of 80°C 4 h. The research results obtained cellulose content in α-cellulose and MCC of corn husks with 3 consecutive treatments of 74.02%, 84.48%, 86.55%, and 84.44%. The result of the analysis test of FTIR, SEM, XRD, and PSA instruments indicate that corn husk MCC has characteristics of commercial MCC as a standard. The resulting corn husk MCC has physicochemical characteristics according to standards that can be used as a pharmaceutical excipient.

Keywords: characterization, fabrication, microcrystalline cellulose, corn husk, pharmaceutical excipient.

1. INTRODUCTION

Corn plants are a staple food that is widely consumed after rice [1]. Corn kernels used in the food sector are only able to represent 5% of the total part of the corn plant; the remaining 95% of the corn plant is in the category of natural waste in the form of stalks, leaves, cobs, and corn husks [2]. The Pharmaceutical Industry in Indonesia still uses 95% of drug raw materials imported from abroad. Corn husks are part of the corn crop waste that has not been utilized optimally and contain quite high cellulose, which is 44.08% [3]. The high cellulose content in corn husks has the potential to be used as pharmaceutical excipient [4].

MCC is pure cellulose that has been isolated using mineral acids from α-cellulose fibrous plants. MCC is widely used as the best excipient in the manufacture of direct printed tablets. In the manufacture of tablets using of direct compression method, MCC is used as a dry binder, tablet disintegrant, filler, or thinner, absorbent, lubricant, and anti-adherent. MCC is widely used as an excipient in the manufacture of direct print tablets because it has good flow properties and compatibility [5,6].

MCC can be made by delignification and then hydrolysis. Delignification is carried out to change the structure of lignocellulose biomass, which aims to degrade lignin polymers bound to cellulose, then lignin will dissolve in water, and the result is α-cellulose. The deignified α-cellulose powder was subjected to controlled hydrolysis using an acidic solution. Acid hydrolysis can damage the amorphous region of the cellulose microfibrils, where the amorphous form will undergo disconnection and then leave a crystalline [7,8]. Several studies on the use of HCl in hydrolysis in the manufacture of MCC from natural materials have been able to increase the yield and crystallinity index [9–12].

Therefore, this study conducted further research with variations in HCl concentration in hydrolysis to produce corn husk MCC that have physicochemical characteristics in accordance with pharmaceutical grade standards and test the physicochemical characteristics of corn husk MCC compared to commercial MCC as a standard.

2. MATERIALS AND METHODS

2.1. Materials

Commented [DI1]: Please complete the manufacturer of the material used and its grade.

Corn husks from plantation waste in the Semarang area, NaOH 2%, NaOCl 5%, HCl, and aquadest. Materials for testing the physicochemical characteristics of MCC: Avicel PH 102, H_2SO_4 1N, H_2SO_4 72%, ethanol, iodized zinc chloride solution (zinc chloride, potassium iodide and iodine) and iodine 0.05 M.

2.2. Methods

2.2.1. Fabrication of MCC Corn Husk

a. Alkaline Delignification

The corn husks are sorted wet, washed clean, and dried for 2 days in the sun. The dried corn husks are then mashed and sifted using a mesh no. 40. The corn husk powder is deignified with a 2% NaOH solution at 80-90°C for 4 h, the residue is filtered and washed down to a neutral pH of 6-7. The next stage is bleaching with a solution of NaOCl 5% at 70°C for 1 hour and NaOCl 5% for 24 h at room temperature. The residue is filtered and washed to a neutral pH of 6-7. Cellulose is produced, dried, and mashed [13].

b. Acid Hydrolysis

The α-cellulose sample was hydrolyzed with a variation in HCl concentrations of 2 N, 4 N, and 6 N for 80°C 4 hours and then filtered and washed until a neutral pH of 6-7. The next stage is bleached 2 times with a 5% NaOCl solution of 70°C for 1 hour and soaked in the same solution for 24 hours at room temperature. The residue is filtered and washed until a neutral pH of 6-7. MCC is dried and smoothed then sifted mesh no. 60 [14].

2.2.2 Physicochemical Characterization of MCC Corn Husk

a. Determination of Cellulose Concentration

The precentage of cellulose concentration was determinated using the Che.sson-datta method [15].

b. Moisture Content

Determined using a moisture content tool set at a temperature of 150 °C for automatic time to constant weight. The standar requirement for MCC moisture content was not more than 5% [16].

c. pH

MCC corn husks as much as 1 gram added 50 mL aquadest stirring for 5 minutes then measured the pH using a pH instrument [17].

d. Melting Point

MCC is inserted into a capillary pipe and then put into a melting point device (Mettler Toledo) with a temperature of 200°C when the device is switched on and the temperature is deformed when the solids begin to melt.

e. Flow Rate and Angle of Repose

The flow rate of MCC corn husks using a flowability tester (Erweka GT) with a funnel diameter of 15 mm. The cover at the bottom of the funnel is opened and the flow speed is calculated at the time the granule starts flowing until the granule stops flowing using a stopwatch and then the time obtained and the height and diameter of the cone are measured [18].

f. Density, Carr's Index and Hausner Ratio

40 grams of corn husk MCC is placed in a 100 mL measuring cup. The surface of the powder is carefully leveled without being compressed its volume (Vo) measurement is performed. A measuring cup is installed on the support of the tapped density tester, 10, 500, and 1250 taps are carried out and V10, V500, and V1250 are read on the nearest measuring cup unit. Volume measured to the last tap (Vt) [19]. The density of MCC corn husks was determined by dividing weight by Vo (bulk density) and Vt (tapped density). The true density of MCC is determined by determining the volume using a picnometre. Carr's index and hausner ratio indices were calculated from the results of the bulk and tapped density that had been calculated.

2.2.3. Fourier Transformed Infrared (FTIR)

Fourier Transform Infrared Spectroscopy (FTIR) testing of microcrystalline cellulose from corn husks was used to determine the functional groups of the corn husk MCC using Agilent Technologies Cary 630 FTIR.

2.2.4. Scanning Electrone Microscope (SEM)

Scanning Electron Microscope-Energy Dispersive X-Ray (SEM-EDX) MCC testing of corn husks was used to determine the morphological shape as well as to analyze the elements contained in the sample using the Scanning Electron Microscope-Energy Dispersive X-Ray microscope (JEOLJSM-6510LA).

2.2.5. X-Ray Diffraction (XRD)

X-Ray Diffraction (XRD) analysis of MCC from corn husks was used to determine the crystallinity index produced by corn husk MCC using X-Ray Diffraction (D8 ADVANC X-Ray Diffraction) [20].

2.2.6. Particle Size Analyzer (PSA)

Particle Size Analyzer (PSA) is used to determine the particle size distribution of corn husk MCC using the Particle Size Analyzer tool (Malvern® Mastersizer 3000 (Malvern instrument UK) ([21].

3. RESULTS AND DISCUSSION

3.1. Physicochemical Characterization of MCC Corn Husk

The results of determining cellulose content using the Chasson-datta method were obtained from the average cellulose content of corn husk powder of 42.90%, the results obtained were close to the literature that corn husks have a cellulose content of 44.08% [22]. The yield of cellulose content in corn husk α-cellulose increased by 74.02% due to alkalization treatment with NaOH which caused the loss of lignin, mainly due to the unstable ester bond between cellulose and lignin complex, so that lignin that loosely binds to alkali to form a water-soluble alkaline lignin complex. NaOH can break the bond between cellulose with hemicellulose and lignin, causing changes in cellulose levels to increase [13]. The result of MCC corn husks was 84.48% HCl 2 N and MCC 4 N was 86.55%. The result of the concentration of MCC 6 N of cellulose content decreased by 84.44% (Table 1). The decrease in cellulose levels that occur is caused by the higher concentration of HCl causing an increase in heat (heat) causing the cellulose structure to open up so that cellulose molecules are dispersed freely in the solution, this freely dispersed cellulose structure results in the presence of dissolved cellulose carried away in the solution when the filtration process is carried out [23].

Table 1. Physical Chemical Characterization Test of MCC Corn Husk

Type of Assay		Result				Limit Requirements
		MCC Corn Husk 2 N	**MCC Corn Husk 4 N**	**MCC Corn Husk 6 N**	**MCC Commercial**	
Determination of Cellulose Levels (%)		84.48±2.99	86.55±0.91	84.44±2.34	80.81±1.14	80.81
Moisture Content (%)		5.82±0.41	5.66±0.29	3.33±0.93	4.93±0.11	<5
pH		6±0	6±0	6±0	6±0	5-7,5
Melting Point (°C)		299.67±0.58	299.67±0.58	270.66±0.58	315.33±0.58	260-270
Flow rate (g/s)		19.87±3.16	27.66±3.30	31.20±5.12	29.104±3.32	1.41
Angle of Repose (°)		29.59±1.01	28.45±1.12	25.98±3.14	45.27±1.22	34.4-49
Bulk Density (g/mL)		0.341±0.02	0.397±0.01	0.617±0.13	0.371±0.01	0.337 g/cm^3
Tapped Density (g/mL)		0.460±0.005	0.532±0.03	0.751±0.13	0.457±0.002	0.478 g/cm^3
True density (g/mL)		1.401±0.05	1.399±0.03	1.512±0.08	1.466±0.04	1.512-1.668 g/cm^3
Hausner Ratio		1.35±0.07	1.34±0.14	1.22±0.06	1.23±0.02	1.00-1.11 = Very Good
Carr's Index (%)		25.98±3.57	25.15±7.79	17.99±3.74	18.67±1.53	1-10 = Very Good
Levels (%)	Chlor	0.15	0.25	0.35	0.10	0.10%; 0.36% & 0.24%
	Calcium	0.26	0.49	0.92	0.36	
	Natrium	0.13	0.09	0.13	0.24	
CrI (%)		34.1	34.7	34.3	34.5	34,5%
Particle size (μm)		362	362	395	332	20-200 μm

The results of the MCC moisture content test from corn husks (Table 1.). The MCC samples treated with 2 N and 4 N HCl showed moisture content values close to that of commercial MCC (pH 102), which has a reference moisture content of 5.37%. The moisture content of MCC treated with 6 N HCl aligns with literature values, which are typically below 5%. If the moisture content is relatively high, it can increase the cohesion between similar particles, causing the powder to lose its ability to flow properly [24]. pH MCC corn husk and the comparator, Avicel PH 102 (Table1.), also exhibited the same pH value of 6, which is consistent with the literature pH range is between 5 and 7.5 [16]. The results of the MCC melting point test from corn husks at each HCl concentration, as well as Avicel PH 102, showed that the powder did not melt but instead changed color to black within the temperature range of 270°C to 400°C. These results do not align with the literature, which states that the melting point should be around 260°C to 270°C [16].

The flow rate of MCC corn husk is better than Avicel PH 102 as a commercial standard (Table 1.), because a good flow rate is indicated by a value greater than 10 g/s. This shows that increasing the concentration of HCl can affect the density and particle size of MCC. Powders with smaller particle sizes tend to have poor flowability due to the larger surface area per unit mass, which increases contact between particles. This greater contact increases cohesive and frictional forces, thus inhibiting the flow of the powder [25]. The results of the angle of repose for MCC corn husk show that, on average, the faster the flow of MCC, the smaller the angle of repose formed. This is believed to be due to the larger particle size and low cohesiveness of the powder, which contribute to its good flow properties. Smaller particle size, higher cohesiveness, and greater frictional forces, thus inhibiting the flow of the powder [26].

The results of bulk and tapping density of MCC corn husks showed that samples treated with 2 N and 4 N HCl, as well as Avicel PH 102, produced values close to the limit requirements (Table 1). MCC corn husks treated with 6 N HCl produced higher values compared to Avicel PH 102 and the literature. The actual density of MCC corn husks treated with 6 N HCl was within the limit requirement range of 1.512–1.668 g/cm^3 [16], while MCC treated with 2 N, 4 N HCl, and Avicel PH 102 showed lower actual density values than those reported. The hausner ratio value for MCC corn husks treated with 2 N, 4 N, and 6 N HCl concentrations was comparable to Avicel PH 102. The higher concentration of HCl used in the hydrolysis process had an effect on reducing the carr's index and hausner ratio values. MCC corn husks resulting from hydrolysis with 6 N HCl showed better flow properties and compressibility compared to Avicel PH 102.

3.2. Fourier Transformed Infrared (FTIR)

FTIR Spectra of MCC corn huks (Figure 1.) showed the presence of characteristic cellulose absorption bands. The absorption band at wavelengths of 3500–3250 cm^{-1} indicates the O–H stretching vibration of α-cellulose, while the band at 2970–2850 cm^{-1} corresponds to the C–H stretching vibration, further confirming the presence of α-cellulose [27]. Additionally, the absorption band at 900–800 cm^{-1} indicates the presence of β-glycosidic linkages, which are characteristic of microcrystalline cellulose (MCC) [28].

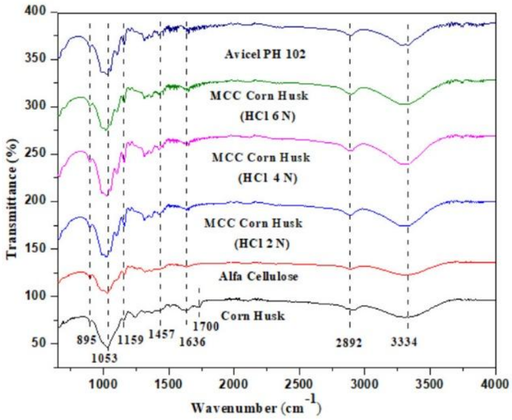

Figure 1. FTIR Spectra of Corn Husk, alfa cellulose, MCC Corn Husk and Avicel PH 102

FTIR analysis also revealed the presence of a lignin absorption band at around 1700 cm^{-1} in raw corn husk powder that had not been treated with NaOH, indicating the presence of lignin prior to the delignification process. In contrast, the FTIR spectra of MCC derived from corn husks treated with HCl concentrations of 2 N, 4 N, and 6 N showed similar spectral patterns to that of Avicel PH 102. These spectra confirmed the presence of cellulose, while the absorption bands associated with hemicellulose and lignin were no longer observed in the MCC samples and Avicel PH 102. This indicates that the non-cellulosic components were effectively removed during the delignification and purification processes, leaving behind primarily α-cellulose [13].

3.3. Scanning Electrone Microscopy (SEM)

The morphological observation of raw corn husk powder revealed a denser surface structure, which is attributed to the presence of lignin still embedded in the cell wall, serving to protect the cellulose. In contrast, the morphology of α-cellulose showed the initial stages of solid peeling, leading to the formation of irregular fibrous structures. MCC derived from corn husks treated with 2 N and 4 N HCl exhibited elongated, stem-like shapes with uneven surfaces, slightly hollow structures, and distinguishable blunt-angled edges (Figure 2.).

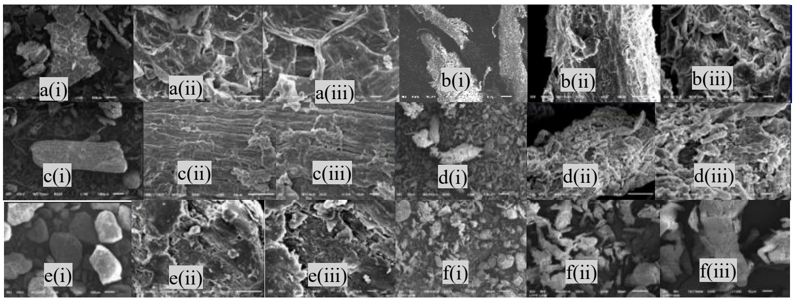

Figure 1. SEM Image of (a) Powder Corn Husk; (b) α-Cellulose; (c) MCC Corn Husk (HCl 2 N); (d) MCC Corn Husk (HCl 4 N); (e) MCC Corn Husk (HCl 6 N); (f) Avicel pH 102; (i) 100x; (ii) 500x; (iii) 1000x.

The comparator, Avicel PH 102, displayed irregularly shaped particles with varying lengths, uneven and slightly hollow surfaces, and both pointed and blunt edges [29]. MCC obtained using 6 N HCl showed a more compact, spherical, and granular morphology compared to MCC 2 N, 4 N, and Avicel PH 102. It also had a smoother surface and blunt angles. Morphology MCC plays an important role in influencing flow properties [9,21,25,30]. In addition to the morphological results of the Scanning Electron Microscope–Energy Dispersive X-Ray (SEM-EDX) analysis, it shows that the chlorine, calcium and sodium levels of corn husk MCC (Figure 3).

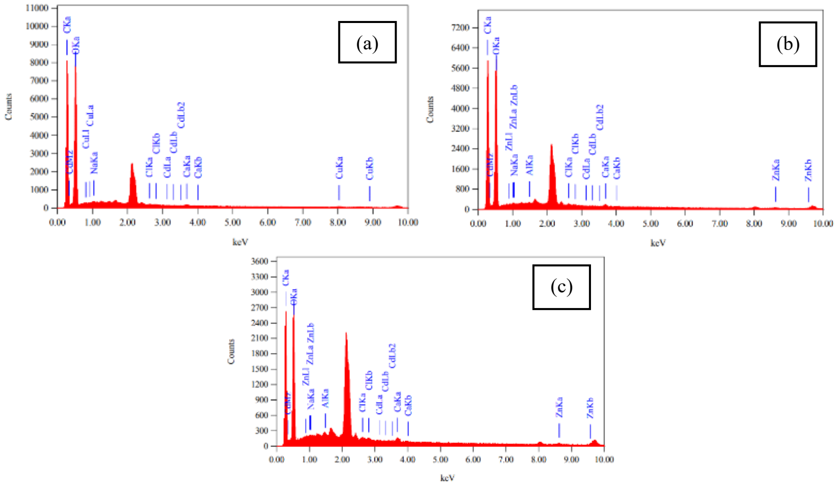

Figure 2. Test SEM-EDX Elements Klor, Calcium, Natrium (a) MCC Corn Husk (HCl 2 N); (b) MCC Corn Husk (HCl 4 N); (c) MCC Corn Husk (HCl 6 N)

3.4. X-Ray Diffraction (XRD)

X-Ray Diffraction (XRD) analysis of MCC corn husks showed that the crystallinity of the samples increased with higher HCl concentrations (Table 1). This increase in crystallinity is attributed to the removal of the amorphous lignin layer from the corn husk samples as evidenced by the absence of peaks at 2θ=24.2° (Figure 4.), which led to a higher cellulose content . However, at a 6 N HCl concentration, the crystallinity index decreased to 34.3%. This reduction is likely due to the high concentration of HCl, which, through the application of heat, caused the crystalline regions of the corn husk MCC to undergo hydrolysis, converting them into amorphous regions and thus reducing the overall crystallinity [31]

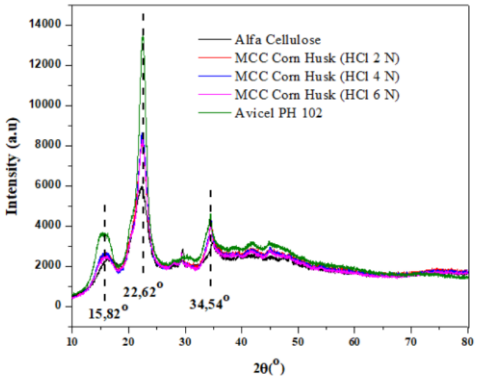

Figure 3. X-ray diffraction patterns of Corn Husk, alfa cellulose, MCC Corn Husk and Avicel PH 102

3.5. Particle Size Analyzer (PSA)

The results of the Particle Size Analysis (Table 2), it can be concluded that larger particle sizes result in better flowability of the MCC derived from corn husks. This improvement in flow

rate is attributed to the stronger gravitational force acting on the larger particles, which outweighs the tensile forces between the powder particles. Additionally, the reduced friction between particles makes it easier for the powder to flow [32].

Table 2. Average Particle Size Analyzer MCC Corn Husk

	Sample	**Dx (10) (μm)**	**Dx (50) (μm)**	**Dx (90) (μm)**
	Avicel pH 102	37.9	139	332
	MCC 1	36.1	144	362
	MCC 2	26.5	142	362
	MCC 3	84.1	216	395
Mean		46.1	160	363
1xStd Dev		25.8	37.2	25.4
1xRSD (%)		55.8	23.3	7.01

4. CONCLUSION

The results of the physicochemical characterization tests conducted on the three MCC samples showed FTIR absorption patterns similar to that of Avicel PH 102. The surface morphology of the corn husk MCC particles varied with HCl concentration, with the highest concentration (6 N) resulting in round, dense, and more granular particles compared to those obtained at lower concentrations. The crystallinity index of α-cellulose for the three MCC samples was as follows: 30.7% for the raw corn husk, 34.1% for the 2 N HCl-treated sample, 34.7% for the 4 N HCl treated sample, and 34.3% for the 6 N HCl-treated sample. The particle size distribution for the three MCC samples at Dx 90 was 362 μm, 362 μm, 392 μm, and 332 μm, respectively. The difference in HCl concentration during the hydrolysis process contributed to the varying characteristics of the corn husk MCC, with the 6 N HCl concentration producing MCC that met the characteristics required for pharmaceutical-grade MCC.

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Original Article

Pharmaceutical Grade Microcrystalline Cellulose from Corn Husk (*Zea mays* L.): Fabrication and Characterization

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Abstract: Corn is a plant that grows easily in tropical climates. Corn production in Indonesia reaches 25.18 tons, the use of which in society is still limited to corn kernels as food, while other parts of the corn plant are waste. Corn husks are an abundant natural waste and contain 44.08% cellulose, so they can potentially be a source of pharmaceutical excipients, namely microcrystalline cellulose (MCC). This research aims to isolate and characterize MCC from pharmaceutical grade corn husks with commercial MCC as a comparator. The two methods of making MCC are delignification using 2% NaOH at 80-90°C 4 h. Hydrolysis using variations in HCl concentrations, namely 2 N, 4 N, and 6 N, at a temperature of 80°C 4 h. The research results obtained cellulose content in α-cellulose and MCC of corn husks with 3 consecutive treatments of 74.02%, 84.48%, 86.55%, and 84.44%. The result of the analysis test of FTIR, SEM, XRD, and PSA instruments indicate that corn husk MCC has characteristics of commercial MCC as a standard. The resulting corn husk MCC has physicochemical characteristics according to standards that can be used as a pharmaceutical excipient.

Keywords: characterization, fabrication, microcrystalline cellulose, corn husk, pharmaceutical excipient.

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1. INTRODUCTION

Corn plants are a staple food that is widely consumed after rice [1]. Corn kernels used in the food sector are only able to represent 5% of the total part of the corn plant; the remaining 95% of the corn plant is in the category of natural waste in the form of stalks, leaves, cobs, and corn husks [2]. The Pharmaceutical Industry in Indonesia still uses 95% of drug raw materials imported from abroad. Corn husks are part of the corn crop waste that has not been utilized optimally and contain quite high cellulose, which is 44.08% [3]. The high cellulose content is a consideration for developing its benefits and potential to be used as pharmaceutical excipient [4].

Microcrystalline Cellulose (MCC) is pure cellulose that has been isolated using mineral acids from α-cellulose fibrous plants. MCC is widely used as the best excipient in the manufacture of direct printed tablets. In the manufacture of tablets using of direct compression method, MCC is used as a dry binder, tablet disintegrant, filler, or thinner, absorbent, lubricant, and anti-adherent. MCC is widely used as an excipient in the manufacture of direct print tablets because it has good flow properties and compatibility [5,6].

MCC can be made by delignification and then hydrolysis. Delignification is carried out to change the structure of lignocellulose biomass, which aims to degrade lignin polymers bound to cellulose, then lignin will dissolve in water, and the result is α-cellulose. Delignification of α-cellulose powder was subjected to controlled hydrolysis using an acidic solution. Acid hydrolysis can damage the amorphous region of the cellulose microfibrils, where the amorphous form will undergo disconnection and then leave a crystalline [7,8]. Several studies on the use of HCl in hydrolysis in the manufacture of MCC from other natural materials have been able to increase the yield and crystallinity index [9–12].

The pharmaceutical industry in Indonesia is still dependent on imported raw materials (95%) [13]. The raw materials here are not only active ingredients but also excipients that play an important role in determining the quality of the dosage form. The abundant corn crop yield (38.38%) means that the amount of corn husk waste produced also increases [14]. It is necessary to develop cellulose technology for high in corn husks (44.08%) [3] into MCC as an alternative pharmaceutical excipient native to Indonesia that not only solves the problem of meeting the needs of raw materials for the pharmaceutical industry but also solves the problem of plantation waste. Several studies on the isolation of MCC from corn waste that have been carried out include corn cobs with variations of NaOH in the delignification process and hydrolysis with 10% H_2SO4 obtained a yield of 30% and CrI 91.26% [15]. Hydrolysis of pulut corn husks with 2.5N HCl for 10 minutes produced MCC with CrI 79% [16]. However, this study was limited to CrI and morphology analysis, so it is necessary to conduct research on the fabrication and physicochemical and mechanical characterization of MCC corn husks compared to Avicel PH 102 as a commercial standard so that it can guarantee its quality as a pharmaceutical excipient.

2. MATERIALS AND METHODS

2.1. Materials

Corn husks from plantation waste in the Semarang area (Indonesia) which is dried and ground with 40 mesh. Technical grade material: sodium hydroxide (NaOH) (Hangzhou Lizu Co., Ltd), sodium hypochlorite (NaOCl) (Asahimas), hydrochloric acid (HCl) (Tjiwi Kimia). Pro-analysis material (Merck): sulfuric acid (H_2SO_4), ethanol, iodized zinc chloride solution (zinc chloride, potassium iodide and iodine) and iodine. Pharmaceutical grade material: Avicel PH 102 (American International Chemical/AIC, Inc-Framingham USA) as commercial standard.

2.2. Methods

2.2.1. Fabrication of MCC Corn Husk

a. Alkaline Delignification

Delignification of corn husk powder with 2% NaOH at 80-90°C for 4 h, the residue is filtered and washed down to a neutral pH of 6-7. The next stage is bleaching with a solution of NaOCl 5% at 70°C for 1 hour and NaOCl 5% for 24 h at room temperature. The residue is filtered and washed to a neutral pH of 6-7. Cellulose is produced, dried, and mashed [17].

b. Acid Hydrolysis

Hydrolysis of α-cellulose with variations in HCl concentration of 2 N, 4 N, and 6 N for 80°C 4 hours and then filtered and washed until a neutral pH of 6-7. The next stage is bleached 2 times with a 5% NaOCl solution of 70°C for 1 hour and soaked in the same solution for 24 hours at room temperature. The residue is filtered and washed until a neutral pH of 6-7. MCC is dried and smoothed then sifted mesh no. 60 [18].

2.2.2 Physicochemical Characterization of MCC Corn Husk

a. Determination of Cellulose Concentration

Concentration of cellulose was determined using the Chesson-datta method [19].

b. Moisture Content

Determined using a moisture content tool set at a temperature of 150 °C for automatic time to constant weight. The standard requirement for MCC moisture content was not more than 5% [20].

c. pH

MCC corn husks as much as 1 gram added 50 mL aquadest stirring for 5 minutes then measured the pH using a pH instrument [21].

d. Melting Point

MCC is inserted into a capillary pipe and then put into a melting point device (Mettler Toledo) with a temperature of 200°C when the device is switched on and the temperature is deformed when the solids begin to melt.

e. Flow Rate and Angle of Repose

The flow rate of MCC corn husks using a flowability tester (Erweka GT) with a funnel diameter of 15 mm. The cover at the bottom of the funnel is opened and the flow speed is calculated at the time the granule starts flowing until the granule stops flowing using a stopwatch and then the time obtained and the height and diameter of the cone are measured [22].

f. Density, Carr's Index and Hausner Ratio

40 grams of corn husk MCC is placed in a 100 mL measuring cup. The surface of the powder is carefully leveled without being compressed its volume (Vo) measurement is performed. A measuring cup is installed on the support of the tapped density tester, 10, 500, and 1250 taps are carried out and V10, V500, and V1250 are read on the nearest measuring cup unit. Volume measured to the last tap (Vt) [23]. The density of MCC corn husks was determined by dividing weight by Vo (bulk density) and Vt (tapped density). The true density of MCC is determined by determining the volume using a picnometre. Carr's index and hausner ratio indices were calculated from the results of the bulk and tapped density that had been calculated.

2.2.3. Fourier Transformed Infrared (FTIR)

Fourier Transform Infrared Spectroscopy (FTIR) testing of microcrystalline cellulose from corn husks was used to determine the functional groups of the corn husk MCC using Agilent Technologies Cary 630 FTIR with Attenuated Total Reflectance (ATR).

2.2.4. Scanning Electrone Microscope (SEM)

Scanning Electron Microscope-Energy Dispersive X-Ray (SEM-EDX) MCC testing of corn husks was used to determine the morphological shape as well as to analyze the elements contained in the sample using the Scanning Electron Microscope-Energy Dispersive X-Ray microscope (JEOLJSM-6510LA).

2.2.5. X-Ray Diffraction (XRD)

X-Ray Diffraction (XRD) analysis of MCC from corn husks was used to determine the crystallinity index produced by corn husk MCC using X-Ray Diffraction (D8 ADVANC X-Ray Diffraction) [24].

2.2.6. Particle Size Analyzer (PSA)

Particle Size Analyzer (PSA) is used to determine the particle size distribution of corn husk MCC using the Particle Size Analyzer tool (Malvern® Mastersizer 3000 (Malvern instrument UK) [25].

3. RESULTS AND DISCUSSION

3.1. Physicochemical Characterization of MCC Corn Husk

The results of determining cellulose content using the Chasson-datta method were obtained from the average cellulose content of corn husk powder of 42.90%, the results obtained were close to the literature that corn husks have a cellulose content of 44.08% [3]. The yield of cellulose content in corn husk α-cellulose increased by 74.02% due to alkalization treatment with NaOH which caused the loss of lignin, mainly due to the unstable ester bond between cellulose and lignin complex, so that lignin that loosely binds to alkali to form a water-soluble alkaline lignin complex. NaOH can break the bond between cellulose with hemicellulose and lignin, causing changes in cellulose levels to increase [17]. The result concentration of cellulose MCC corn husks with HCl 2 N 84.48% and 4 N 86.55% and there was a decreased in 6 N 84.44% (Table 1). The decrease in cellulose levels that occur is caused by the higher concentration of HCl causing an increase in heat (heat) causing the cellulose structure to open up so that cellulose molecules are dispersed freely in the solution, this freely dispersed cellulose structure results in the presence of dissolved cellulose carried away in the solution when the filtration process is carried out [26].

Table 1. Physical Chemical Characterization Test of MCC Corn Husk

Type of Assay		Result				Limit Requirements
		MCC Corn Husk with HCL 2 N	**MCC Corn Husk with HCL 4 N**	**MCC Corn Husk with HCL 6 N**	**Standard Commercial (Avicel PH 102)**	
Determination of Cellulose Levels (%)		84.48±2.99	86.55±0.91	84.44±2.34	80.81±1.14	80.81
Moisture Content (%)		5.82±0.41	5.66±0.29	3.33±0.93	4.93±0.11	<5
pH		6±0	6±0	6±0	6±0	5-7,5
Melting Point (°C)		299.67±0.58	299.67±0.58	270.66±0.58	315.33±0.58	260-270
Flow rate (g/s)		19.87±3.16	27.66±3.30	31.20±5.12	29.104±3.32	1.41
Angle of Repose (°)		29.59±1.01	28.45±1.12	25.98±3.14	45.27±1.22	34.4-49
Bulk Density (g/mL)		0.341±0.02	0.397±0.01	0.617±0.13	0.371±0.01	0.337 g/cm^3
Tapped Density (g/mL)		0.460±0.005	0.532±0.03	0.751±0.13	0.457±0.002	0.478 g/cm^3
True density (g/mL)		1.401±0.05	1.399±0.03	1.512±0.08	1.466±0.04	1.512-1.668 g/cm^3
Hausner Ratio		1.35±0.07	1.34±0.14	1.22±0.06	1.23±0.02	1.00-1.11 = Very Good
Carr's Index (%)		25.98±3.57	25.15±7.79	17.99±3.74	18.67±1.53	1-10 = Very Good
Levels (%)	Chlor	0.15	0.25	0.35	0.10	0.10%; 0.36% & 0.24%
	Calcium	0.26	0.49	0.92	0.36	
	Natrium	0.13	0.09	0.13	0.24	
CrI (%)		34.1	34.7	34.3	34.5	34,5%
Particle size (µm)		362	362	395	332	20-200 µm

The results of the MCC moisture content test from corn husks (Table 1.). The MCC samples treated with 2 N and 4 N HCl showed moisture content values close to that of commercial MCC (pH 102), which has a reference moisture content of 5.37%. The moisture content of MCC treated with 6 N HCl aligns with literature values, which are typically below 5%. If the moisture content is relatively high, it can increase the cohesion between similar particles, causing the powder to lose its ability to flow properly [27]. pH MCC corn husk and the comparator, Avicel PH 102 (Table1.), also exhibited the same pH value of 6, which is consistent with the literature pH range is between 5 and 7.5 [20]. The results of the MCC melting point test of corn husks at each HCl concentration gave results in the hydrolysis treatment with 2 and 4 N HCl, it was higher than the limit requirement because there were still many crystalline forms of cellulose that were still bound to the amorphous form, while with 6N HCl, the results showed that they were comparable to the limit requirement of 260°C to 270°C [20].

The flow rate of MCC corn husk is better than Avicel PH 102 as a commercial standard (Table 1.), because a good flow rate is indicated by a value greater than 10 g/s. This shows that increasing the concentration of HCl can affect the density and particle size of MCC. Powders with smaller particle sizes tend to have poor flowability due to the larger surface area per unit mass, which increases contact between particles. This greater contact increases cohesive and frictional forces, thus inhibiting the flow of the powder [28]. The results of the angle of repose for MCC corn husk show that, on average, the faster the flow of MCC, the smaller the angle of repose formed. This is believed to be due to the larger particle size and low cohesiveness of the powder, which contribute to its good flow properties. Smaller particle size, higher cohesiveness, and greater frictional forces, thus inhibiting the flow of the powder [29].

The results of bulk and tapping density of MCC corn husks showed that samples treated with 2 N and 4 N HCl, as well as Avicel PH 102, produced values close to the limit requirements (Table 1). MCC corn husks treated with 6 N HCl produced higher values compared to Avicel PH 102 and the literature. The actual density of MCC corn husks treated with 6 N HCl was within the limit requirement range of 1.512–1.668 g/cm^3 [20], while MCC treated with 2 N, 4 N HCl, and Avicel PH 102 showed lower actual density values than those reported. The hausner ratio value for MCC corn husks treated with 2 N, 4 N, and 6 N HCl concentrations was comparable to Avicel PH 102. The higher concentration of HCl used in the hydrolysis process had an effect on reducing the carr's index and hausner ratio. MCC corn husks resulting from hydrolysis with 6 N HCl showed better flow properties and compressibility compared to Avicel PH 102. The amorphous form of cellulose is very susceptible to HCl so that the higher the concentration of HCl, the more the amorphous form is lost so that denser cellulose crystals will be formed with fewer cavities on the particle surface as seen in the morphology from the SEM analysis results (Figure 2.). The number of cavities on the particle surface can create space between particles which affects the increase in porosity and bulk volume so that its flowability is low [30].

3.2. Fourier Transformed Infrared (FTIR)

FTIR Spectra of MCC corn huks (Figure 1.) showed the presence of characteristic cellulose absorption bands. The absorption band at wavelengths of 3500–3250 cm^{-1} indicates the O–H stretching vibration of α-cellulose, while the band at 2970–2850 cm^{-1} corresponds to the C–H stretching vibration, further confirming the presence of α-cellulose [31]. Additionally, the absorption band at 900–800 cm^{-1} indicates the presence of β-glycosidic linkages, which are characteristic of microcrystalline cellulose (MCC) [32].

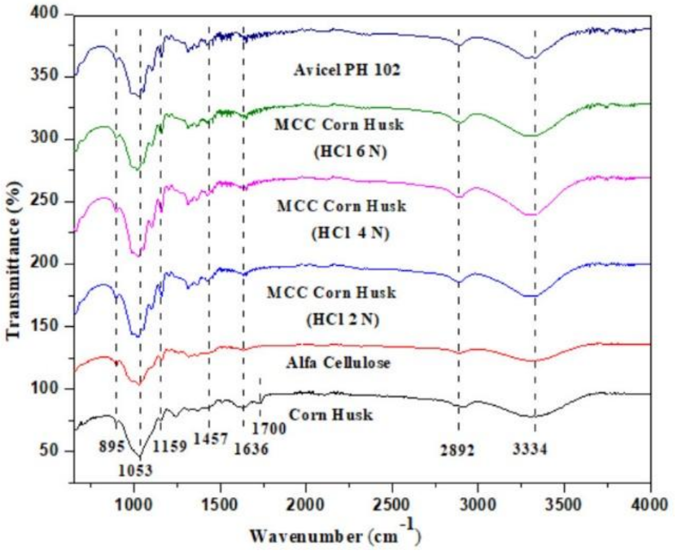

Figure 1. FTIR Spectra of Corn Husk, alfa cellulose, MCC Corn Husk and Avicel PH 102

FTIR analysis also revealed the presence of a lignin absorption band at around 1700 cm^{-1} in raw corn husk powder that had not been treated with NaOH, indicating the presence of lignin prior to the delignification process. In contrast, the FTIR spectra of MCC derived from corn husks treated with HCl concentrations of 2 N, 4 N, and 6 N showed similar spectral patterns to that of Avicel PH 102. These spectra confirmed the presence of cellulose, while the absorption bands associated with hemicellulose and lignin were no longer observed in the MCC samples and Avicel PH 102. This indicates that the non-cellulosic components were effectively removed during the delignification and purification processes, leaving behind primarily α-cellulose [17].

3.3. Scanning Electrone Microscopy (SEM)

The morphological observation of raw corn husk powder revealed a denser surface structure, which is attributed to the presence of lignin still embedded in the cell wall, serving to protect the cellulose. In contrast, the morphology of α-cellulose showed the initial stages of solid peeling, leading to the formation of irregular fibrous structures. MCC derived from corn husks treated with 2 N and 4 N HCl exhibited elongated, stem-like shapes with uneven surfaces, slightly hollow structures, and distinguishable blunt-angled edges (Figure 2.).

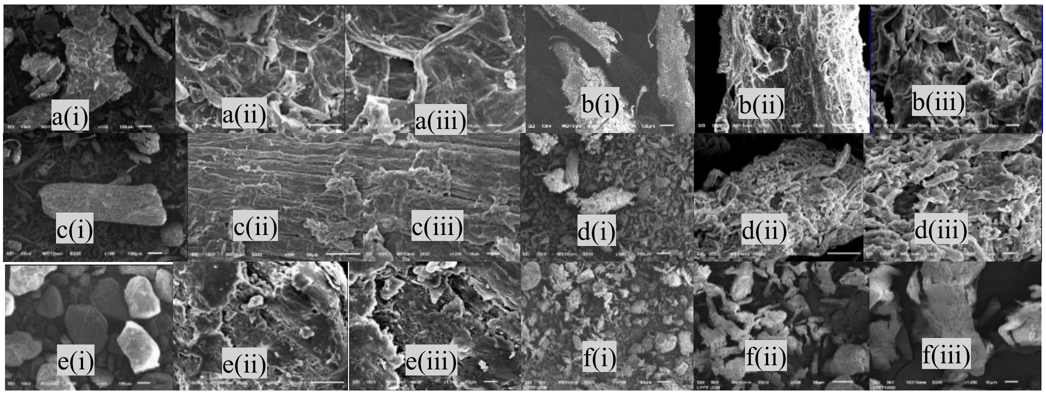

Figure 1. SEM Image of (a) Powder Corn Husk; (b) α-Cellulose; (c) MCC Corn Husk (HCl 2 N); (d) MCC Corn Husk (HCl 4 N); (e) MCC Corn Husk (HCl 6 N); (f) Avicel pH 102; with magnification (i) 100x; (ii) 500x; (iii) 1000x.

The comparator, Avicel PH 102, displayed irregularly shaped particles with varying lengths, uneven and slightly hollow surfaces, and both pointed and blunt edges [33]. MCC obtained using 6

N HCl showed a more compact, spherical, and granular morphology compared to MCC 2 N, 4 N, and Avicel PH 102. It also had a smoother surface and blunt angles. Morphology MCC plays an important role in influencing flow properties [9,21,25,30]. In addition to the morphological results of the Scanning Electron Microscope–Energy Dispersive X-Ray (SEM-EDX) analysis, it shows that the chlorine, calcium and sodium levels of corn husk MCC (Figure 3).

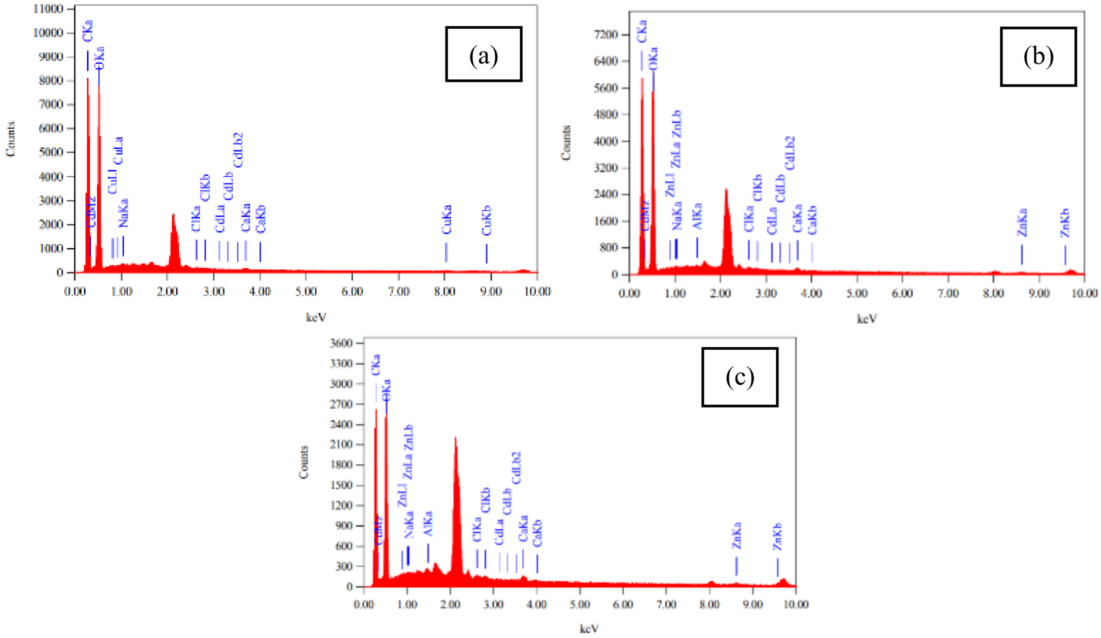

Figure 2. Test SEM-EDX Elements Klor, Calcium, Natrium (a) MCC Corn Husk (HCl 2 N); (b) MCC Corn Husk (HCl 4 N); (c) MCC Corn Husk (HCl 6 N)

3.4. X-Ray Diffraction (XRD)

X-Ray Diffraction (XRD) analysis of MCC corn husks showed that the crystallinity of the samples increased with higher HCl concentrations (Table 1). This increase in crystallinity is due to the loss of the lignin layer from the corn husk sample as evidenced by the absence of a peak at $2\theta=24.2°$ so that α-cellulose remains and it is indicated that there is no peak related to semicrystalline cellulose in the corn husk MCC due to the loss of amorphous properties during hydrolysis with increasing HCl concentrations shown in Figure 4 with an increase in peak intensity at $2\theta= \pm 20°$ [34-35]. However, at a 6 N HCl concentration, the crystallinity index decreased to 34.3% (Table 1.). This reduction is likely due to the high concentration of HCl, which, through the application of heat, caused the crystalline regions of the corn husk MCC to undergo hydrolysis, converting them into amorphous regions and thus reducing the overall crystallinity [36]

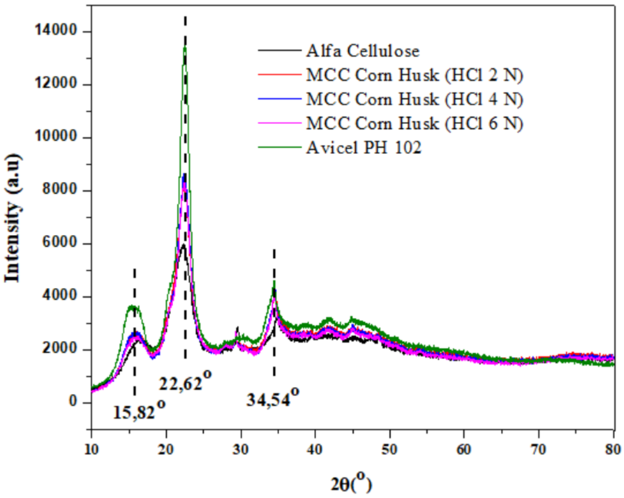

Figure 3. X-ray diffraction patterns of Corn Husk, alfa cellulose, MCC Corn Husk and Avicel PH 102

3.5. Particle Size Analyzer (PSA)

The results of the Particle Size Analysis (Table 2), it can be concluded that larger particle sizes result in better flowability of the MCC derived from corn husks. This improvement in flow rate is attributed to the stronger gravitational force acting on the larger particles, which outweighs the tensile forces between the powder particles. Additionally, the reduced friction between particles makes it easier for the powder to flow [37].

Table 2. Average Particle Size Analyzer MCC Corn Husk

Sample	Dx (10) (μm)	Dx (50) (μm)	Dx (90) (μm)
Avicel pH 102	37.9	139	332
MCC 1	36.1	144	362
MCC 2	26.5	142	362
MCC 3	84.1	216	395
Mean	46.1	160	363
1xStd Dev	25.8	37.2	25.4
1xRSD (%)	55.8	23.3	7.01

Note: Dv 10 – the size of particle below which 10% of the sample lies, Dv 50 (50%) and Dv 90 (90%).

4. CONCLUSION

The results of the physicochemical characterization tests conducted on the three MCC samples showed FTIR absorption patterns similar to that of Avicel PH 102. The surface morphology of the corn husk MCC particles varied with HCl concentration, with the highest concentration (6 N) resulting in round, dense, and more granular particles compared to those obtained at lower concentrations. The crystallinity index of α-cellulose for the three MCC samples was as follows: 30.7% for the raw corn husk, 34.1% for the 2 N HCl-treated sample, 34.7% for the 4 N HCl treated sample, and 34.3% for the 6 N HCl-treated sample. The particle size distribution for the three MCC samples at Dx 90 was 362 μm, 362 μm, 392 μm, and 332 μm, respectively. The difference in HCl concentration during the hydrolysis process contributes to various characteristics of corn husk MCC. The results of the analysis consistently show that hydrolysis with 6N HCl can produce MCC corn husks with pharmaceutical grade characteristics so that they can be used as an alternative excipient by the pharmaceutical industry which can realize the independence of the national raw material industry. Therefore, corn husks powder has the potential to become an alternative source for microcrystalline cellulose fabrication which is expected to not only address the problem of raw material needs for the pharmaceutical industry but also the problem of waste.

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Conflicts of interest: The authors declare no conflict of interest.

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Original Article

Pharmaceutical Grade Microcrystalline Cellulose from Corn Husk (*Zea mays* L.): Fabrication and Characterization

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Abstract: Corn is a plant that grows easily in tropical climates. Corn production in Indonesia reaches 25.18 tons, the use of which in society is still limited to corn kernels as food, while other parts of the corn plant are waste. Corn husks are an abundant natural waste and contain 44.08% cellulose, so they can potentially be a source of pharmaceutical excipients, namely microcrystalline cellulose (MCC). This research aims to isolate and characterize MCC from pharmaceutical grade corn husks with commercial MCC as a comparator. The two methods of making MCC are delignification using 2% NaOH at 80-90°C 4 h. Hydrolysis using variations in HCl concentrations, namely 2 N, 4 N, and 6 N, at a temperature of 80°C 4 h. The research results obtained cellulose content in α-cellulose and MCC of corn husks with 3 consecutive treatments of 74.02%, 84.48%, 86.55%, and 84.44%. The result of the analysis test of FTIR, SEM, XRD, and PSA instruments indicate that corn husk MCC has characteristics of commercial MCC as a standard. The resulting corn husk MCC has physicochemical characteristics according to standards that can be used as a pharmaceutical excipient.

Keywords: characterization, fabrication, microcrystalline cellulose, corn husk, pharmaceutical excipient

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1. INTRODUCTION

Corn plants are a staple food that is widely consumed after rice [1]. Corn kernels used in the food sector are only able to represent 5% of the total part of the corn plant; the remaining 95% of the corn plant is in the category of natural waste in the form of stalks, leaves, cobs, and corn husks [2]. The Pharmaceutical Industry in Indonesia still uses 95% of drug raw materials imported from abroad. Corn husks are part of the corn crop waste that has not been utilized optimally and contain quite high cellulose, which is 44.08% [3]. The high cellulose content is a consideration for developing its benefits and potential to be used as pharmaceutical excipient [4].

Microcrystalline Cellulose (MCC) is pure cellulose that has been isolated using mineral acids from α-cellulose fibrous plants. MCC is widely used as the best excipient in the manufacture of direct printed tablets. In the manufacture of tablets using of direct compression method, MCC is used as a dry binder, tablet disintegrant, filler, or thinner, absorbent, lubricant, and anti-adherent. MCC is widely used as an excipient in the manufacture of direct print tablets because it has good flow properties and compatibility [5,6].

MCC can be made by delignification and then hydrolysis. Delignification is carried out to change the structure of lignocellulose biomass, which aims to degrade lignin polymers bound to cellulose, then lignin will dissolve in water, and the result is α-cellulose. Delignification of α-cellulose powder was subjected to controlled hydrolysis using an acidic solution. Acid hydrolysis can damage the amorphous region of the cellulose microfibrils, where the amorphous form will undergo disconnection and then leave a crystalline [7,8]. Several studies on the use of HCl in hydrolysis in the manufacture of MCC from other natural materials have been able to increase the yield and crystallinity index [9–12].

The pharmaceutical industry in Indonesia is still dependent on imported raw materials (95%) [13]. The raw materials here are not only active ingredients but also excipients that play an important role in determining the quality of the dosage form. The abundant corn crop yield (38.38%) means that the amount of corn husk waste produced also increases [14]. It is necessary to develop cellulose technology for high in corn husks (44.08%) [3] into MCC as an alternative pharmaceutical excipient native to Indonesia that not only solves the problem of meeting the needs of raw materials for the pharmaceutical industry but also solves the problem of plantation waste. Several studies on the isolation of MCC from corn waste that have been carried out include corn cobs with variations of NaOH in the delignification process and hydrolysis with 10% H_2SO4 obtained a yield of 30% and CrI 91.26% [15]. Hydrolysis of pulut corn husks with 2.5N HCl for 10 minutes produced MCC with CrI 79% [16]. However, this study was limited to CrI and morphology analysis, so it is necessary to conduct research on the fabrication and physicochemical and mechanical characterization of MCC corn husks compared to Avicel PH 102 as a commercial standard so that it can guarantee its quality as a pharmaceutical excipient.

2. MATERIALS AND METHODS

2.1. Materials

Corn husks from plantation waste in the Semarang area (Indonesia) which is dried and ground with 40 mesh. Technical grade material: sodium hydroxide (NaOH) (Hangzhou Lizu Co., Ltd), sodium hypochlorite (NaOCl) (Asahimas), hydrochloric acid (HCl) (Tjiwi Kimia). Pro-analysis material (Merck): sulfuric acid (H_2SO_4), ethanol, iodized zinc chloride solution (zinc chloride, potassium iodide and iodine) and iodine. Pharmaceutical grade material: Avicel PH 102 (American International Chemical/AIC, Inc-Framingham USA) as commercial standard.

2.2. Methods

2.2.1. Fabrication of MCC Corn Husk

a. Alkaline Delignification

Delignification of corn husk powder with 2% NaOH at 80-90°C for 4 h, the residue is filtered and washed down to a neutral pH of 6-7. The next stage is bleaching with a solution of NaOCl 5% at 70°C for 1 hour and NaOCl 5% for 24 h at room temperature. The residue is filtered and washed to a neutral pH of 6-7. Cellulose is produced, dried, and mashed [17].

b. Acid Hydrolysis

Hydrolysis of α-cellulose with variations in HCl concentration of 2 N, 4 N, and 6 N for 80°C 4 hours and then filtered and washed until a neutral pH of 6-7. The next stage is bleached 2 times with a 5% NaOCl solution of 70°C for 1 hour and soaked in the same solution for 24 hours at room temperature. The residue is filtered and washed until a neutral pH of 6-7. MCC is dried and smoothed then sifted mesh no. 60 [18].

2.2.2 Physicochemical Characterization of MCC Corn Husk

a. Determination of Cellulose Concentration

Concentration of cellulose was determined using the Chesson-datta method [19].

b. Moisture Content

Determined using a moisture content tool set at a temperature of 150 °C for automatic time to constant weight. The standard requirement for MCC moisture content was not more than 5% [20].

c. pH

MCC corn husks as much as 1 gram added 50 mL aquadest stirring for 5 minutes then measured the pH using a pH instrument [21].

d. Melting Point

MCC is inserted into a capillary pipe and then put into a melting point device (Mettler Toledo) with a temperature of 200°C when the device is switched on and the temperature is deformed when the solids begin to melt.

e. Flow Rate and Angle of Repose

The flow rate of MCC corn husks using a flowability tester (Erweka GT) with a funnel diameter of 15 mm. The cover at the bottom of the funnel is opened and the flow speed is calculated at the time the granule starts flowing until the granule stops flowing using a stopwatch and then the time obtained and the height and diameter of the cone are measured [22].

f. Density, Carr's Index and Hausner Ratio

40 grams of corn husk MCC is placed in a 100 mL measuring cup. The surface of the powder is carefully leveled without being compressed its volume (Vo) measurement is performed. A measuring cup is installed on the support of the tapped density tester, 10, 500, and 1250 taps are carried out and V10, V500, and V1250 are read on the nearest measuring cup unit. Volume measured to the last tap (Vt) [23]. The density of MCC corn husks was determined by dividing weight by Vo (bulk density) and Vt (tapped density). The true density of MCC is determined by determining the volume using a picnometre. Carr's index and hausner ratio indices were calculated from the results of the bulk and tapped density that had been calculated.

2.2.3. Fourier Transformed Infrared (FTIR)

Fourier Transform Infrared Spectroscopy (FTIR) testing of microcrystalline cellulose from corn husks was used to determine the functional groups of the corn husk MCC using Agilent Technologies Cary 630 FTIR with Attenuated Total Reflectance (ATR).

2.2.4. Scanning Electrone Microscope (SEM)

Scanning Electron Microscope-Energy Dispersive X-Ray (SEM-EDX) MCC testing of corn husks was used to determine the morphological shape as well as to analyze the elements contained in the sample using the Scanning Electron Microscope-Energy Dispersive X-Ray microscope (JEOLJSM-6510LA).

2.2.5. X-Ray Diffraction (XRD)

X-Ray Diffraction (XRD) analysis of MCC from corn husks was used to determine the crystallinity index produced by corn husk MCC using X-Ray Diffraction (D8 ADVANC X-Ray Diffraction) [24].

2.2.6. Particle Size Analyzer (PSA)

Particle Size Analyzer (PSA) is used to determine the particle size distribution of corn husk MCC using the Particle Size Analyzer tool (Malvern® Mastersizer 3000 (Malvern instrument UK) [25].

3. RESULTS AND DISCUSSION

3.1. Physicochemical Characterization of MCC Corn Husk

The results of determining cellulose content using the Chasson-datta method were obtained from the average cellulose content of corn husk powder of 42.90%, the results obtained were close to the literature that corn husks have a cellulose content of 44.08% [3]. The yield of cellulose content in corn husk α-cellulose increased by 74.02% due to alkalization treatment with NaOH which caused the loss of lignin, mainly due to the unstable ester bond between cellulose and lignin complex, so that lignin that loosely binds to alkali to form a water-soluble alkaline lignin complex. NaOH can break the bond between cellulose with hemicellulose and lignin, causing changes in cellulose levels to increase [17]. The result concentration of cellulose MCC corn husks with HCl 2 N 84.48% and 4 N 86.55% and there was a decreased in 6 N 84.44% (Table 1). The decrease in cellulose levels that occur is caused by the higher concentration of HCl causing an increase in heat (heat) causing the cellulose structure to open up so that cellulose molecules are dispersed freely in the solution, this freely dispersed cellulose structure results in the presence of dissolved cellulose carried away in the solution when the filtration process is carried out [26].

Table 1. Physical Chemical Characterization Test of MCC Corn Husk

Type of Assay		**Result**				**Limit Requirements**
		MCC Corn Husk with HCL 2 N	**MCC Corn Husk with HCL 4 N**	**MCC Corn Husk with HCL 6 N**	**Standard Commercial (Avicel PH 102)**	
Determination of Cellulose Levels (%)		84.48±2.99	86.55±0.91	84.44±2.34	80.81±1.14	80.81
Moisture Content (%)		5.82±0.41	5.66±0.29	3.33±0.93	4.93±0.11	<5
pH		6±0	6±0	6±0	6±0	5-7.5
Melting Point (°C)		299.67±0.58	299.67±0.58	270.66±0.58	315.33±0.58	260-270
Flow rate (g/s)		19.87±3.16	27.66±3.30	31.20±5.12	29.104±3.32	1.41
Angle of Repose (°)		29.59±1.01	28.45±1.12	25.98±3.14	45.27±1.22	34.4-49
Bulk Density (g/mL)		0.341±0.02	0.397±0.01	0.617±0.13	0.371±0.01	0.337 g/cm^3
Tapped Density (g/mL)		0.460±0.005	0.532±0.03	0.751±0.13	0.457±0.002	0.478 g/cm^3
True density (g/mL)		1.401±0.05	1.399±0.03	1.512±0.08	1.466±0.04	1.512-1.668 g/cm^3
Hausner Ratio		1.35±0.07	1.34±0.14	1.22±0.06	1.23±0.02	1.00-1.11 = Very Good
Carr's Index (%)		25.98±3.57	25.15±7.79	17.99±3.74	18.67±1.53	1-10 = Very Good
Levels (%)	Chlor	0.15	0.25	0.35	0.10	0.10%; 0.36% & 0.24%
	Calcium	0.26	0.49	0.92	0.36	
	Natrium	0.13	0.09	0.13	0.24	
CrI (%)		34.1	34.7	34.3	34.5	34.5%
Particle size (µm)		362	362	395	332	20-200 µm

The results of the MCC moisture content test from corn husks (Table 1.). The MCC samples treated with 2 N and 4 N HCl showed moisture content values close to that of commercial MCC (pH 102), which has a reference moisture content of 5.37%. The moisture content of MCC treated with 6 N HCl aligns with literature values, which are typically below 5%. If the moisture content is relatively high, it can increase the cohesion between similar particles, causing the powder to lose its ability to flow properly [27]. pH MCC corn husk and the comparator, Avicel PH 102 (Table1.), also exhibited the same pH value of 6, which is consistent with the literature pH range is between 5 and 7.5 [20]. The results of the MCC melting point test of corn husks at each HCl concentration gave results in the hydrolysis treatment with 2 and 4 N HCl, it was higher than the limit requirement because there were still many crystalline forms of cellulose that were still bound to the amorphous form, while with 6N HCl, the results showed that they were comparable to the limit requirement of 260°C to 270°C [20].

The flow rate of MCC corn husk is better than Avicel PH 102 as a commercial standard (Table 1), because a good flow rate is indicated by a value greater than 10 g/s. This shows that increasing the concentration of HCl can affect the density and particle size of MCC. Powders with smaller particle sizes tend to have poor flowability due to the larger surface area per unit mass, which increases contact between particles. This greater contact increases cohesive and frictional forces, thus inhibiting the flow of the powder [28]. The results of the angle of repose for MCC corn husk show that, on average, the faster the flow of MCC, the smaller the angle of repose formed. This is believed to be due to the larger particle size and low cohesiveness of the powder, which contribute to its good flow properties. Smaller particle size, higher cohesiveness, and greater frictional forces, thus inhibiting the flow of the powder [29].

The results of bulk and tapping density of MCC corn husks showed that samples treated with 2 N and 4 N HCl, as well as Avicel PH 102, produced values close to the limit requirements (Table 1). MCC corn husks treated with 6 N HCl produced higher values compared to Avicel PH 102 and the literature. The actual density of MCC corn husks treated with 6 N HCl was within the limit requirement range of 1.512–1.668 g/cm³ [20], while MCC treated with 2 N, 4 N HCl, and Avicel PH 102 showed lower actual density values than those reported. The hausner ratio value for MCC corn husks treated with 2 N, 4 N, and 6 N HCl concentrations was comparable to Avicel PH 102. The higher concentration of HCl used in the hydrolysis process had an effect on reducing the carr's index and hausner ratio. MCC corn husks resulting from hydrolysis with 6 N HCl showed better flow properties and compressibility compared to Avicel PH 102. The amorphous form of cellulose is very susceptible to HCl so that the higher the concentration of HCl, the more the amorphous form is lost so that denser cellulose crystals will be formed with fewer cavities on the particle surface as seen in the morphology from the SEM analysis results (Figure 2.). The number of cavities on the particle surface can create space between particles which affects the increase in porosity and bulk volume so that its flowability is low [30].

3.2. Fourier Transformed Infrared (FTIR)

FTIR Spectra of MCC corn huks (Figure 1.) showed the presence of characteristic cellulose absorption bands. The absorption band at wavelengths of 3500–3250 cm⁻¹ indicates the O–H stretching vibration of α-cellulose, while the band at 2970–2850 cm⁻¹ corresponds to the C–H stretching vibration, further confirming the presence of α-cellulose [31]. Additionally, the absorption band at 900–800 cm⁻¹ indicates the presence of β-glycosidic linkages, which are characteristic of microcrystalline cellulose (MCC) [32].

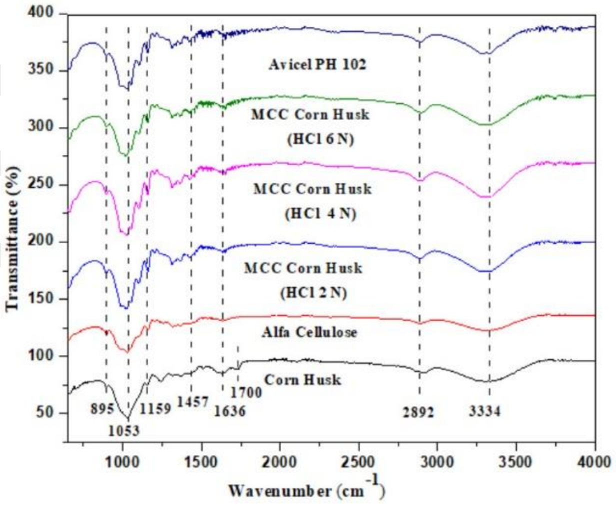

Figure 1. FTIR Spectra of Corn Husk, alfa cellulose, MCC Corn Husk and Avicel PH 102

FTIR analysis also revealed the presence of a lignin absorption band at around 1700 cm⁻¹ in raw corn husk powder that had not been treated with NaOH, indicating the presence of lignin prior to the delignification process. In contrast, the FTIR spectra of MCC derived from corn husks treated with HCl concentrations of 2 N, 4 N, and 6 N showed similar spectral patterns to that of Avicel PH 102. These spectra confirmed the presence of cellulose, while the absorption bands associated with hemicellulose and lignin were no longer observed in the MCC samples and Avicel PH 102. This indicates that the non-cellulosic components were effectively removed during the delignification and purification processes, leaving behind primarily α-cellulose [17].

3.3. Scanning Electrone Microscopy (SEM)

The morphological observation of raw corn husk powder revealed a denser surface structure, which is attributed to the presence of lignin still embedded in the cell wall, serving to protect the cellulose. In contrast, the morphology of α-cellulose showed the initial stages of solid peeling, leading to the formation of irregular fibrous structures. MCC derived from corn husks treated with 2 N and 4 N HCl exhibited elongated, stem-like shapes with uneven surfaces, slightly hollow structures, and distinguishable blunt-angled edges (Figure 2).

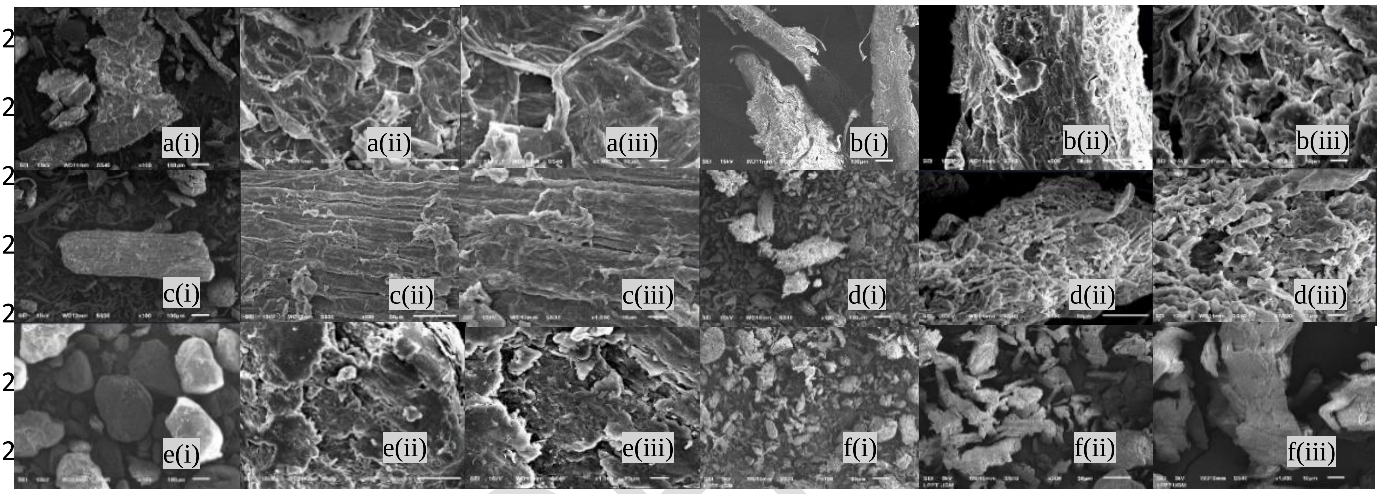

Figure 1. SEM Image of (a) Powder Corn Husk; (b) α-Cellulose; (c) MCC Corn Husk (HCl 2 N); (d) MCC Corn Husk (HCl 4 N); (e) MCC Corn Husk (HCl 6 N); (f) Avicel pH 102; with magnification (i) 100x; (ii) 500x; (iii) 1000x.

The comparator, Avicel PH 102, displayed irregularly shaped particles with varying lengths, uneven and slightly hollow surfaces, and both pointed and blunt edges [33]. MCC obtained using 6 N HCl showed a more compact, spherical, and granular morphology compared to MCC 2 N, 4 N, and Avicel PH 102. It also had a smoother surface and blunt angles. Morphology MCC plays an important role in influencing flow properties [9,21,25,30]. In addition to the morphological results of the Scanning Electron Microscope–Energy Dispersive X-Ray (SEM-EDX) analysis, it shows that the chlorine, calcium and sodium levels of corn husk MCC (Figure 3).

3.4. X-Ray Diffraction (XRD)

X-Ray Diffraction (XRD) analysis of MCC corn husks showed that the crystallinity of the samples increased with higher HCl concentrations (Table 1). This increase in crystallinity is due to the loss of the lignin layer from the corn husk sample as evidenced by the absence of a peak at 2θ=24.2° so that α-cellulose remains and it is indicated that there is no peak related to semicrystalline cellulose in the corn husk MCC due to the loss of amorphous properties during hydrolysis with increasing HCl concentrations shown in Figure 4 with an increase in peak intensity at 2θ= ± 20° [34-35]. However, at a 6 N HCl concentration, the crystallinity index decreased to 34.3% (Table 1.). This reduction is likely due to the high concentration of HCl, which, through the application of heat, caused the crystalline regions of the corn husk MCC to undergo hydrolysis, converting them into amorphous regions and thus reducing the overall crystallinity [36].

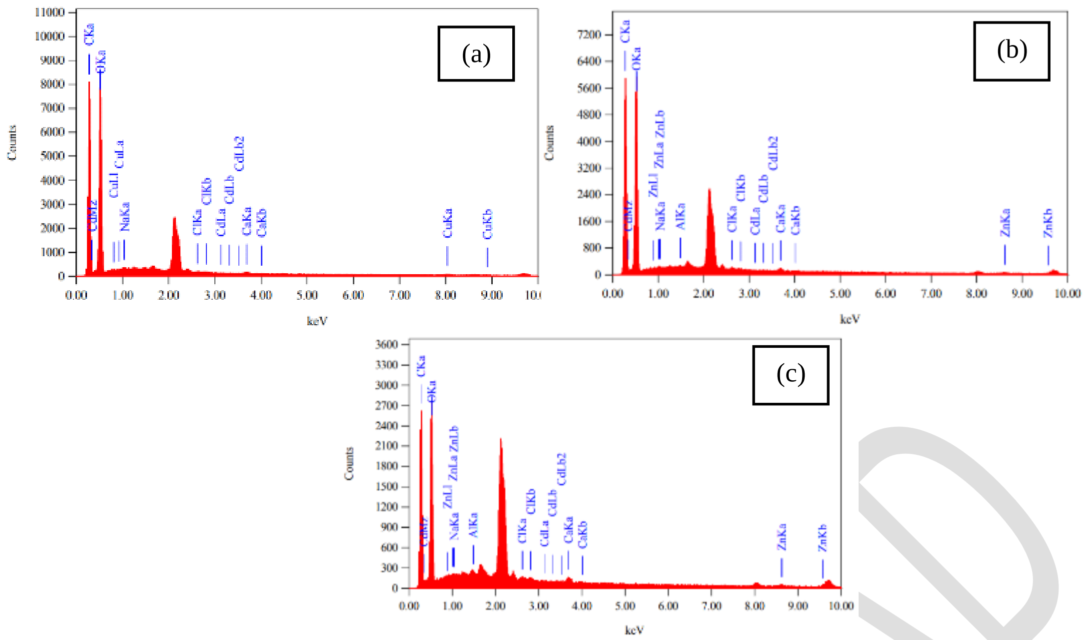

Figure 2. Test SEM-EDX Elements Klor, Calcium, Natrium (a) MCC Corn Husk (HCl 2 N); (b) MCC Corn Husk (HCl 4 N); (c) MCC Corn Husk (HCl 6 N)

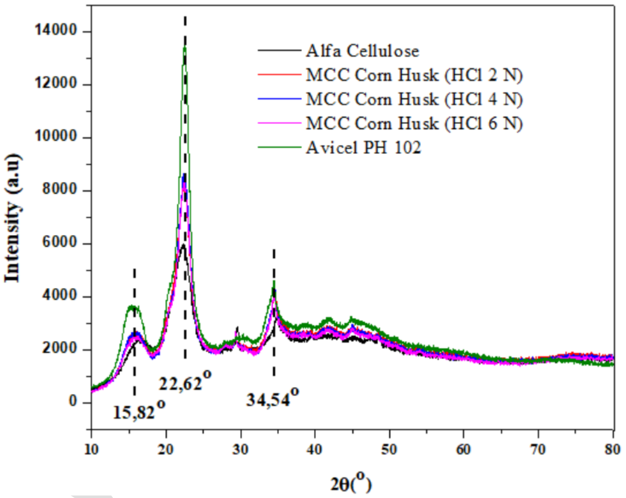

Figure 3. X-ray diffraction patterns of Corn Husk, alfa cellulose, MCC Corn Husk and Avicel PH 102

3.5. Particle Size Analyzer (PSA)

Table 2. Average Particle Size Analyzer MCC Corn Husk

Sample	Dx (10) (μm)	Dx (50) (μm)	Dx (90) (μm)
Avicel pH 102	37.9	139	332
MCC 1	36.1	144	362
MCC 2	26.5	142	362
MCC 3	84.1	216	395
Mean	46.1	160	363
1xStd Dev	25.8	37.2	25.4
1xRSD (%)	55.8	23.3	7.01

Note: Dx 10 – the size of particle below which 10% of the sample lies, Dx 50 (50%) and Dx 90 (90%).

The results of the Particle Size Analysis (Table 2), it can be concluded that larger particle sizes result in better flowability of the MCC derived from corn husks. This improvement in flow rate is attributed to the stronger gravitational force acting on the larger particles, which outweighs the tensile forces between the powder particles. Additionally, the reduced friction between particles makes it easier for the powder to flow [37].

4. CONCLUSION

The results of the physicochemical characterization tests conducted on the three MCC samples showed FTIR absorption patterns similar to that of Avicel PH 102. The surface morphology of the corn husk MCC particles varied with HCl concentration, with the highest concentration (6 N) resulting in round, dense, and more granular particles compared to those obtained at lower concentrations. The crystallinity index of α-cellulose for the three MCC samples was as follows: 30.7% for the raw corn husk, 34.1% for the 2 N HCl-treated sample, 34.7% for the 4 N HCl treated sample, and 34.3% for the 6 N HCl-treated sample. The particle size distribution for the three MCC samples at Dx 90 was 362 µm, 362 µm, 392 µm, and 332 µm, respectively. The difference in HCl concentration during the hydrolysis process contributes to various characteristics of corn husk MCC. The results of the analysis consistently show that hydrolysis with 6N HCl can produce MCC corn husks with pharmaceutical grade characteristics so that they can be used as an alternative excipient by the pharmaceutical industry which can realize the independence of the national raw material industry. Therefore, corn husks powder has the potential to become an alternative source for microcrystalline cellulose fabrication which is expected to not only address the problem of raw material needs for the pharmaceutical industry but also the problem of waste.

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Conflicts of interest: The authors declare no conflict of interest.

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Original Article

Pharmaceutical Grade Microcrystalline Cellulose from Corn Husk (*Zea mays* L.): Fabrication and Characterization

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Abstract: Corn is a plant that grows easily in tropical climates. Corn production in Indonesia reaches 25.18 tons, the use of which in society is still limited to corn kernels as food, while other parts of the corn plant are waste. Corn husks are an abundant natural waste and contain 44.08% cellulose, so they can potentially be a source of pharmaceutical excipients, namely microcrystalline cellulose (MCC). This research aims to isolate and characterize MCC from pharmaceutical grade corn husks with commercial MCC as a comparator. The two methods of making MCC are delignification using 2% NaOH at 80-90°C 4 h. Hydrolysis using variations in HCl concentrations, namely 2 N, 4 N, and 6 N, at a temperature of 80°C 4 h. The research results obtained cellulose content in α-cellulose and MCC of corn husks with 3 consecutive treatments of 74.02%, 84.48%, 86.55%, and 84.44%. The result of the analysis test of FTIR, SEM, XRD, and PSA instruments indicate that corn husk MCC has characteristics of commercial MCC as a standard. The resulting corn husk MCC has physicochemical characteristics according to standards that can be used as a pharmaceutical excipient.

Keywords: characterization, fabrication, microcrystalline cellulose, corn husk, pharmaceutical excipient

1. INTRODUCTION

Corn plants are a staple food that is widely consumed after rice [1]. Corn kernels used in the food sector are only able to represent 5% of the total part of the corn plant; the remaining 95% of the corn plant is in the category of natural waste in the form of stalks, leaves, cobs, and corn husks [2]. The Pharmaceutical Industry in Indonesia still uses 95% of drug raw materials imported from abroad. Corn husks are part of the corn crop waste that has not been utilized optimally and contain quite high cellulose, which is 44.08% [3]. The high cellulose content is a consideration for developing its benefits and potential to be used as pharmaceutical excipient [4].

Microcrystalline Cellulose (MCC) is pure cellulose that has been isolated using mineral acids from α-cellulose fibrous plants. MCC is widely used as the best excipient in the manufacture of direct printed tablets. In the manufacture of tablets using of direct compression method, MCC is used as a dry binder, tablet disintegrant, filler, or thinner, absorbent, lubricant, and anti-adherent. MCC is widely used as an excipient in the manufacture of direct print tablets because it has good flow properties and compatibility [5,6].

MCC can be made by delignification and then hydrolysis. Delignification is carried out to change the structure of lignocellulose biomass, which aims to degrade lignin polymers bound to cellulose, then lignin will dissolve in water, and the result is α-cellulose. Delignification of α-cellulose powder was subjected to controlled hydrolysis using an acidic solution. Acid hydrolysis can damage the amorphous region of the cellulose microfibrils, where the amorphous form will undergo disconnection and then leave a crystalline [7,8]. Several studies on the use of HCl in hydrolysis in the manufacture of MCC from other natural materials have been able to increase the yield and crystallinity index [9–12].

The pharmaceutical industry in Indonesia is still dependent on imported raw materials (95%) [13]. The raw materials here are not only active ingredients but also excipients that play an important role in determining the quality of the dosage form. The abundant corn crop yield (38.38%) means that the amount of corn husk waste produced also increases [14]. It is necessary to develop cellulose technology for high in corn husks (44.08%) [3] into MCC as an alternative pharmaceutical excipient native to Indonesia that not only solves the problem of meeting the needs of raw materials for the pharmaceutical industry but also solves the problem of plantation waste. Several studies on the isolation of MCC from corn waste that have been carried out include corn cobs with variations of NaOH in the delignification process and hydrolysis with 10% H_2SO4 obtained a yield of 30% and CrI 91.26% [15]. Hydrolysis of pulut corn husks with 2.5N HCl for 10 minutes produced MCC with CrI 79% [16]. However, this study was limited to CrI and morphology analysis, so it is necessary to conduct research on the fabrication and physicochemical and mechanical characterization of MCC corn husks compared to Avicel PH 102 as a commercial standard so that it can guarantee its quality as a pharmaceutical excipient.

2. MATERIALS AND METHODS

2.1. Materials

Corn husks from plantation waste in the Semarang area (Indonesia) which is dried and ground with 40 mesh. Technical grade material: sodium hydroxide (NaOH) (Hangzhou Lizu Co., Ltd), sodium hypochlorite (NaOCl) (Asahimas), hydrochloric acid (HCl) (Tjiwi Kimia). Pro-analysis material (Merck): sulfuric acid (H_2SO_4), ethanol, iodized zinc chloride solution (zinc chloride, potassium iodide and iodine) and iodine. Pharmaceutical grade material: Avicel PH 102 (American International Chemical/AIC, Inc-Framingham USA) as commercial standard.

2.2. Methods

2.2.1. Fabrication of MCC Corn Husk

a. Alkaline Delignification

Delignification of corn husk powder with 2% NaOH at 80-90°C for 4 h, the residue is filtered and washed down to a neutral pH of 6-7. The next stage is bleaching with a solution of NaOCl 5% at 70°C for 1 hour and NaOCl 5% for 24 h at room temperature. The residue is filtered and washed to a neutral pH of 6-7. Cellulose is produced, dried, and mashed [17].

b. Acid Hydrolysis

Hydrolysis of α-cellulose with variations in HCl concentration of 2 N, 4 N, and 6 N for 80°C 4 hours and then filtered and washed until a neutral pH of 6-7. The next stage is bleached 2 times with a 5% NaOCl solution of 70°C for 1 hour and soaked in the same solution for 24 hours at room temperature. The residue is filtered and washed until a neutral pH of 6-7. MCC is dried and smoothed then sifted mesh no. 60 [18].

2.2.2 Physicochemical Characterization of MCC Corn Husk

a. Determination of Cellulose Concentration

Concentration of cellulose was determined using the Chesson-datta method [19].

b. Moisture Content

Determined using a moisture content tool set at a temperature of 150 °C for automatic time to constant weight. The standard requirement for MCC moisture content was not more than 5% [20].

c. pH

MCC corn husks as much as 1 gram added 50 mL aquadest stirring for 5 minutes then measured the pH using a pH instrument [21].

d. Melting Point

MCC is inserted into a capillary pipe and then put into a melting point device (Mettler Toledo) with a temperature of 200°C when the device is switched on and the temperature is deformed when the solids begin to melt.

e. Flow Rate and Angle of Repose

The flow rate of MCC corn husks using a flowability tester (Erweka GT) with a funnel diameter of 15 mm. The cover at the bottom of the funnel is opened and the flow speed is calculated at the time the granule starts flowing until the granule stops flowing using a stopwatch and then the time obtained and the height and diameter of the cone are measured [22].

f. Density, Carr's Index and Hausner Ratio

40 grams of corn husk MCC is placed in a 100 mL measuring cup. The surface of the powder is carefully leveled without being compressed its volume (Vo) measurement is performed. A measuring cup is installed on the support of the tapped density tester, 10, 500, and 1250 taps are carried out and V10, V500, and V1250 are read on the nearest measuring cup unit. Volume measured to the last tap (Vt) [23]. The density of MCC corn husks was determined by dividing weight by Vo (bulk density) and Vt (tapped density). The true density of MCC is determined by determining the volume using a picnometre. Carr's index and hausner ratio indices were calculated from the results of the bulk and tapped density that had been calculated.

2.2.3. Fourier Transformed Infrared (FTIR)

Fourier Transform Infrared Spectroscopy (FTIR) testing of microcrystalline cellulose from corn husks was used to determine the functional groups of the corn husk MCC using Agilent Technologies Cary 630 FTIR with Attenuated Total Reflectance (ATR).

2.2.4. Scanning Electrone Microscope (SEM)

Scanning Electron Microscope-Energy Dispersive X-Ray (SEM-EDX) MCC testing of corn husks was used to determine the morphological shape as well as to analyze the elements contained in the sample using the Scanning Electron Microscope-Energy Dispersive X-Ray microscope (JEOLJSM-6510LA).

2.2.5. X-Ray Diffraction (XRD)

X-Ray Diffraction (XRD) analysis of MCC from corn husks was used to determine the crystallinity index produced by corn husk MCC using X-Ray Diffraction (D8 ADVANC X-Ray Diffraction) [24].

2.2.6. Particle Size Analyzer (PSA)

Particle Size Analyzer (PSA) is used to determine the particle size distribution of corn husk MCC using the Particle Size Analyzer tool (Malvern® Mastersizer 3000 (Malvern instrument UK) [25].

3. RESULTS AND DISCUSSION

3.1. Physicochemical Characterization of MCC Corn Husk

The results of determining cellulose content using the Chasson-datta method were obtained from the average cellulose content of corn husk powder of 42.90%, the results obtained were close to the literature that corn husks have a cellulose content of 44.08% [3]. The yield of cellulose content in corn husk α-cellulose increased by 74.02% due to alkalization treatment with NaOH which caused the loss of lignin, mainly due to the unstable ester bond between cellulose and lignin complex, so that lignin that loosely binds to alkali to form a water-soluble alkaline lignin complex. NaOH can break the bond between cellulose with hemicellulose and lignin, causing changes in cellulose levels to increase [17]. The result concentration of cellulose MCC corn husks with HCl 2 N 84.48% and 4 N 86.55% and there was a decreased in 6 N 84.44% (Table 1). The decrease in cellulose levels that occur is caused by the higher concentration of HCl causing an increase in heat (heat) causing the cellulose structure to open up so that cellulose molecules are dispersed freely in the solution, this freely dispersed cellulose structure results in the presence of dissolved cellulose carried away in the solution when the filtration process is carried out [26].

Table 1. Physical Chemical Characterization Test of MCC Corn Husk

Type of Assay		**Result**				**Limit Requirements**
		MCC Corn Husk with HCL 2 N	**MCC Corn Husk with HCL 4 N**	**MCC Corn Husk with HCL 6 N**	**Standard Commercial (Avicel PH 102)**	
Determination of Cellulose Levels (%)		84.48±2.99	86.55±0.91	84.44±2.34	80.81±1.14	80.81
Moisture Content (%)		5.82±0.41	5.66±0.29	3.33±0.93	4.93±0.11	<5
pH		6±0	6±0	6±0	6±0	5-7.5
Melting Point (°C)		299.67±0.58	299.67±0.58	270.66±0.58	315.33±0.58	260-270
Flow rate (g/s)		19.87±3.16	27.66±3.30	31.20±5.12	29.104±3.32	1.41
Angle of Repose (°)		29.59±1.01	28.45±1.12	25.98±3.14	45.27±1.22	34.4-49
Bulk Density (g/mL)		0.341±0.02	0.397±0.01	0.617±0.13	0.371±0.01	0.337 g/cm^3
Tapped Density (g/mL)		0.460±0.005	0.532±0.03	0.751±0.13	0.457±0.002	0.478 g/cm^3
True density (g/mL)		1.401±0.05	1.399±0.03	1.512±0.08	1.466±0.04	1.512-1.668 g/cm^3
Hausner Ratio		1.35±0.07	1.34±0.14	1.22±0.06	1.23±0.02	1.00-1.11 = Very Good
Carr's Index (%)		25.98±3.57	25.15±7.79	17.99±3.74	18.67±1.53	1-10 = Very Good
Levels (%)	Chlor	0.15	0.25	0.35	0.10	0.10%; 0.36% & 0.24%
	Calcium	0.26	0.49	0.92	0.36	
	Natrium	0.13	0.09	0.13	0.24	
CrI (%)		34.1	34.7	34.3	34.5	34.5%
Particle size (µm)		362	362	395	332	20-200 µm

The results of the MCC moisture content test from corn husks (Table 1.). The MCC samples treated with 2 N and 4 N HCl showed moisture content values close to that of commercial MCC (pH 102), which has a reference moisture content of 5.37%. The moisture content of MCC treated with 6 N HCl aligns with literature values, which are typically below 5%. If the moisture content is relatively high, it can increase the cohesion between similar particles, causing the powder to lose its ability to flow properly [27]. pH MCC corn husk and the comparator, Avicel PH 102 (Table1.), also exhibited the same pH value of 6, which is consistent with the literature pH range is between 5 and 7.5 [20]. The results of the MCC melting point test of corn husks at each HCl concentration gave results in the hydrolysis treatment with 2 and 4 N HCl, it was higher than the limit requirement because there were still many crystalline forms of cellulose that were still bound to the amorphous form, while with 6N HCl, the results showed that they were comparable to the limit requirement of 260°C to 270°C [20].

The flow rate of MCC corn husk is better than Avicel PH 102 as a commercial standard (Table 1), because a good flow rate is indicated by a value greater than 10 g/s. This shows that increasing the concentration of HCl can affect the density and particle size of MCC. Powders with smaller particle sizes tend to have poor flowability due to the larger surface area per unit mass, which increases contact between particles. This greater contact increases cohesive and frictional forces, thus inhibiting the flow of the powder [28]. The results of the angle of repose for MCC corn husk show that, on average, the faster the flow of MCC, the smaller the angle of repose formed. This is believed to be due to the larger particle size and low cohesiveness of the powder, which contribute to its good flow properties. Smaller particle size, higher cohesiveness, and greater frictional forces, thus inhibiting the flow of the powder [29].

The results of bulk and tapping density of MCC corn husks showed that samples treated with 2 N and 4 N HCl, as well as Avicel PH 102, produced values close to the limit requirements (Table 1). MCC corn husks treated with 6 N HCl produced higher values compared to Avicel PH 102 and the literature. The actual density of MCC corn husks treated with 6 N HCl was within the limit requirement range of 1.512–1.668 g/cm^3 [20], while MCC treated with 2 N, 4 N HCl, and Avicel PH 102 showed lower actual density values than those reported. The hausner ratio value for MCC corn husks treated with 2 N, 4 N, and 6 N HCl concentrations was comparable to Avicel PH 102. The higher concentration of HCl used in the hydrolysis process had an effect on reducing the carr's index and hausner ratio. MCC corn husks resulting from hydrolysis with 6 N HCl showed better flow properties and compressibility compared to Avicel PH 102. The amorphous form of cellulose is very susceptible to HCl so that the higher the concentration of HCl, the more the amorphous form is lost so that denser cellulose crystals will be formed with fewer cavities on the particle surface as seen in the morphology from the SEM analysis results (Figure 2.). The number of cavities on the particle surface can create space between particles which affects the increase in porosity and bulk volume so that its flowability is low [30].

3.2. Fourier Transformed Infrared (FTIR)

FTIR Spectra of MCC corn huks (Figure 1.) showed the presence of characteristic cellulose absorption bands. The absorption band at wavelengths of 3500–3250 cm^{-1} indicates the O–H stretching vibration of α-cellulose, while the band at 2970–2850 cm^{-1} corresponds to the C–H stretching vibration, further confirming the presence of α-cellulose [31]. Additionally, the absorption band at 900–800 cm^{-1} indicates the presence of β-glycosidic linkages, which are characteristic of microcrystalline cellulose (MCC) [32].

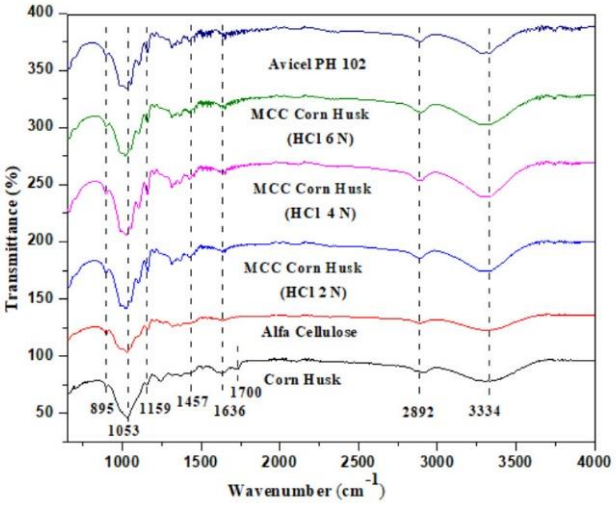

Figure 1. FTIR Spectra of Corn Husk, alfa cellulose, MCC Corn Husk and Avicel PH 102

FTIR analysis also revealed the presence of a lignin absorption band at around 1700 cm^{-1} in raw corn husk powder that had not been treated with NaOH, indicating the presence of lignin prior to the delignification process. In contrast, the FTIR spectra of MCC derived from corn husks treated with HCl concentrations of 2 N, 4 N, and 6 N showed similar spectral patterns to that of Avicel PH 102. These spectra confirmed the presence of cellulose, while the absorption bands associated with hemicellulose and lignin were no longer observed in the MCC samples and Avicel PH 102. This indicates that the non-cellulosic components were effectively removed during the delignification and purification processes, leaving behind primarily α-cellulose [17].

3.3. Scanning Electrone Microscopy (SEM)

The morphological observation of raw corn husk powder revealed a denser surface structure, which is attributed to the presence of lignin still embedded in the cell wall, serving to protect the cellulose. In contrast, the morphology of α-cellulose showed the initial stages of solid peeling, leading to the formation of irregular fibrous structures. MCC derived from corn husks treated with 2 N and 4 N HCl exhibited elongated, stem-like shapes with uneven surfaces, slightly hollow structures, and distinguishable blunt-angled edges (Figure 2).

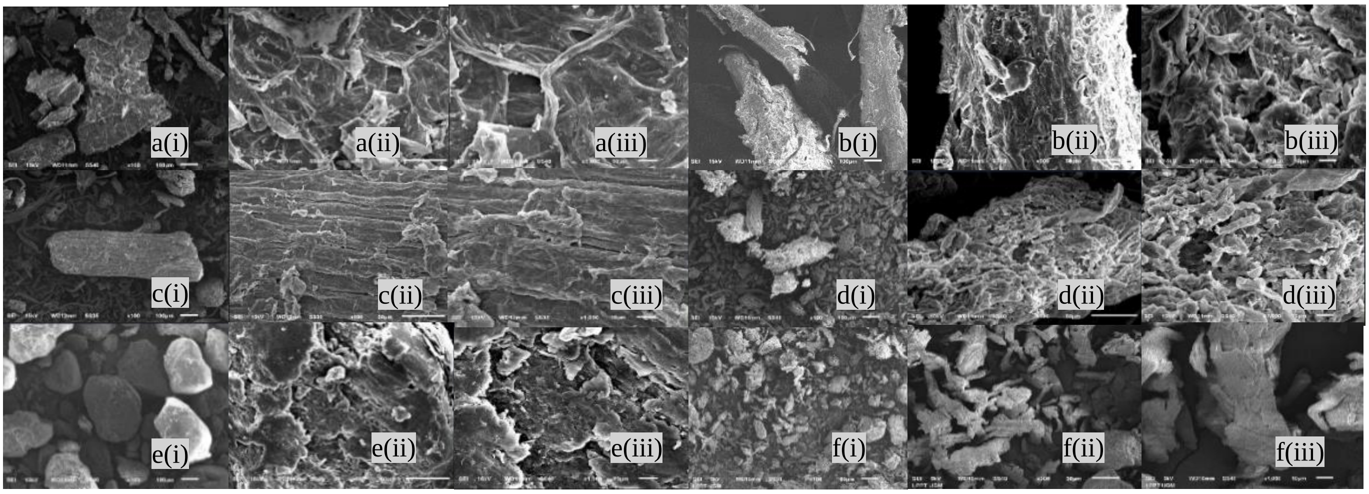

Figure 1. SEM Image of (a) Powder Corn Husk; (b) α-Cellulose; (c) MCC Corn Husk (HCl 2 N); (d) MCC Corn Husk (HCl 4 N); (e) MCC Corn Husk (HCl 6 N); (f) Avicel pH 102; with magnification (i) 100x; (ii) 500x; (iii) 1000x.

The comparator, Avicel PH 102, displayed irregularly shaped particles with varying lengths, uneven and slightly hollow surfaces, and both pointed and blunt edges [33]. MCC obtained using 6 N HCl showed a more compact, spherical, and granular morphology compared to MCC 2 N, 4 N, and Avicel PH 102. It also had a smoother surface and blunt angles. Morphology MCC plays an important role in influencing flow properties [9,21,25,30]. In addition to the morphological results of the Scanning Electron Microscope–Energy Dispersive X-Ray (SEM-EDX) analysis, it shows that the chlorine, calcium and sodium levels of corn husk MCC (Figure 3).

3.4. X-Ray Diffraction (XRD)

X-Ray Diffraction (XRD) analysis of MCC corn husks showed that the crystallinity of the samples increased with higher HCl concentrations (Table 1). This increase in crystallinity is due to the loss of the lignin layer from the corn husk sample as evidenced by the absence of a peak at $2\theta=24.2°$ so that α-cellulose remains and it is indicated that there is no peak related to semicrystalline cellulose in the corn husk MCC due to the loss of amorphous properties during hydrolysis with increasing HCl concentrations shown in Figure 4 with an increase in peak intensity at $2\theta= \pm 20°$ [34-35]. However, at a 6 N HCl concentration, the crystallinity index decreased to 34.3% (Table 1.). This reduction is likely due to the high concentration of HCl, which, through the application of heat, caused the crystalline regions of the corn husk MCC to undergo hydrolysis, converting them into amorphous regions and thus reducing the overall crystallinity [36].

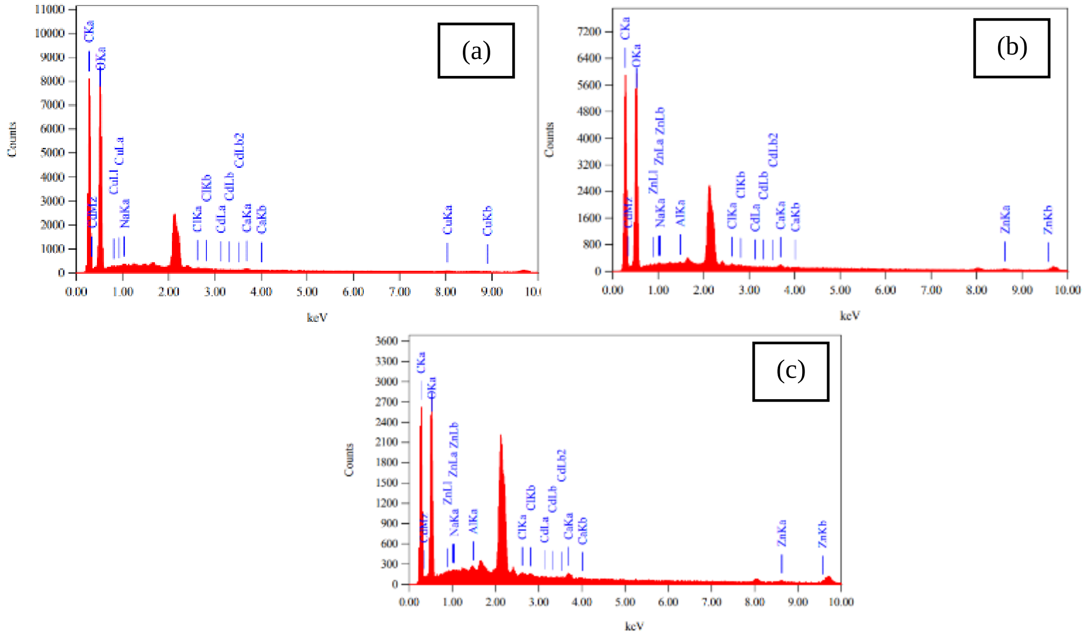

Figure 2. Test SEM-EDX Elements Klor, Calcium, Natrium (a) MCC Corn Husk (HCl 2 N); (b) MCC Corn Husk (HCl 4 N); (c) MCC Corn Husk (HCl 6 N)

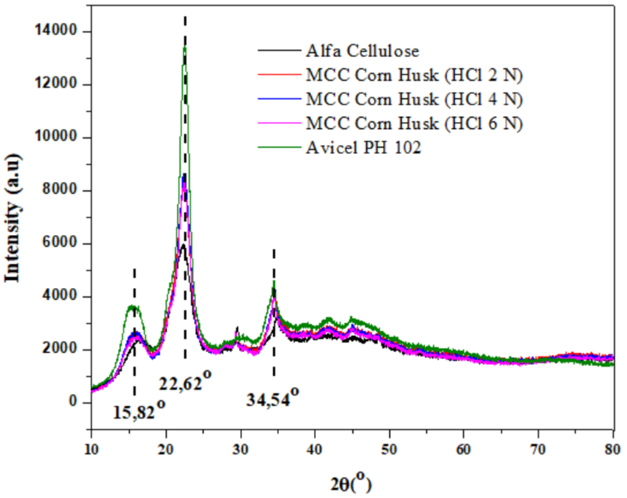

Figure 3. X-ray diffraction patterns of Corn Husk, alfa cellulose, MCC Corn Husk and Avicel PH 102

3.5. Particle Size Analyzer (PSA)

Table 2. Average Particle Size Analyzer MCC Corn Husk

	Sample	Dx (10) (μm)	Dx (50) (μm)	Dx (90) (μm)
	Avicel pH 102	37.9	139	332
	MCC 1	36.1	144	362
	MCC 2	26.5	142	362
	MCC 3	84.1	216	395
Mean		46.1	160	363
1xStd Dev		25.8	37.2	25.4
1xRSD (%)		55.8	23.3	7.01

Note: Dx 10 – the size of particle below which 10% of the sample lies, Dx 50 (50%) and Dx 90 (90%).

The results of the Particle Size Analysis (Table 2), it can be concluded that larger particle sizes result in better flowability of the MCC derived from corn husks. This improvement in flow rate is attributed to the stronger gravitational force acting on the larger particles, which outweighs the tensile forces between the powder particles. Additionally, the reduced friction between particles makes it easier for the powder to flow [37].

4. CONCLUSION

The results of the physicochemical characterization tests conducted on the three MCC samples showed FTIR absorption patterns similar to that of Avicel PH 102. The surface morphology of the corn husk MCC particles varied with HCl concentration, with the highest concentration (6 N) resulting in round, dense, and more granular particles compared to those obtained at lower concentrations. The crystallinity index of α-cellulose for the three MCC samples was as follows: 30.7% for the raw corn husk, 34.1% for the 2 N HCl-treated sample, 34.7% for the 4 N HCl treated sample, and 34.3% for the 6 N HCl-treated sample. The particle size distribution for the three MCC samples at Dx 90 was 362 μm, 362 μm, 392 μm, and 332 μm, respectively. The difference in HCl concentration during the hydrolysis process contributes to various characteristics of corn husk MCC. The results of the analysis consistently show that hydrolysis with 6N HCl can produce MCC corn husks with pharmaceutical grade characteristics so that they can be used as an alternative excipient by the pharmaceutical industry which can realize the independence of the national raw material industry. Therefore, corn husks powder has the potential to become an alternative source for microcrystalline cellulose fabrication which is expected to not only address the problem of raw material needs for the pharmaceutical industry but also the problem of waste.

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