

BUKTI KORESPONDENSI**JURNAL NASIONAL TERAKREDITASI KEMENRISTEKDIKTI PERINGKAT 2**

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

Jurnal : Journal of Food and Pharmaceutical Sciences

Edisi : Tahun 2025, Vol 13, No.2 Halaman 82-91

Penerbit : Integrated Research and Testing Laboratory, Universitas Gadjah Mada (LPPT-UGM)

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

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Pengusul


Dr. apt. Intan Martha Cahyani, M.Sc

1. Manuscript Submission

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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

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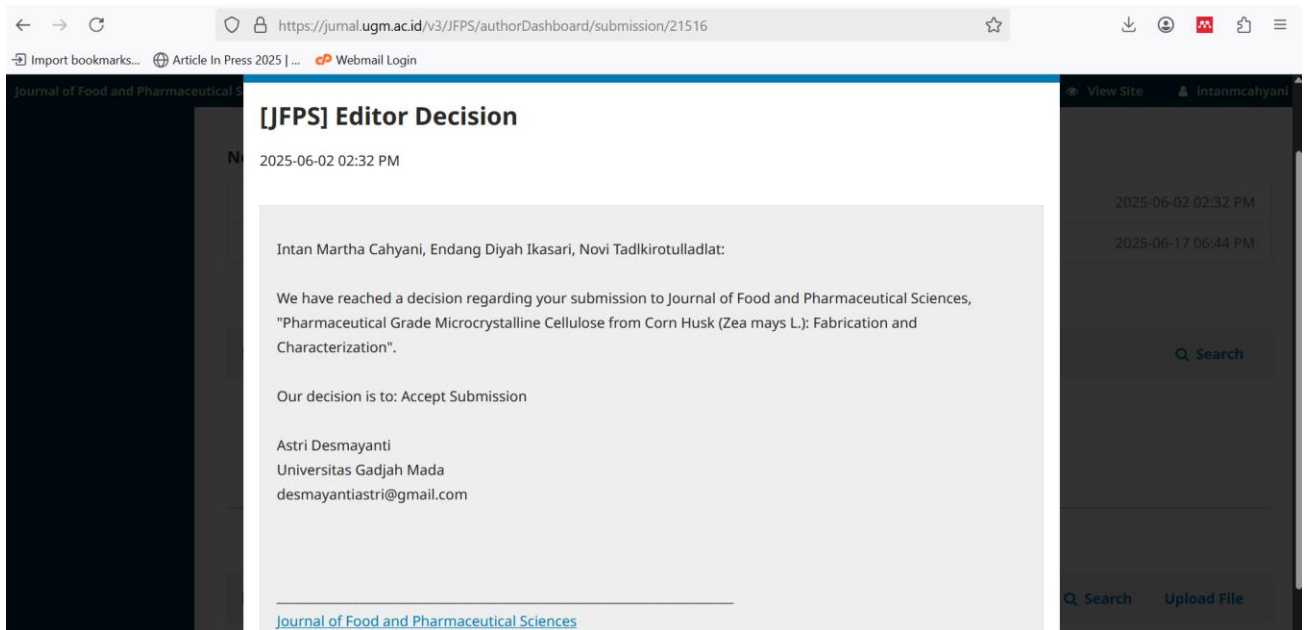
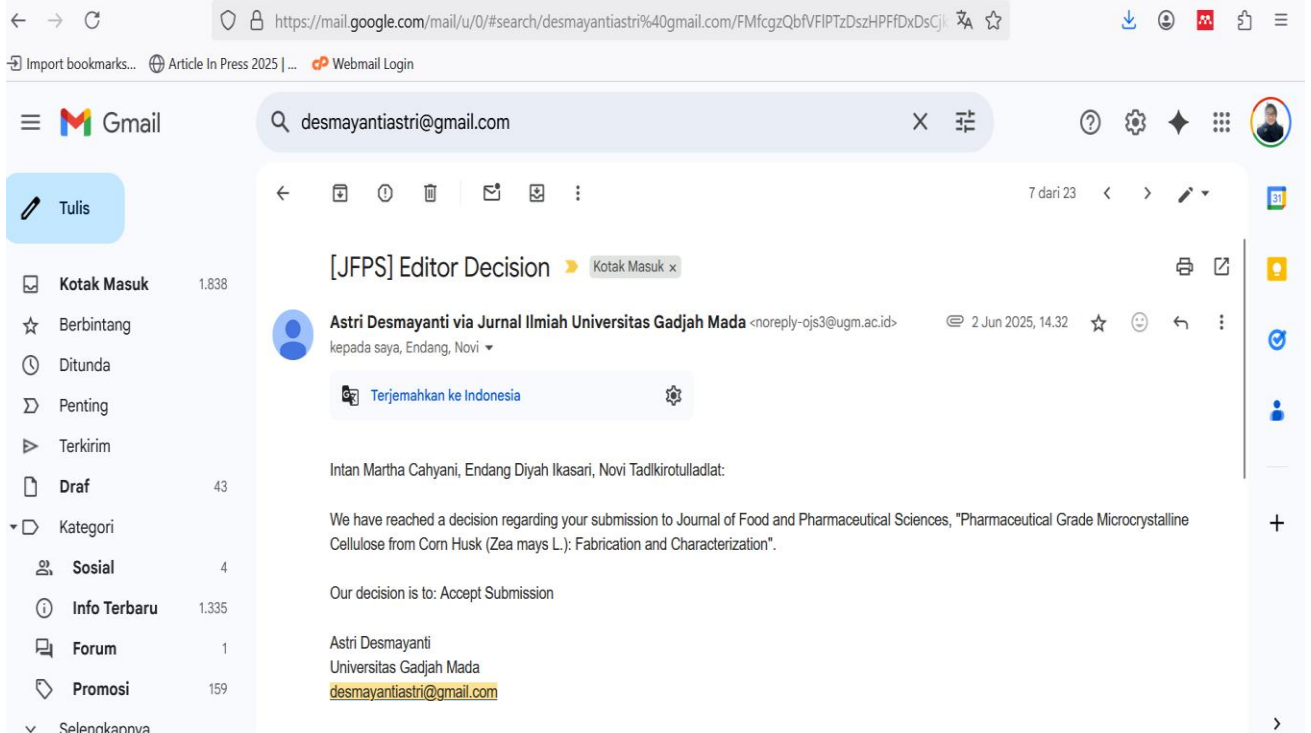
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5. Editor Decision: Accept Submission (Article Acceptance)



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 Diah 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

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

The screenshot shows a web browser window with the URL <https://jurnal.ugm.ac.id/v3/JFPS/authorDashboard/submission/21516>. The page title is "[JFPS] Editor Decision". The email content is as follows:

2025-06-17 06:44 PM

Intan Martha Cahyani, Endang Diyah Ikasari, Novi Tadkirotulladlat:

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>

Astri Desmayanti
Universitas Gadjah Mada
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7. Check Galley Proof

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8. Confirm Final Proofreading Approval

The screenshot shows an email interface with a message from "intanmcahyani" dated "2025-06-19 02:11 PM". The message text reads: "Dear editor Thank you for sending the proofreading file, and we have read all parts of the file according to the specifications. Best regards".

9. Published Information by email

The screenshot shows a Gmail interface with a notification email from "Astri Desmayanti via Jurnal Ilmiah Universitas Gadjah Mada". The email subject is "[JFPS] New notification from Journal of Food and Pharmaceutical Sciences". The body text reads: "You have a new notification from Journal of Food and Pharmaceutical Sciences: An issue has been published. Link: <https://jurnal.ugm.ac.id/v3/JFPS/issue/current> Prof. Dr. Abdul Rohman, M.Si., Apt".

10. Article Published Vol 13 issue 2 (30 Juni 2025) on website

The screenshot shows the top portion of the journal's website. The browser address bar displays the URL <https://jurnal.ugm.ac.id/v3/JFPS/article/view/21516>. The journal's logo, "jfoodpharmsci", is prominently displayed in the center, with the full name "Journal of Food and Pharmaceutical Sciences" above it. The author's name, "Intan Martha Cahyani", is listed in the top right corner. Below the logo, a navigation menu includes links for HOME, ABOUT, EDITORIAL TEAM, REVIEWER, PUBLICATION ETHICS, ANNOUNCEMENTS, CURRENT, ARCHIVES, and ARCHIVES VOL 1-6. A search bar is also present. The main content area features the article title: "Pharmaceutical Grade Microcrystalline Cellulose from Corn Husk (Zea mays L.): Fabrication and Characterization". The author's name, "Intan Martha Cahyani", is listed below the title, along with her affiliation: "Stifar Yayasan Farmasi, Letjend Sarwo Edie Wibowo KM 1, Semarang, 50192, Indonesia" and her ORCID iD: <https://orcid.org/0000-0001-9870-1048>. On the right side, there are buttons for "New Submission", "JOURNAL MENU", "Focus & Scope", "Open Access Policy", and "Copyright Notice".

The screenshot shows the lower portion of the journal's website. The browser address bar displays the URL <https://jurnal.ugm.ac.id/v3/JFPS/article/view/21516>. The article title, "Pharmaceutical Grade Microcrystalline Cellulose from Corn Husk (Zea mays L.): Fabrication and Characterization", is repeated at the top. Below the title, the authors' names and affiliations are listed: "Endang Diyah Ikasari", "Novi Tadkirotulladlat", and "Novita Sindy Anggraini", all from "Stifar Yayasan Farmasi, Letjend Sarwo Edie Wibowo KM 1, Semarang, 50192, Indonesia". The DOI is provided as <https://doi.org/10.22146/jfps.21516>. The keywords are: "characterization, fabrication, microcrystalline cellulose, corn husk, pharmaceutical excipient". The abstract section is titled "ABSTRACT" and contains the text: "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". On the right side, there are buttons for "Sponsorship Disclosure", "Journal History", "Privacy Statement", "Archival System", and "Author Fees". Below these buttons, the "Editorial Member" section lists: "Editor in Chief: Prof. Dr. Abdul Rohman", "Associate Editors: Prof. Shuhaimi Mustafa", "Associate Editors: Dr. Ghada Suddek", "Associate Editors: Dr. Yangchao Luo", and "Associate Editors: Dr. Ashutosh Kumar S".

1 *Original Article*

2 **Pharmaceutical Grade Microcrystalline Cellulose** 3 **from Corn Husk (*Zea mays* L.): Fabrication and** 4 **Characterization**

5 **Intan Martha Cahyani***, Novi Tadlkirotulladlat, Endang Diyah Ikasari

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8 Received: date; Accepted: date; Published: date

9

10 **Abstract:** Corn is a plant that grows easily in tropical climates. Corn production in Indonesia reaches 25.18
11 tons, the use of which in society is still limited to corn kernels as food, while other parts of the corn plant are
12 waste. Corn husks are an abundant natural waste and contain 44.08% cellulose, so they can potentially be a
13 source of pharmaceutical excipients, namely microcrystalline cellulose (MCC). This research aims to isolate
14 and characterize MCC from pharmaceutical grade corn husks with commercial MCC as a comparator. The two
15 methods of making MCC are delignification using 2% NaOH at 80-90°C 4 h. Hydrolysis using variations in
16 HCl concentrations, namely 2 N, 4 N, and 6 N, at a temperature of 80°C 4 h. The research results obtained
17 cellulose content in α -cellulose and MCC of corn husks with 3 consecutive treatments of 74.02%, 84.48%,
18 86.55%, and 84.44%. The result of the analysis test of FTIR, SEM, XRD, and PSA instruments indicate that corn
19 husk MCC has characteristics of commercial MCC as a standard. The resulting corn husk MCC has
20 physicochemical characteristics according to standards that can be used as a pharmaceutical excipient.

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

23

24 1. INTRODUCTION

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

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

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

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

50 2. MATERIALS AND METHODS

51 2.1. Materials

52 Corn husks from plantation waste in the Semarang area, NaOH 2%, NaOCl 5%, HCl, and
53 aquadest. Materials for testing the physicochemical characteristics of MCC: Avicel PH 102, H₂SO₄ 1N,
54 H₂SO₄ 72%, ethanol, iodized zinc chloride solution (zinc chloride, potassium iodide and iodine) and
55 iodine 0.05 M.

56 2.2. Methods

57 2.2.1. Fabrication of MCC Corn Husk

58 a. Alkaline Delignification

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

65 b. Acid Hydrolysis

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

71 2.2.2 Physicochemical Characterization of MCC Corn Husk

72 a. Determination of Cellulose Concentration

73 The percentage of cellulose concentration was determined using the Che.sson-datta
74 method [15].

75 b. Moisture Content

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

78 c. pH

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

81

82

83 d. Melting Point

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

87 e. Flow Rate and Angle of Repose

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

92 f. Density, Carr's Index and Hausner Ratio

93 40 grams of corn husk MCC is placed in a 100 mL measuring cup. The surface of the powder
94 is carefully leveled without being compressed its volume (V_0) measurement is performed. A
95 measuring cup is installed on the support of the tapped density tester, 10, 500, and 1250 taps are
96 carried out and V_{10} , V_{500} , and V_{1250} are read on the nearest measuring cup unit. Volume measured
97 to the last tap (V_t) [19]. The density of MCC corn husks was determined by dividing weight by V_0
98 (bulk density) and V_t (tapped density). The true density of MCC is determined by determining the
99 volume using a pycnometer. Carr's index and hausner ratio indices were calculated from the
100 results of the bulk and tapped density that had been calculated.

101 2.2.3. Fourier Transformed Infrared (FTIR)

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

105 2.2.4. Scanning Electron Microscope (SEM)

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

110 2.2.5. X-Ray Diffraction (XRD)

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

114 2.2.6. Particle Size Analyzer (PSA)

115 Particle Size Analyzer (PSA) is used to determine the particle size distribution of corn husk
116 MCC using the Particle Size Analyzer tool (Malvern® Mastersizer 3000 (Malvern instrument UK)
117 ([21]).

118 3. RESULTS AND DISCUSSION

119 3.1. Physicochemical Characterization of MCC Corn Husk

120 The results of determining cellulose content using the Chasson-datta method were obtained
121 from the average cellulose content of corn husk powder of 42.90%, the results obtained were close to
122 the literature that corn husks have a cellulose content of 44.08% [22]. The yield of cellulose content in
123 corn husk α -cellulose increased by 74.02% due to alkalization treatment with NaOH which caused
124 the loss of lignin, mainly due to the unstable ester bond between cellulose and lignin complex, so that
125 lignin that loosely binds to alkali to form a water-soluble alkaline lignin complex. NaOH can break

126 the bond between cellulose with hemicellulose and lignin, causing changes in cellulose levels to
 127 increase [13]. The result of MCC corn husks was 84.48% HCl 2 N and MCC 4 N was 86.55%. The
 128 result of the concentration of MCC 6 N of cellulose content decreased by 84.44% (Table 1). The
 129 decrease in cellulose levels that occur is caused by the higher concentration of HCl causing an increase
 130 in heat (heat) causing the cellulose structure to open up so that cellulose molecules are dispersed
 131 freely in the solution, this freely dispersed cellulose structure results in the presence of dissolved
 132 cellulose carried away in the solution when the filtration process is carried out [23].

133 **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%; 0.36% & 0.24%
	Calcium	0.26	0.49	0.92	
	Natrium	0.13	0.09	0.13	
CrI (%)	34.1	34.7	34.3	34.5	34,5%
Particle size (µm)	362	362	395	332	20-200 µm

134
 135 The results of the MCC moisture content test from corn husks (Table 1.). The MCC samples
 136 treated with 2 N and 4 N HCl showed moisture content values close to that of commercial MCC (pH
 137 102), which has a reference moisture content of 5.37%. The moisture content of MCC treated with 6
 138 N HCl aligns with literature values, which are typically below 5%. If the moisture content is relatively
 139 high, it can increase the cohesion between similar particles, causing the powder to lose its ability to
 140 flow properly [24]. pH MCC corn husk and the comparator, Avicel PH 102 (Table1.), also exhibited
 141 the same pH value of 6, which is consistent with the literature pH range is between 5 and 7.5 [16].
 142 The results of the MCC melting point test from corn husks at each HCl concentration, as well as
 143 Avicel PH 102, showed that the powder did not melt but instead changed color to black within the

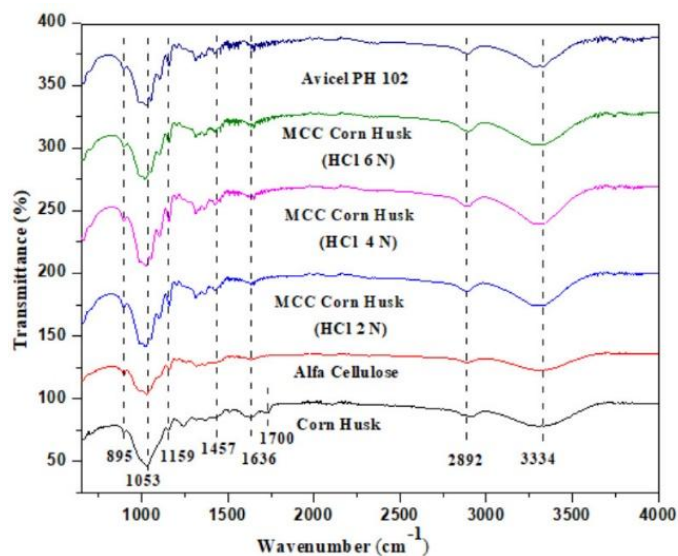
144 temperature range of 270°C to 400°C. These results do not align with the literature, which states that
145 the melting point should be around 260°C to 270°C [16].

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

156 The results of bulk and tapping density of MCC corn husks showed that samples treated
157 with 2 N and 4 N HCl, as well as Avicel PH 102, produced values close to the limit requirements
158 (Table 1). MCC corn husks treated with 6 N HCl produced higher values compared to Avicel PH 102
159 and the literature. The actual density of MCC corn husks treated with 6 N HCl was within the limit
160 requirement range of 1.512–1.668 g/cm³ [16], while MCC treated with 2 N, 4 N HCl, and Avicel PH
161 102 showed lower actual density values than those reported. The hausner ratio value for MCC corn
162 husks treated with 2 N, 4 N, and 6 N HCl concentrations was comparable to Avicel PH 102. The
163 higher concentration of HCl used in the hydrolysis process had an effect on reducing the carr's index
164 and hausner ratio values. MCC corn husks resulting from hydrolysis with 6 N HCl showed better
165 flow properties and compressibility compared to Avicel PH 102.

166 3.2. Fourier Transformed Infrared (FTIR)

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



173

174

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

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

183 3.3. Scanning Electrone Microscopy (SEM)

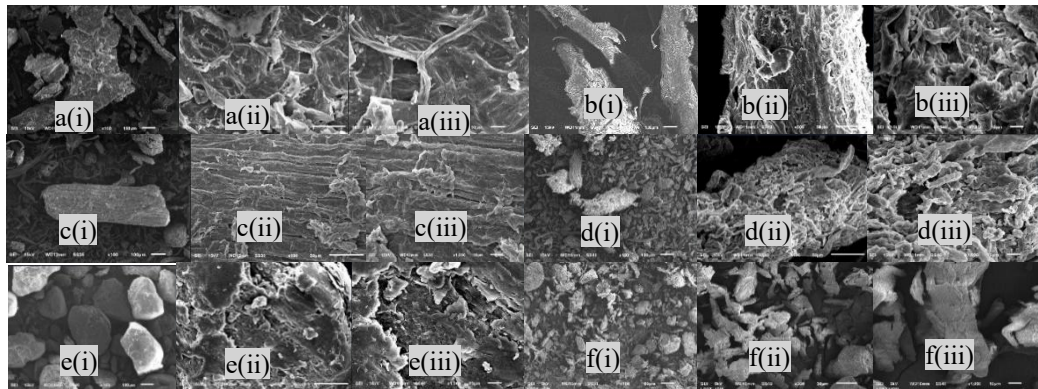
184 The morphological observation of raw corn husk powder revealed a denser surface structure,
 185 which is attributed to the presence of lignin still embedded in the cell wall, serving to protect the
 186 cellulose. In contrast, the morphology of α -cellulose showed the initial stages of solid peeling, leading
 187 to the formation of irregular fibrous structures. MCC derived from corn husks treated with 2 N and
 188 4 N HCl exhibited elongated, stem-like shapes with uneven surfaces, slightly hollow structures, and
 189 distinguishable blunt-angled edges (Figure 2).

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Figure 1. SEM Image of (a) Powder Corn Husk; (b) α -Cellulose; (c) MCC Corn Husk (HCl 2 N); (d) MCC Corn
 195 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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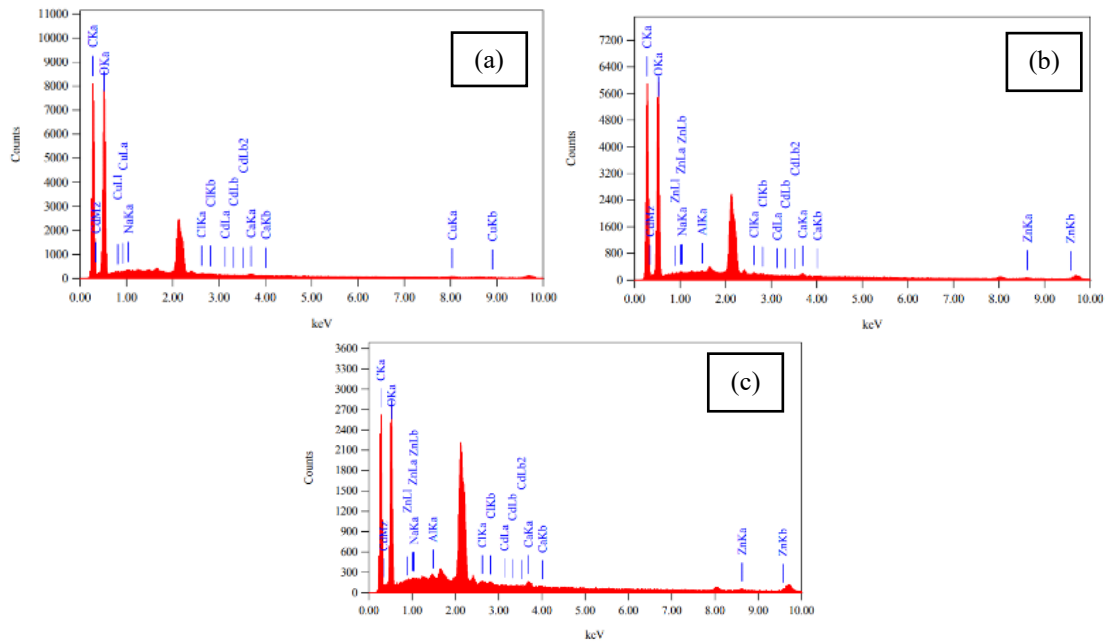
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The comparator, Avicel PH 102, displayed irregularly shaped particles with varying lengths,
 uneven and slightly hollow surfaces, and both pointed and blunt edges [29]. MCC obtained using 6
 N HCl showed a more compact, spherical, and granular morphology 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).



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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)

210

3.4. X-Ray Diffraction (XRD)

211

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 $2\theta=24.2^\circ$ (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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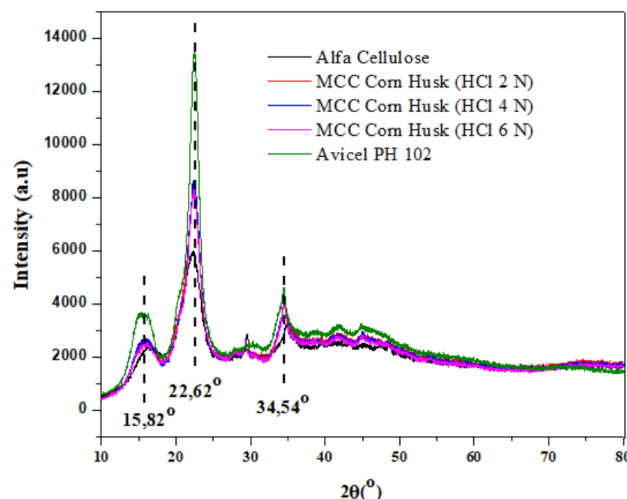
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Figure 3. X-ray diffraction patterns of Corn Husk, alfa cellulose, MCC Corn Husk and Avicel PH 102

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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

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

228 **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

229

230 4. CONCLUSION

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

242 **Funding:** This research received no external funding

243 **Acknowledgments:** The author would like to acknowledge the support from Stifar Yayasan
 244 Pharmasi Semarang

245 **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

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].

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Commented [V2]: Could you added the figure of part of corn like corn husks

Commented [V3]: How much percent the content? And please explain the other contain

Commented [V4]: Please explain what is the full form of the abbreviation mcc

Commented [V5]: Is the hydrolysis using corn or not?

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.

Commented [V6]: Why do you use corn in this research? Please explain gap analysis from the previous research

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, H₂SO₄ 1N, H₂SO₄ 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 percentage of cellulose concentration was determined 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 (V_0) 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 V_{10} , V_{500} , and V_{1250} are read on the nearest measuring cup unit. Volume measured to the last tap (V_t) [19]. The density of MCC corn husks was determined by dividing weight by V_0 (bulk density) and V_t (tapped density). The true density of MCC is determined by determining the volume using a pycnometer. 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 Electron 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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Commented [V13]: Hcl or mcc?

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.

3.2. Fourier Transformed Infrared (FTIR)

FTIR Spectra of MCC corn husks (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].

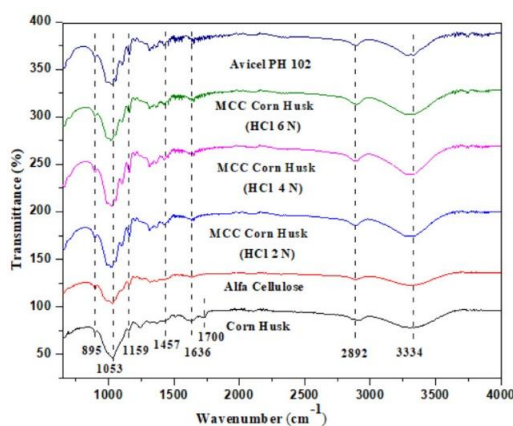


Figure 1. FTIR Spectra of Corn Husk, alfa cellulose, MCC Corn Husk and Avicel PH 102

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

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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 Electron 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.).

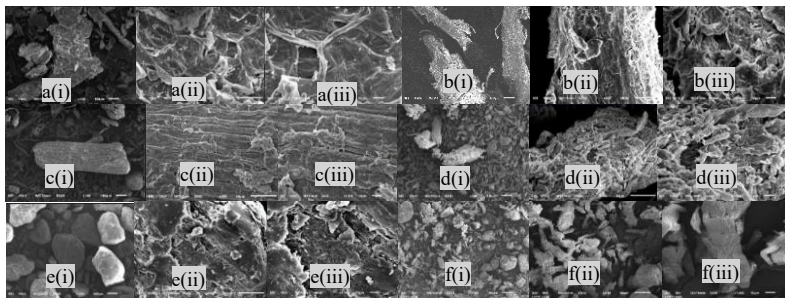


Figure 1. SEM Image of (a) Powder Corn Husk; (b) α -Cellulose; (c) MCC Corn Husk (HCl 2 N); (d) MCC Corn Husk (HCl 4 N); (e) MCC Corn Husk (HCl 6 N); (f) Avicel pH 102; (i) 100x; (ii) 500x; (iii) 1000x.

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).

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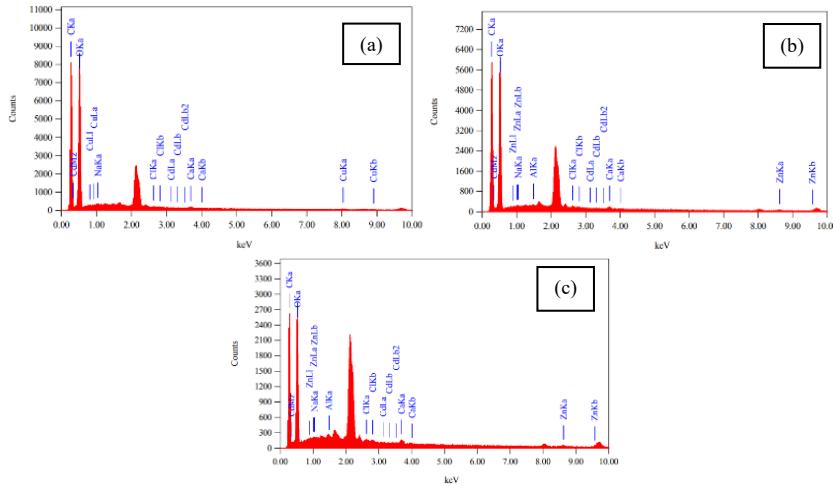


Figure 2. Test SEM-EDX Elements Klor, Calcium, Natrium (a) MCC Corn Husk (HCl 2 N); (b) MCC Corn Husk (HCl 4 N); (c) MCC Corn Husk (HCl 6 N)

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 $2\theta=24.2^\circ$ (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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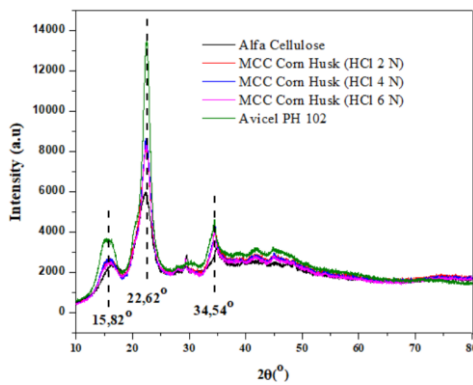


Figure 3. X-ray diffraction patterns of Corn Husk, alfa cellulose, MCC Corn Husk and Avicel PH 102

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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Funding: This research received no external funding

Acknowledgments: The author would like to acknowledge the support from Stifar Yayasan Pharmasi Semarang

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

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 delignified 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, H₂SO₄ 1N, H₂SO₄ 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 percentage of cellulose concentration was determined 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.

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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 (V_0) 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 V_{10} , V_{500} , and V_{1250} are read on the nearest measuring cup unit. Volume measured to the last tap (V_t) [19]. The density of MCC corn husks was determined by dividing weight by V_0 (bulk density) and V_t (tapped density). The true density of MCC is determined by determining the volume using a pycnometer. 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 Electron 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%; 0.36% & 0.24%
	Calcium	0.26	0.49	0.92	
	Natrium	0.13	0.09	0.13	
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 husks (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].

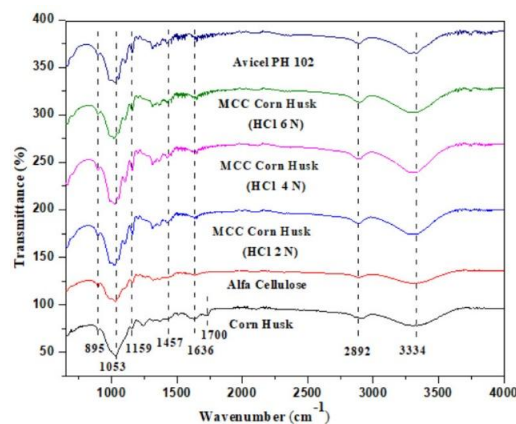


Figure 1. FTIR Spectra of Corn Husk, alfa cellulose, MCC Corn Husk and Avicel PH 102

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 Electron 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.).

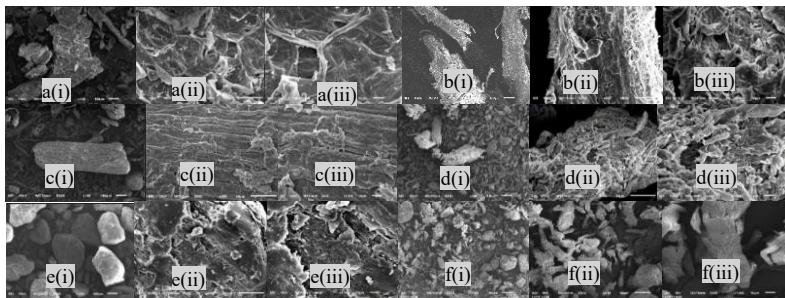


Figure 1. SEM Image of (a) Powder Corn Husk; (b) α -Cellulose; (c) MCC Corn Husk (HCl 2 N); (d) MCC Corn Husk (HCl 4 N); (e) MCC Corn Husk (HCl 6 N); (f) Avicel pH 102; (i) 100x; (ii) 500x; (iii) 1000x.

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).

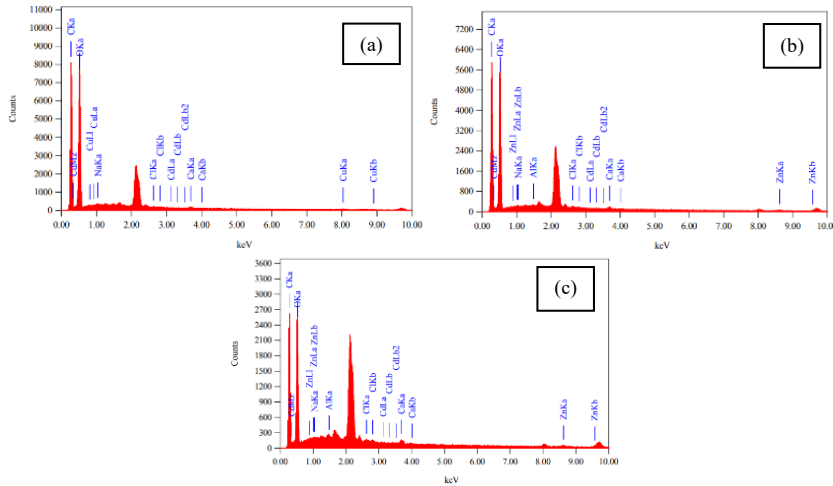


Figure 2. Test SEM-EDX Elements Klor, Calcium, Natrium (a) MCC Corn Husk (HCl 2 N); (b) MCC Corn Husk (HCl 4 N); (c) MCC Corn Husk (HCl 6 N)

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 $2\theta=24.2^\circ$ (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]

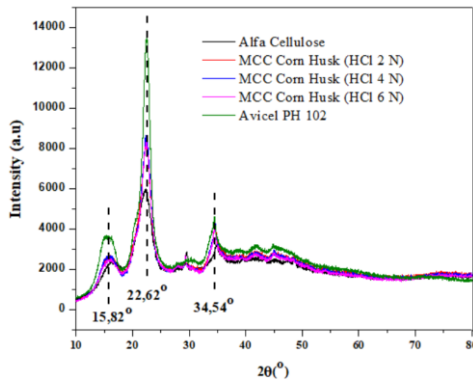


Figure 3. X-ray diffraction patterns of Corn Husk, alfa cellulose, MCC Corn Husk and Avicel PH 102

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.

Funding: This research received no external funding

Acknowledgments: The author would like to acknowledge the support from Stifar Yayasan Pharmasi Semarang

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

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1 *Original Article*

2 **Pharmaceutical Grade Microcrystalline Cellulose** 3 **from Corn Husk (*Zea mays* L.): Fabrication and** 4 **Characterization**

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8 Received: date; Accepted: date; Published: date

9

10 **Abstract:** Corn is a plant that grows easily in tropical climates. Corn production in Indonesia reaches 25.18
11 tons, the use of which in society is still limited to corn kernels as food, while other parts of the corn plant are
12 waste. Corn husks are an abundant natural waste and contain 44.08% cellulose, so they can potentially be a
13 source of pharmaceutical excipients, namely microcrystalline cellulose (MCC). This research aims to isolate
14 and characterize MCC from pharmaceutical grade corn husks with commercial MCC as a comparator. The two
15 methods of making MCC are delignification using 2% NaOH at 80-90°C 4 h. Hydrolysis using variations in
16 HCl concentrations, namely 2 N, 4 N, and 6 N, at a temperature of 80°C 4 h. The research results obtained
17 cellulose content in α -cellulose and MCC of corn husks with 3 consecutive treatments of 74.02%, 84.48%,
18 86.55%, and 84.44%. The result of the analysis test of FTIR, SEM, XRD, and PSA instruments indicate that corn
19 husk MCC has characteristics of commercial MCC as a standard. The resulting corn husk MCC has
20 physicochemical characteristics according to standards that can be used as a pharmaceutical excipient.

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

23

24 1. INTRODUCTION

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

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

38 MCC can be made by delignification and then hydrolysis. Delignification is carried out to
39 change the structure of lignocellulose biomass, which aims to degrade lignin polymers bound to
40 cellulose, then lignin will dissolve in water, and the result is α -cellulose. Delignification of α -cellulose
41 powder was subjected to controlled hydrolysis using an acidic solution. Acid hydrolysis can damage
42 the amorphous region of the cellulose microfibrils, where the amorphous form will undergo
43 disconnection and then leave a crystalline [7,8]. **Several studies on the use of HCl in hydrolysis in the**

44 manufacture of MCC from other natural materials have been able to increase the yield and
45 crystallinity index [9–12].

46 The pharmaceutical industry in Indonesia is still dependent on imported raw materials (95%)
47 [13]. The raw materials here are not only active ingredients but also excipients that play an important
48 role in determining the quality of the dosage form. The abundant corn crop yield (38.38%) means that
49 the amount of corn husk waste produced also increases [14]. It is necessary to develop cellulose
50 technology for high in corn husks (44.08%) [3] into MCC as an alternative pharmaceutical excipient
51 native to Indonesia that not only solves the problem of meeting the needs of raw materials for the
52 pharmaceutical industry but also solves the problem of plantation waste. Several studies on the
53 isolation of MCC from corn waste that have been carried out include corn cobs with variations of
54 NaOH in the delignification process and hydrolysis with 10% H₂SO₄ obtained a yield of 30% and CrI
55 91.26% [15]. Hydrolysis of pulut corn husks with 2.5N HCl for 10 minutes produced MCC with CrI
56 79% [16]. However, this study was limited to CrI and morphology analysis, so it is necessary to
57 conduct research on the fabrication and physicochemical and mechanical characterization of MCC
58 corn husks compared to Avicel PH 102 as a commercial standard so that it can guarantee its quality
59 as a pharmaceutical excipient.

60

61 2. MATERIALS AND METHODS

62 2.1. Materials

63 Corn husks from plantation waste in the Semarang area (Indonesia) which is dried and
64 ground with 40 mesh. Technical grade material: sodium hydroxide (NaOH) (Hangzhou Lizu Co.,
65 Ltd), sodium hypochlorite (NaOCl) (Asahimas), hydrochloric acid (HCl) (Tjiwi Kimia). Pro-analysis
66 material (Merck): sulfuric acid (H₂SO₄), ethanol, iodized zinc chloride solution (zinc chloride,
67 potassium iodide and iodine) and iodine. Pharmaceutical grade material: Avicel PH 102 (American
68 International Chemical/AIC, Inc-Framingham USA) as commercial standard.

69 2.2. Methods

70 2.2.1. Fabrication of MCC Corn Husk

71 a. Alkaline Delignification

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

76 b. Acid Hydrolysis

77 Hydrolysis of α -cellulose with variations in HCl concentration of 2 N, 4 N, and 6 N for 80°C
78 4 hours and then filtered and washed until a neutral pH of 6-7. The next stage is bleached 2 times
79 with a 5% NaOCl solution of 70°C for 1 hour and soaked in the same solution for 24 hours at room
80 temperature. The residue is filtered and washed until a neutral pH of 6-7. MCC is dried and smoothed
81 then sifted mesh no. 60 [18].

82 2.2.2 Physicochemical Characterization of MCC Corn Husk

83 a. Determination of Cellulose Concentration

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

85

86 b. Moisture Content

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

89 c. pH

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

92 d. Melting Point

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

96 e. Flow Rate and Angle of Repose

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

101 f. Density, Carr's Index and Hausner Ratio

102 40 grams of corn husk MCC is placed in a 100 mL measuring cup. The surface of the powder
103 is carefully leveled without being compressed its volume (V_0) measurement is performed. A
104 measuring cup is installed on the support of the tapped density tester, 10, 500, and 1250 taps are
105 carried out and V_{10} , V_{500} , and V_{1250} are read on the nearest measuring cup unit. Volume measured
106 to the last tap (V_t) [23]. The density of MCC corn husks was determined by dividing weight by V_0
107 (bulk density) and V_t (tapped density). The true density of MCC is determined by determining the
108 volume using a picnometre. Carr's index and hausner ratio indices were calculated from the
109 results of the bulk and tapped density that had been calculated.

110 2.2.3. Fourier Transformed Infrared (FTIR)

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

114 2.2.4. Scanning Electrone Microscope (SEM)

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

119 2.2.5. X-Ray Diffraction (XRD)

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

123 2.2.6. Particle Size Analyzer (PSA)

124 Particle Size Analyzer (PSA) is used to determine the particle size distribution of corn husk
125 MCC using the Particle Size Analyzer tool (Malvern® Mastersizer 3000 (Malvern instrument UK)
126 [25].

127 **3. RESULTS AND DISCUSSION**128 **3.1. Physicochemical Characterization of MCC Corn Husk**

129 The results of determining cellulose content using the Chasson-datta method were obtained
 130 from the average cellulose content of corn husk powder of 42.90%, the results obtained were close to
 131 the literature that corn husks have a cellulose content of 44.08% [3]. The yield of cellulose content in
 132 corn husk α -cellulose increased by 74.02% due to alkalization treatment with NaOH which caused
 133 the loss of lignin, mainly due to the unstable ester bond between cellulose and lignin complex, so that
 134 lignin that loosely binds to alkali to form a water-soluble alkaline lignin complex. NaOH can break
 135 the bond between cellulose with hemicellulose and lignin, causing changes in cellulose levels to
 136 increase [17]. The result concentration of cellulose MCC corn husks with HCl 2 N 84.48% and 4 N
 137 86.55% and there was a decreased in 6 N 84.44% (Table 1). The decrease in cellulose levels that occur
 138 is caused by the higher concentration of HCl causing an increase in heat (heat) causing the cellulose
 139 structure to open up so that cellulose molecules are dispersed freely in the solution, this freely
 140 dispersed cellulose structure results in the presence of dissolved cellulose carried away in the solution
 141 when the filtration process is carried out [26].

142 **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 (μ m)	362	362	395	332	20-200 μ m	

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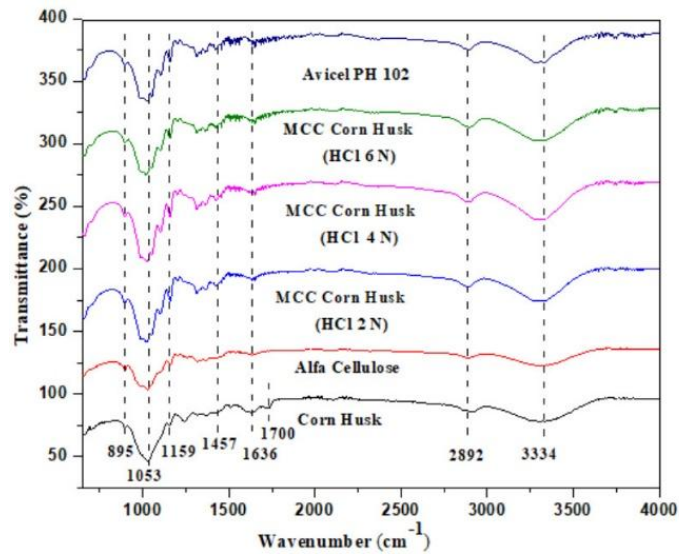
144 The results of the MCC moisture content test from corn husks (Table 1.). The MCC samples
145 treated with 2 N and 4 N HCl showed moisture content values close to that of commercial MCC (pH
146 102), which has a reference moisture content of 5.37%. The moisture content of MCC treated with 6
147 N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

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

165 The results of bulk and tapping density of MCC corn husks showed that samples treated with 2 N
166 and 4 N HCl, as well as Avicel PH 102, produced values close to the limit requirements (Table 1).
167 MCC corn husks treated with 6 N HCl produced higher values compared to Avicel PH 102 and the
168 literature. The actual density of MCC corn husks treated with 6 N HCl was within the limit
169 requirement range of 1.512–1.668 g/cm³ [20], while MCC treated with 2 N, 4 N HCl, and Avicel PH
170 102 showed lower actual density values than those reported. The hausner ratio value for MCC corn
171 husks treated with 2 N, 4 N, and 6 N HCl concentrations was comparable to Avicel PH 102. The
172 higher concentration of HCl used in the hydrolysis process had an effect on reducing the carr's index
173 and hausner ratio. MCC corn husks resulting from hydrolysis with 6 N HCl showed better flow
174 properties and compressibility compared to Avicel PH 102. The amorphous form of cellulose is very
175 susceptible to HCl so that the higher the concentration of HCl, the more the amorphous form is lost
176 so that denser cellulose crystals will be formed with fewer cavities on the particle surface as seen in
177 the morphology from the SEM analysis results (Figure 2.). The number of cavities on the particle
178 surface can create space between particles which affects the increase in porosity and bulk volume so
179 that its flowability is low [30].

180 3.2. Fourier Transformed Infrared (FTIR)

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



187

188

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

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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].

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3.3. Scanning Electron Microscopy (SEM)

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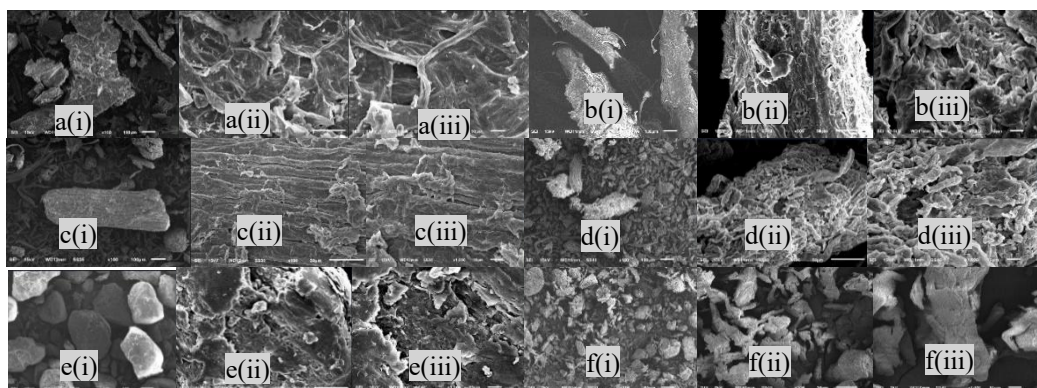
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The morphological observation of raw corn husk powder revealed a denser surface structure, which is attributed to the presence of lignin still embedded in the cell wall, serving to protect the cellulose. In contrast, the morphology of α -cellulose 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).

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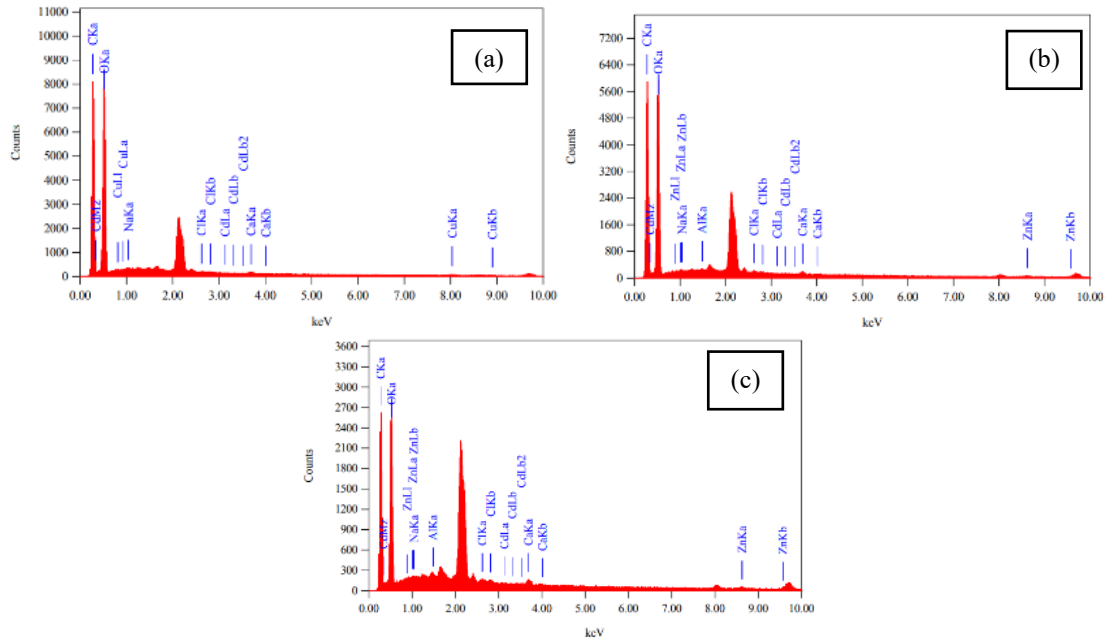
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Figure 1. SEM Image of (a) Powder Corn Husk; (b) α -Cellulose; (c) MCC Corn Husk (HCl 2 N); (d) MCC Corn Husk (HCl 4 N); (e) MCC Corn Husk (HCl 6 N); (f) Avicel pH 102; with magnification (i) 100x; (ii) 500x; (iii) 1000x.

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

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



217

218 **Figure 2.** Test SEM-EDX Elements Klor, Calcium, Natrium (a) MCC Corn Husk (HCl 2 N); (b) MCC Corn Husk
 219 (HCl 4 N); (c) MCC Corn Husk (HCl 6 N)
 220

221 **3.4. X-Ray Diffraction (XRD)**

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

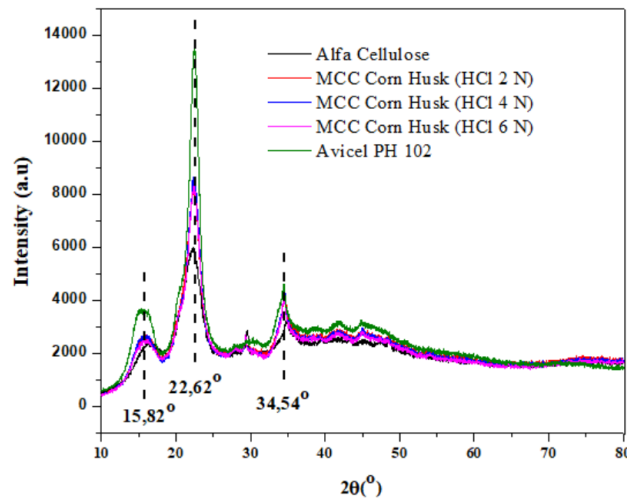


Figure 3. X-ray diffraction patterns of Corn Husk, alfa cellulose, MCC Corn Husk and Avicel PH 102

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.

259 **Funding:** This research received no external funding

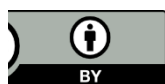
260 **Acknowledgments:** The author would like to acknowledge the support from Stifar Yayasan
261 Pharmasi Semarang

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

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1 Original Article

2 **Pharmaceutical Grade Microcrystalline Cellulose from Corn** 3 **Husk (*Zea mays* L.): Fabrication and Characterization**

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7 Received: 13 May 2025; Revised: 23 May 2025; Accepted: 02 June 2025; Published: June 2025

8 **Abstract:** Corn is a plant that grows easily in tropical climates. Corn production in Indonesia reaches 25.18
9 tons, the use of which in society is still limited to corn kernels as food, while other parts of the corn plant are
10 waste. Corn husks are an abundant natural waste and contain 44.08% cellulose, so they can potentially be a
11 source of pharmaceutical excipients, namely microcrystalline cellulose (MCC). This research aims to isolate
12 and characterize MCC from pharmaceutical grade corn husks with commercial MCC as a comparator. The two
13 methods of making MCC are delignification using 2% NaOH at 80-90°C 4 h. Hydrolysis using variations in
14 HCl concentrations, namely 2 N, 4 N, and 6 N, at a temperature of 80°C 4 h. The research results obtained
15 cellulose content in α -cellulose and MCC of corn husks with 3 consecutive treatments of 74.02%, 84.48%,
16 86.55%, and 84.44%. The result of the analysis test of FTIR, SEM, XRD, and PSA instruments indicate that corn
17 husk MCC has characteristics of commercial MCC as a standard. The resulting corn husk MCC has
18 physicochemical characteristics according to standards that can be used as a pharmaceutical excipient.

19 **Keywords:** characterization, fabrication, microcrystalline cellulose, corn husk, pharmaceutical
20 excipient

22 1. INTRODUCTION

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

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

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

44 The pharmaceutical industry in Indonesia is still dependent on imported raw materials (95%) [13].
45 The raw materials here are not only active ingredients but also excipients that play an important role

46 in determining the quality of the dosage form. The abundant corn crop yield (38.38%) means that the
47 amount of corn husk waste produced also increases [14]. It is necessary to develop cellulose
48 technology for high in corn husks (44.08%) [3] into MCC as an alternative pharmaceutical excipient
49 native to Indonesia that not only solves the problem of meeting the needs of raw materials for the
50 pharmaceutical industry but also solves the problem of plantation waste. Several studies on the
51 isolation of MCC from corn waste that have been carried out include corn cobs with variations of
52 NaOH in the delignification process and hydrolysis with 10% H₂SO₄ obtained a yield of 30% and CrI
53 91.26% [15]. Hydrolysis of pulut corn husks with 2.5N HCl for 10 minutes produced MCC with CrI
54 79% [16]. However, this study was limited to CrI and morphology analysis, so it is necessary to
55 conduct research on the fabrication and physicochemical and mechanical characterization of MCC
56 corn husks compared to Avicel PH 102 as a commercial standard so that it can guarantee its quality
57 as a pharmaceutical excipient.

58 2. MATERIALS AND METHODS

59 2.1. Materials

60 Corn husks from plantation waste in the Semarang area (Indonesia) which is dried and
61 ground with 40 mesh. Technical grade material: sodium hydroxide (NaOH) (Hangzhou Lizu Co.,
62 Ltd), sodium hypochlorite (NaOCl) (Asahimas), hydrochloric acid (HCl) (Tjiwi Kimia). Pro-analysis
63 material (Merck): sulfuric acid (H₂SO₄), ethanol, iodized zinc chloride solution (zinc chloride,
64 potassium iodide and iodine) and iodine. Pharmaceutical grade material: Avicel PH 102 (American
65 International Chemical/AIC, Inc-Framingham USA) as commercial standard.

66 2.2. Methods

67 2.2.1. Fabrication of MCC Corn Husk

68 a. Alkaline Delignification

69 Delignification of corn husk powder with 2% NaOH at 80-90°C for 4 h, the residue is filtered
70 and washed down to a neutral pH of 6-7. The next stage is bleaching with a solution of NaOCl 5% at
71 70°C for 1 hour and NaOCl 5% for 24 h at room temperature. The residue is filtered and washed to a
72 neutral pH of 6-7. Cellulose is produced, dried, and mashed [17].

73 b. Acid Hydrolysis

74 Hydrolysis of α -cellulose with variations in HCl concentration of 2 N, 4 N, and 6 N for 80°C
75 4 hours and then filtered and washed until a neutral pH of 6-7. The next stage is bleached 2 times
76 with a 5% NaOCl solution of 70°C for 1 hour and soaked in the same solution for 24 hours at room
77 temperature. The residue is filtered and washed until a neutral pH of 6-7. MCC is dried and smoothed
78 then sifted mesh no. 60 [18].

79 2.2.2 Physicochemical Characterization of MCC Corn Husk

80 a. Determination of Cellulose Concentration

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

82 b. Moisture Content

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

85 c. pH

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

88 d. Melting Point

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

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93 e. Flow Rate and Angle of Repose

94 The flow rate of MCC corn husks using a flowability tester (Erweka GT) with a funnel diameter of
95 15 mm. The cover at the bottom of the funnel is opened and the flow speed is calculated at the time
96 the granule starts flowing until the granule stops flowing using a stopwatch and then the time
97 obtained and the height and diameter of the cone are measured [22].

98 f. Density, Carr's Index and Hausner Ratio

99 40 grams of corn husk MCC is placed in a 100 mL measuring cup. The surface of the powder is
100 carefully leveled without being compressed its volume (V_0) measurement is performed. A measuring
101 cup is installed on the support of the tapped density tester, 10, 500, and 1250 taps are carried out and
102 V_{10} , V_{500} , and V_{1250} are read on the nearest measuring cup unit. Volume measured to the last tap
103 (V_t) [23]. The density of MCC corn husks was determined by dividing weight by V_0 (bulk density)
104 and V_t (tapped density). The true density of MCC is determined by determining the volume using a
105 picnometer. Carr's index and hausner ratio indices were calculated from the results of the bulk
106 and tapped density that had been calculated.

107 2.2.3. Fourier Transformed Infrared (FTIR)

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

111 2.2.4. Scanning Electron Microscope (SEM)

112 Scanning Electron Microscope-Energy Dispersive X-Ray (SEM-EDX) MCC testing of corn husks
113 was used to determine the morphological shape as well as to analyze the elements contained in the
114 sample using the Scanning Electron Microscope-Energy Dispersive X-Ray microscope (JEOLJSM-
115 6510LA).

116 2.2.5. X-Ray Diffraction (XRD)

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

120 2.2.6. Particle Size Analyzer (PSA)

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

123 **3. RESULTS AND DISCUSSION**

124 *3.1. Physicochemical Characterization of MCC Corn Husk*

125 The results of determining cellulose content using the Chasson-datta method were obtained
126 from the average cellulose content of corn husk powder of 42.90%, the results obtained were close to
127 the literature that corn husks have a cellulose content of 44.08% [3]. The yield of cellulose content in
128 corn husk α -cellulose increased by 74.02% due to alkalization treatment with NaOH which caused
129 the loss of lignin, mainly due to the unstable ester bond between cellulose and lignin complex, so that
130 lignin that loosely binds to alkali to form a water-soluble alkaline lignin complex. NaOH can break
131 the bond between cellulose with hemicellulose and lignin, causing changes in cellulose levels to
132 increase [17]. The result concentration of cellulose MCC corn husks with HCl 2 N 84.48% and 4 N
133 86.55% and there was a decreased in 6 N 84.44% (Table 1). The decrease in cellulose levels that occur
134 is caused by the higher concentration of HCl causing an increase in heat (heat) causing the cellulose
135 structure to open up so that cellulose molecules are dispersed freely in the solution, this freely
136 dispersed cellulose structure results in the presence of dissolved cellulose carried away in the solution
137 when the filtration process is carried out [26].

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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	

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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 [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].

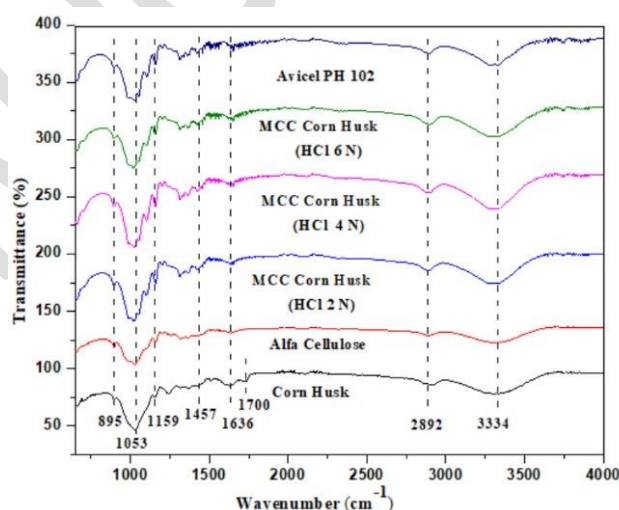
The flow rate of MCC corn husk is better than Avicel PH 102 as a commercial standard (Table 1), because a good flow rate is indicated by a value greater than 10 g/s. This shows that increasing the concentration of HCl can affect the density and particle size of MCC. Powders with smaller particle sizes 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

157 the flow of the powder [28]. The results of the angle of repose for MCC corn husk show that, on
158 average, the faster the flow of MCC, the smaller the angle of repose formed. This is believed to be
159 due to the larger particle size and low cohesiveness of the powder, which contribute to its good flow
160 properties. Smaller particle size, higher cohesiveness, and greater frictional forces, thus inhibiting the
161 flow of the powder [29].

162 The results of bulk and tapping density of MCC corn husks showed that samples treated with
163 2 N and 4 N HCl, as well as Avicel PH 102, produced values close to the limit requirements (Table 1).
164 MCC corn husks treated with 6 N HCl produced higher values compared to Avicel PH 102 and the
165 literature. The actual density of MCC corn husks treated with 6 N HCl was within the limit
166 requirement range of 1.512–1.668 g/cm³ [20], while MCC treated with 2 N, 4 N HCl, and Avicel PH
167 102 showed lower actual density values than those reported. The hausner ratio value for MCC corn
168 husks treated with 2 N, 4 N, and 6 N HCl concentrations was comparable to Avicel PH 102. The
169 higher concentration of HCl used in the hydrolysis process had an effect on reducing the carr's index
170 and hausner ratio. MCC corn husks resulting from hydrolysis with 6 N HCl showed better flow
171 properties and compressibility compared to Avicel PH 102. The amorphous form of cellulose is very
172 susceptible to HCl so that the higher the concentration of HCl, the more the amorphous form is lost
173 so that denser cellulose crystals will be formed with fewer cavities on the particle surface as seen in
174 the morphology from the SEM analysis results (Figure 2.). The number of cavities on the particle
175 surface can create space between particles which affects the increase in porosity and bulk volume so
176 that its flowability is low [30].

177 3.2. Fourier Transformed Infrared (FTIR)

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



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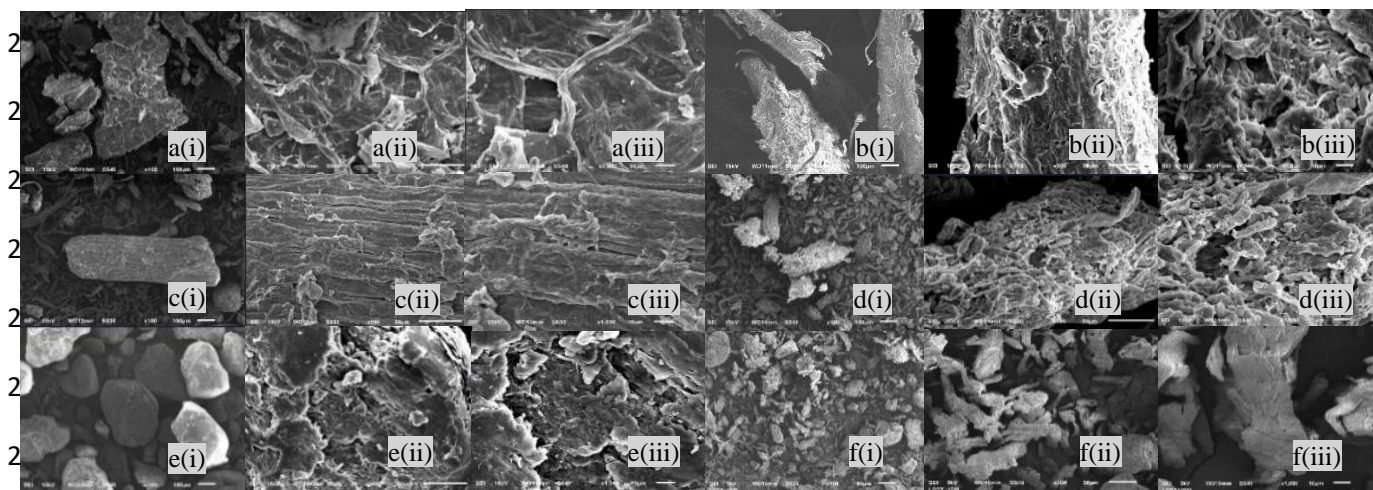
185 **Figure 1.** FTIR Spectra of Corn Husk, alfa cellulose, MCC Corn Husk and Avicel PH 102

186 FTIR analysis also revealed the presence of a lignin absorption band at around 1700 cm⁻¹ in raw
187 corn husk powder that had not been treated with NaOH, indicating the presence of lignin prior to
188 the delignification process. In contrast, the FTIR spectra of MCC derived from corn husks treated
189 with HCl concentrations of 2 N, 4 N, and 6 N showed similar spectral patterns to that of Avicel PH
190 102. These spectra confirmed the presence of cellulose, while the absorption bands associated with

191 hemicellulose and lignin were no longer observed in the MCC samples and Avicel PH 102. This
 192 indicates that the non-cellulosic components were effectively removed during the delignification and
 193 purification processes, leaving behind primarily α -cellulose [17].

194 3.3. Scanning Electrone Microscopy (SEM)

195 The morphological observation of raw corn husk powder revealed a denser surface structure,
 196 which is attributed to the presence of lignin still embedded in the cell wall, serving to protect the
 197 cellulose. In contrast, the morphology of α -cellulose showed the initial stages of solid peeling, leading
 198 to the formation of irregular fibrous structures. MCC derived from corn husks treated with 2 N and
 199 4 N HCl exhibited elongated, stem-like shapes with uneven surfaces, slightly hollow structures, and
 200 distinguishable blunt-angled edges (Figure 2).



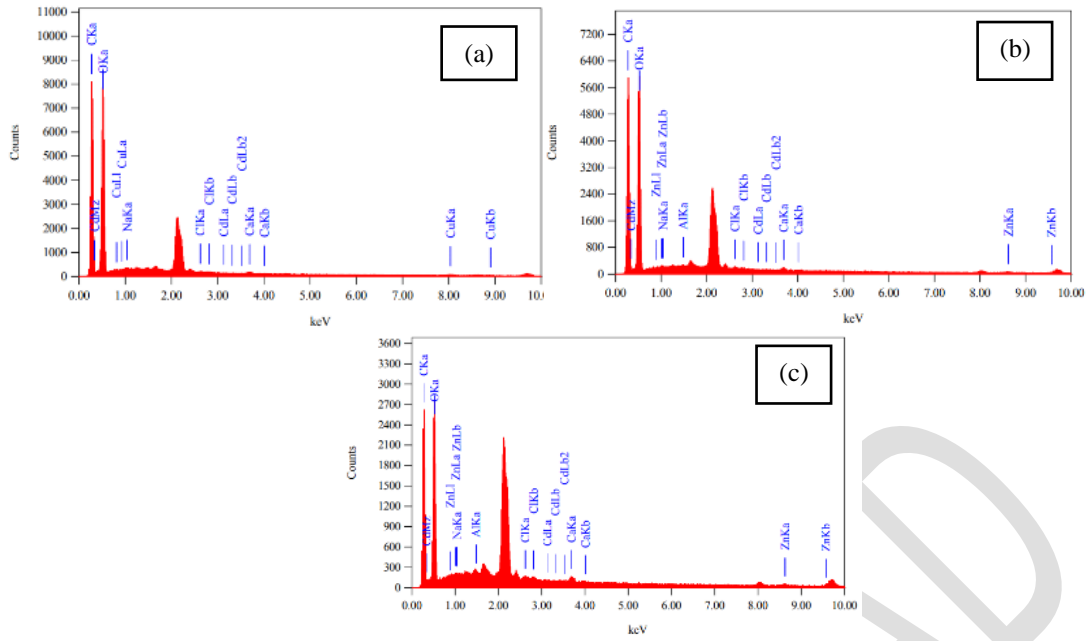
208 **Figure 1.** SEM Image of (a) Powder Corn Husk; (b) α -Cellulose; (c) MCC Corn Husk (HCl 2 N); (d) MCC Corn
 209 Husk (HCl 4 N); (e) MCC Corn Husk (HCl 6 N); (f) Avicel pH 102; with magnification (i) 100x; (ii) 500x; (iii)
 210 1000x.

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

218 3.4. X-Ray Diffraction (XRD)

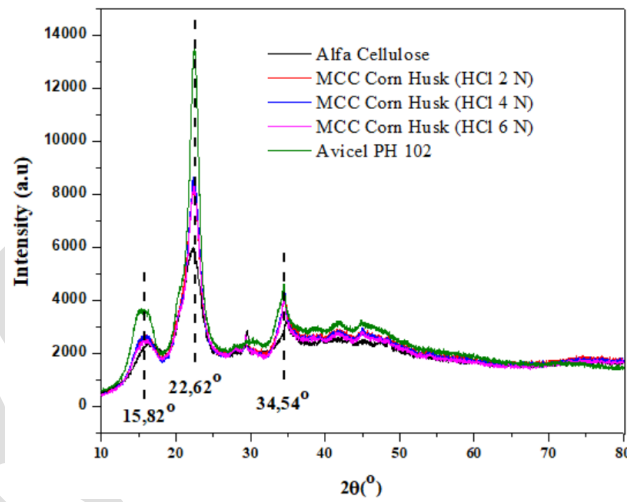
219 X-Ray Diffraction (XRD) analysis of MCC corn husks showed that the crystallinity of the samples
 220 increased with higher HCl concentrations (Table 1). This increase in crystallinity is due to the loss of
 221 the lignin layer from the corn husk sample as evidenced by the absence of a peak at $2\theta=24.2^\circ$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

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

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

246 3.5. Particle Size Analyzer (PSA)

247 **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

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

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

254 4. CONCLUSION

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

270 **Funding:** This research received no external funding

271 **Acknowledgments:** The author would like to acknowledge the support from Stifar Yayasan
272 Pharmasi Semarang

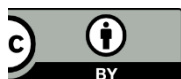
273 **Conflicts of interest:** The authors declare no conflict of interest.

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Original Article

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

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Received: 13 May 2025; Revised: 23 May 2025; Accepted: 02 June 2025; Published: 30 June 2025

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% H₂SO₄ 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 (H₂SO₄), 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 (V_0) 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 V_{10} , V_{500} , and V_{1250} are read on the nearest measuring cup unit. Volume measured to the last tap (V_t) [23]. The density of MCC corn husks was determined by dividing weight by V_0 (bulk density) and V_t (tapped density). The true density of MCC is determined by determining the volume using a picnometer. 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 Electron 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].

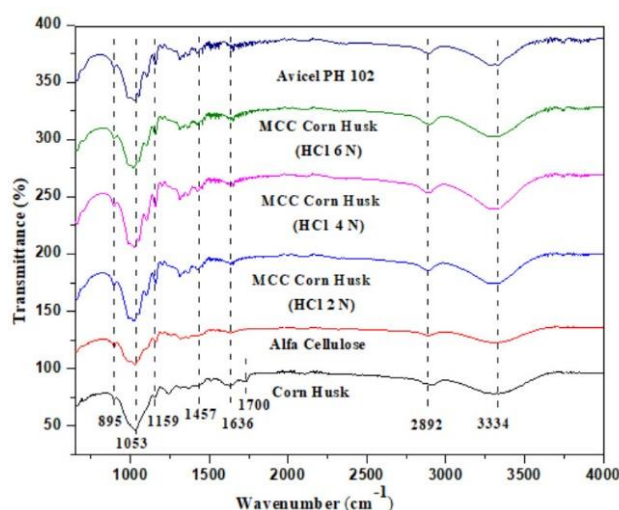


Figure 1. FTIR Spectra of Corn Husk, alfa cellulose, MCC Corn Husk and Avicel PH 102

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 Electron 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).

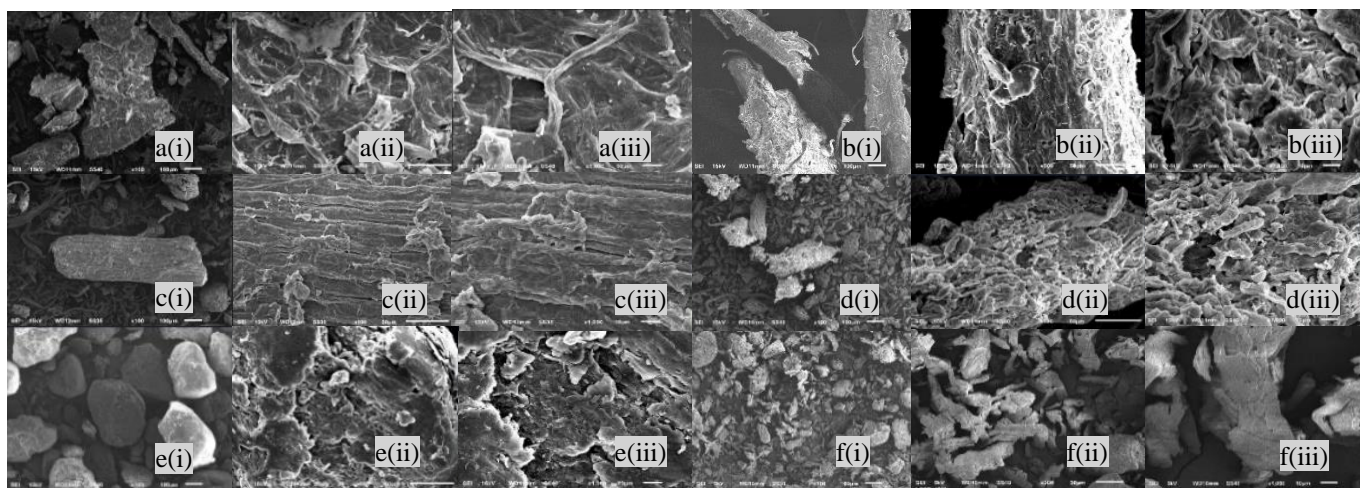


Figure 1. SEM Image of (a) Powder Corn Husk; (b) α -Cellulose; (c) MCC Corn Husk (HCl 2 N); (d) MCC Corn Husk (HCl 4 N); (e) MCC Corn Husk (HCl 6 N); (f) Avicel pH 102; with magnification (i) 100x; (ii) 500x; (iii) 1000x.

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 $2\theta=24.2^\circ$ 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 $2\theta= \pm 20^\circ$ [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].

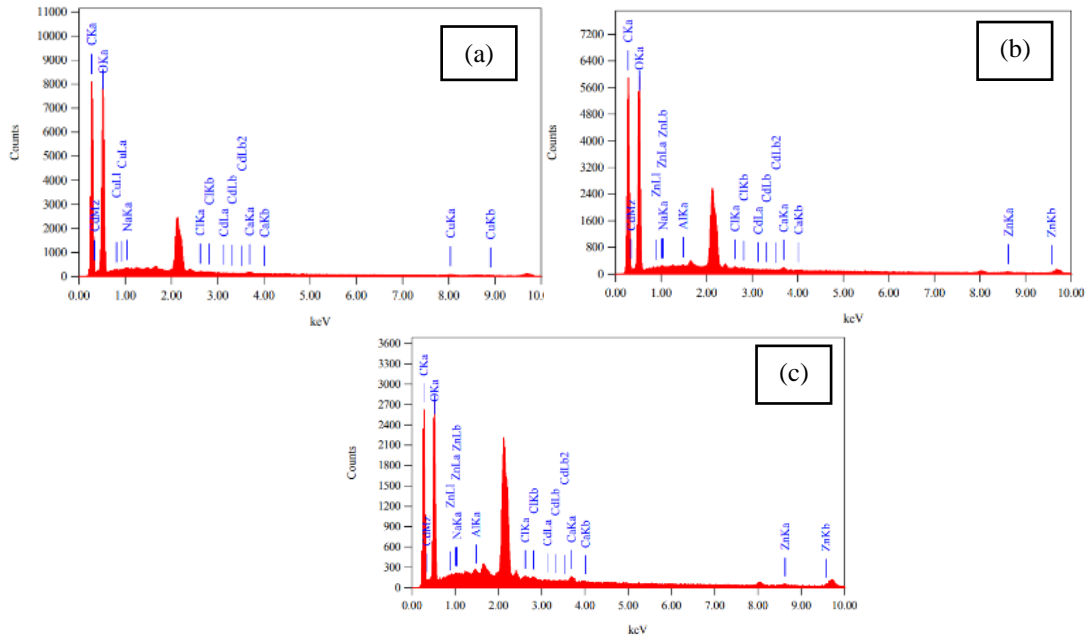


Figure 2. Test SEM-EDX Elements Klor, Calcium, Natrium (a) MCC Corn Husk (HCl 2 N); (b) MCC Corn Husk (HCl 4 N); (c) MCC Corn Husk (HCl 6 N)

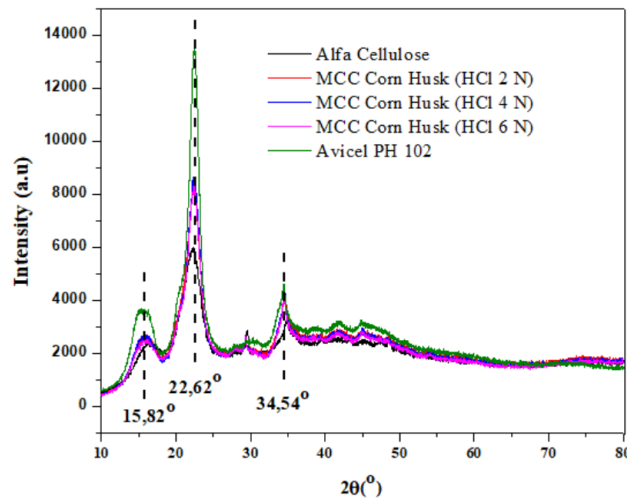


Figure 3. X-ray diffraction patterns of Corn Husk, alfa cellulose, MCC Corn Husk and Avicel PH 102

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.

Funding: This research received no external funding

Acknowledgments: The author would like to acknowledge the support from Stifar Yayasan Pharmasi Semarang

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

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