OPTIMIZATION OF CARBOXYMETHYL CELLULOSE PRODUCTION FROM RICE STRAW USING RESPONSE SURFACE METHODOLOGY

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Rice straw is an abundant lignocellulosic residue with potential as a feedstock for carboxymethyl cellulose (CMC) production. This study optimized sodium monochloroacetate (NaMCA) dosage and reaction time for obtaining CMC derived from Ciherang rice straw using response surface methodology with a central composite design (RSM-CCD). The optimal conditions, 4.40 g NaMCA and 3 h, yielded 25.31% CMC with pH 7.58, whiteness of 74.23%, moisture content of 4.12%, purity of 94.09%, viscosity of 6.8 cP, NaCl content of 5.91%, and desirability of 0.58. ANOVA showed that NaMCA dosage and reaction time significantly affected the yield and pH, whereas the other responses were less sensitive. FTIR confirmed characteristic CMC functional groups, and SEM revealed a distinct fibrillar morphology relative to commercial CMC. Although the product did not fully satisfy food-grade requirements for viscosity and NaCl content, the findings support Ciherang rice straw as a sustainable CMC feedstock and establish a predictive framework for further process optimization.

Keywords: rice straw, carboxymethyl cellulose, agricultural waste, physicochemical properties, optimization, sodium monochloroacetate, reaction time

INTRODUCTION

China, India, and Indonesia are among the leading rice-straw-producing countries in Asia.[1,2] Indonesia produced 52.66 million tons of milled dry grain (GKG) in 2024, with rice straw generation estimated at 50–68% of grain production. Approximately 18–30% of rice straw is burned or left in agricultural fields because of inadequate residue management,[3] thereby contributing to carbon emissions and air pollution. These environmental concerns underscore the need for more effective and value-added utilization of rice straw.

Rice straw biomass contains cellulose, hemicelluloses, and lignin, making it a potentially valuable lignocellulosic feedstock.[4] Carboxymethyl cellulose (CMC) is a cellulose derivative produced through chemical modification of cellulose[5] and is widely used as a hydrocolloid in both food and non-food applications.[6] Its broad functionality, ease of use, and relatively low cost contribute to its substantial industrial demand.[7]

Previous studies have investigated CMC production from a range of lignocellulosic resources, including bagasse,[8] water hyacinth,[9] banana peel,[10] corn husk,[11] cashew tree material,[12] sago palm waste,[13] papaya peel,[14] rice straw,[15] and oil-palm-derived lignocellulosic waste,[16] including investigations of the influence of sodium monochloroacetate (NaMCA) concentration on CMC quality.

A previous study[17] reported that the use of 2 g NaMCA produced a CMC yield of 200.68%, a viscosity of 300 cP, a pH of 7.8, a degree of substitution of 0.4, and a moisture content of 2.73%. Hariani *et al*.[18] reported that CMC produced from palm-frond cellulose using 8 g NaMCA had a degree of substitution of 0.81, a viscosity of 5.72 cP, a pH of 7.67, and a purity of 99.56%. Masrullita *et al*.[19] further demonstrated that NaMCA dosage markedly influences the chemical characteristics of CMC. Nevertheless, studies on CMC production specifically from rice straw remain comparatively limited.

Ciherang rice straw has been reported to contain 31.37% cellulose, 18.14% hemicelluloses, and 7.93% lignin,[20,21] supporting its potential as a cellulose source for the production of carboxymethyl cellulose. Despite this potential, research focusing specifically on local Ciherang rice straw as a raw material for CMC remains scarce. Moreover, studies integrating the physicochemical characterization of rice-straw-derived CMC with optimization based on central composite design (CCD) and response surface methodology (RSM) are still limited.

The novelty of this study lies in the use of Ciherang rice straw, a locally available and underexplored lignocellulosic biomass, as a feedstock for CMC production. In contrast to studies based on other agricultural residues, such as bagasse, banana peel, and water hyacinth, this work evaluates the valorization of rice straw through an optimized carboxymethylation process. By integrating response surface methodology (RSM) with a central composite design (CCD), the study establishes predictive regression models describing the effects of sodium monochloroacetate concentration and reaction time on yield, purity, viscosity, and other physicochemical properties of CMC. Structural and morphological characterization by FTIR and SEM further confirms functional groups consistent with commercial CMC, while revealing distinct morphological features attributable to the raw material and synthesis conditions. These findings contribute to the development of cellulose derivatives from agricultural residues and support the potential use of rice straw as a sustainable feedstock for high-value polysaccharide-based materials.

Given these research gaps, this study aimed to optimize NaMCA concentration and reaction time using an RSM-CCD approach to obtain high-quality CMC from Ciherang rice straw and to characterize the physicochemical properties of the resulting product.

EXPERIMENTAL

Materials

The principal raw material was Ciherang rice straw collected from a local field in Ciagel Village, Kibin District, Serang, Indonesia. The straw was collected 2–3 days after harvest in a dry, brownish-yellow condition. The additional materials used were CH_3COOH (Merck, Germany), isopropanol (Merck, Germany), ethanol (PT. Sarana Mitra Anugrah), sodium monochloroacetate (NaMCA; Merck, Germany), NaOH (Merck, Germany), NaCl (PT. Sarana Mitra Anugrah), and NaOCl (PT. Sarana Mitra Anugrah).

Preparation and pretreatment of rice straw

Sample preparation followed the method of Nur *et al*.,[21] with minor modifications to the drying and sieving steps. Rice straw was washed with water, dried in an oven at 60 °C for 24 h, cut into small pieces with scissors, and milled using a blender. The resulting powder was passed through a 60-mesh sieve.

Cellulose extraction

Cellulose extraction from rice straw followed the method of Masrullita *et al*.,[19] with modifications to the hydrolysis, bleaching, and drying stages. The production of rice-straw-derived CMC followed previously reported methods,[22,23] with modifications to the carboxymethylation stage. A 25 g portion of rice straw powder was hydrolyzed with 125 mL of 15% NaOH at 100 °C for 3 h. The delignified material was filtered, after which 5 mL of 10% glacial acetic acid and 10 g of NaCl were added. The sample was filtered again and washed with distilled water. The cellulose was then subjected to a two-stage bleaching process. In the first stage, the sample was treated with 125 mL of 15% NaOCl and 500 mL of distilled water, heated at 60 °C for 3 h, and subsequently filtered. In the second stage, the cellulose was washed with 50 mL of 15% NaOCl and rinsed with 250 mL of distilled water. After bleaching, the wet cellulose was dried at 60 °C for 24 h and then ground using a blender.

Production of carboxymethyl cellulose from rice straw

CMC production comprised two principal stages: alkalization and carboxymethylation. For alkalization, 5 g of powdered rice-straw cellulose, 80 mL of ethanol, 20 mL of isopropanol, and 20 mL of 10% NaOH were gradually combined in a glass beaker. The mixture was maintained at 25 °C for 1 h under continuous stirring at

500 rpm. For carboxymethylation, 2–8 g NaMCA was dissolved in 60 mL of ethanol, and the reaction was conducted at 55 °C for 1–3 h. The mixture was neutralized by adding 2 mL of glacial acetic acid, filtered, and washed four times with 50 mL of ethanol. The wet CMC was then dried at 60 °C for 24 h.

Characterization and analysis of CMC

Analysis of physicochemical responses

Yield,[24] pH,[25] moisture content,[26] degree of whiteness,[27] viscosity,[28] purity,[29] and NaCl content[30] were determined according to the corresponding analytical methods.

CMC yield was expressed as a percentage and calculated from the ratio of the mass of the obtained CMC to the initial mass of rice straw. Yield was calculated using Equation (1):

$$\text{Yield (\%)} = \frac{\text{CMC weight (g)}}{\text{Rice straw weight (g)}} \times 100\% \quad (1)$$

The pH was determined by dissolving 1 g of CMC in 100 mL of distilled water, heating the mixture to 60–70 °C, and stirring until complete dissolution. The solution was subsequently cooled to room temperature, and the pH was measured using a pH meter.

Moisture content was determined by placing 1 g of CMC in a preweighed empty cup. The sample was dried in an oven at 105 °C for 6 h, cooled in a desiccator for 30 min, and weighed until a constant mass was obtained. Moisture content (%) was calculated using Equation (2):

$$\text{Moisture content (\%)} = \frac{w1_{(g)} - w2_{(g)}}{w1_{(g)}} \times 100\ \% \quad (2)$$

where w1 and w2 denote the sample masses before and after drying, respectively, in grams.

The degree of whiteness was measured using a Minolta CR-410 Chromameter. The instrument was calibrated before measurement. Each CMC sample was positioned directly beneath the Chromameter without an intervening gap, after which the measurement was initiated and reflectance was recorded. Color was expressed using the L* (lightness), a* (+red, −green), and b* (+yellow, −blue) coordinates. These values were used to calculate the degree of whiteness according to Equation (3):

$$W = 100 - \sqrt{(100-L^*)^2+(a^*)^2+(b^*)^2} \quad (3)$$

where W denotes the degree of whiteness, L* represents lightness, a* represents the red–green coordinate, and b* represents the yellow–blue coordinate.

CMC viscosity was measured by dissolving 1 g of sample in 100 mL of distilled water. The solution was heated to 80 °C and stirred for 10 min until homogeneous. Viscosity was then measured using a viscometer equipped with a spindle and expressed in centipoise (cP).

NaCl content was determined by weighing 0.5 g of CMC into an Erlenmeyer flask, adding 100 mL of distilled water, and heating the mixture to 70 °C. A 10 mL aliquot was transferred to a 250 mL Erlenmeyer flask, and five drops of 5% K_2CrO_4 indicator were added. The solution was titrated with 0.0105 N $AgNO_3$, and the NaCl content was calculated using Equation (4):

$$\text{NaCl content (\%)} = \frac{V_{(mL)} \times N_{(N)} \times 58.45 \times FP}{A_{(g)} \times (100-B_{(\%)})} \times 100 \quad (4)$$

where V is the volume of 0.0105 N $AgNO_3$ (mL); N is the normality of $AgNO_3$ (N); the molecular weight of NaCl is 58.45; A is the sample mass (g); B is the moisture content (%); and FP is the dilution factor.

CMC purity was determined according to the method of Wijayani *et al*.[27] and calculated using Equation (5):

$$\text{Purity (\%)} = 100\ \% - \text{NaCl content (\%)} \quad (5)$$

Analysis of optimized CMC

Among all experimental treatments, only the samples exhibiting the highest purity and the highest yield were selected for FTIR and SEM analyses. Sample selection was based on the analytical results processed using Design-Expert software.

Functional-group characterization of the selected CMC samples was performed by Fourier transform infrared (FTIR) spectroscopy using a Thermo Scientific Nicolet iS-10 instrument. Pellets were prepared by grinding 2–5 mg of CMC with 200–250 mg of KBr. Transmission spectra of the Na-CMC samples were recorded over a wavenumber range of 4000–400 cm^{-1}. Two samples were analyzed by FTIR: the sample with the highest yield and the sample with the highest purity.

CMC morphology was examined by scanning electron microscopy (SEM) using a JEOL JSM-IT200 instrument. Samples were mounted on SEM specimen holders with double-sided carbon tape, with the cross-sectional surface oriented vertically toward the objective lens. The SEM was operated at an accelerating voltage of 20 kV, a spot size of 50, and a working distance of 10 mm. The 10 mm working distance was selected as a compromise for signal acquisition to optimize X-ray detection and counting. Only the samples with the highest yield and the highest purity were subjected to SEM analysis.

Experimental design and statistical analysis

A central composite design (CCD) implemented in Design-Expert 13 was used to optimize NaMCA dosage and reaction time with respect to the physicochemical properties of CMC. The independent variables were sodium monochloroacetate mass (X) and reaction time (Y). The lower, central, and upper factor levels are presented in Table 1.

The CCD, including five center-point runs, generated 13 experimental treatments, as shown in Table 2. Data analysis comprised three stages: response analysis, optimization, and verification.

Table 1
Levels of the experimental variables

Variable		Limits		
		Lower	Central	Upper
Sodium monochloroacetate (g)	X	2.00	5.00	8.00
Reaction time (h)	Y	1.00	2.00	3.00

Table 2
Central composite design for optimization

Run	Factor 1 NaMCA (X)	Factor 2 Reaction time (Y)
1	5.00	2.00
2	8.00	3.00
3	5.00	2.00
4	2.00	1.00
5	0.76	2.00
6	2.00	3.00
7	8.00	1.00
8	9.24	2.00
9	5.00	3.41
10	5.00	2.00
11	5.00	0.58
12	5.00	2.00
13	5.00	2.00

Response analysis

Response models were evaluated using analysis of variance (ANOVA). Model selection considered statistical significance and model order, with an acceptable model requiring a significant overall ANOVA and a non-significant lack of fit to establish the effects of the experimental factors on each response.[31] ANOVA was used to determine whether the tested factors significantly influenced the responses. The coefficient of determination (R^2) ranges from 0 to 1, with values closer to 1 indicating a greater proportion of response variability explained by the fitted model.

Optimization analysis

Numerical optimization was performed using the fitted response models to identify the optimal factor combination. The responses considered were yield, pH, moisture content, degree of whiteness, viscosity, purity, and NaCl content. Design-Expert generated candidate solutions based on overall desirability, which ranges from 0 to 1; values approaching 1 indicate a greater ability of the factor combination to satisfy the specified response targets. The objective of the optimization was therefore to maximize desirability by identifying an appropriate combination of the experimental variables.[32]

Verification phase

After the optimal formulation was identified, the formulation predicted by Design-Expert 13.0 was prepared experimentally. Verification was conducted to assess agreement between the predicted and measured response values. Two replicate verification experiments were performed, and the measured responses were compared with the values predicted by Design-Expert 13. The verified formulation was subsequently selected as the best formulation for rice-straw-derived CMC.[32]

RESULTS AND DISCUSSION

Response analysis of CMC optimization variables

The response values obtained from the 13 experimental runs are presented in Table 3. Yield, pH, moisture content, degree of whiteness, viscosity, purity, and NaCl content ranged from 21.74–28.91%, 6.66–8.77, 3.21–5.14%, 73.37–80.34%, 5.73–7.60 cP, 36.84–95.10%, and 4.90–63.16%, respectively.

The response-model analysis performed using Design-Expert 13.0 is summarized in Table 4. A linear model was selected for all responses on the basis of the sequential model sum of squares, lack-of-fit test, and model summary statistics. In the sequential model assessment, the preferred model was identified from the corresponding p-values, while the lack-of-fit criterion favored models with larger p-values. Model summary statistics additionally considered lower standard deviation (SD), higher R^2, and lower predicted residual error sum of squares (PRESS). Collectively, these criteria supported the selection of the linear model for subsequent response analysis.

The linear regression equations for each response are presented in Table 5, while Figure 1 illustrates the corresponding response surfaces and contour plots. The positive coefficients of NaMCA and reaction time in the yield model indicate increasing CMC yield with increases in both factors. Consistently, Figure 1a shows higher predicted yield as the response surface approaches the yellow-to-orange region.[33]

Changes in contour color indicate variation in the magnitude of the response across the experimental domain.[34,35] Consistent with previous findings,[36] increasing NaMCA addition increased CMC yield because NaMCA participates in substitution reactions during carboxymethylation. Increased yield may also be associated with longer contact between the solvent and cellulose.[37] Under suitable temperature and reaction-time conditions, a greater number of hydroxyl groups in alkali cellulose are replaced by carboxymethyl groups, thereby increasing CMC formation.[38]

The pH response decreased with increasing NaMCA dosage and reaction time (Table 3). In the contour plot (Fig. 1b), the pH response varied across the blue-to-orange color range. NaMCA dosage and reaction time significantly affected pH; however, the maximum response was not reached within the evaluated domain because the contour scale did not extend fully into the yellow-to-orange region. A previous study[39] reported that increasing trichloroacetic acid concentration from 20% to 30% decreased the pH from 8.32 to 7.48. Fatimah[40] similarly noted that the pH of CMC is influenced by neutralization conditions during the addition of 90% acetic acid. The SNI 06-3736-1995[25] and FAO standards specify a pH range of 6–8.5 for CMC. All experimental samples met this range, except R4, which had a pH of 8.77 (Table 3), indicating incomplete neutralization.

Table 3
Response data for the 13 experimental runs

Sample	Response						
	Yield (%)	pH	Moisture content (%)	Degree of whiteness (%)	Viscosity (cP)	Purity (%)	NaCl content (%)
R1	25.04	7.36	5.14	75.63	5.73	81.05	18.95
R2	28.91	6.66	4.82	75.17	6.00	81.84	18.16
R3	26.89	6.72	3.83	77.12	6.27	76.65	23.35
R4	21.74	8.77	3.21	76.31	6.13	69.05	30.95
R5	23.66	8.42	5.12	73.37	6.53	95.10	4.90
R6	24.51	8.22	3.98	76.24	6.13	84.07	15.93
R7	26.23	7.47	5.11	77.00	5.73	80.84	19.16
R8	28.03	6.76	4.30	80.34	5.87	36.84	63.16
R9	27.97	6.87	3.66	77.24	7.07	62.50	37.50
R10	23.56	7.32	3.86	75.97	7.60	88.17	11.83
R11	23.50	7.09	4.92	73.26	6.40	87.06	12.94
R12	24.86	6.83	4.29	76.90	5.87	76.66	23.34
R13	25.68	7.39	4.39	76.71	6.93	64.41	35.59

Table 4
Response model analysis

Response	Model	Sequential model sum of squares			Lack of fit			Model summary statistics				
		f	p	Significance	f	p	Significance	SD	R^2	Adj R^2	Pred R^2	PRESS
Yield	Linear	29.53	<0.0001	Significant	0.20	0.96	Non-significant	0.88	0.85	0.83	0.79	10.71
pH	Linear	9.45	0.0050	Significant	2.51	0.19	Non-significant	0.44	0.65	0.58	0.37	3.61
Moisture content	Linear	0.62	0.56	Non-significant	1.84	0.29	Non-significant	0.65	0.11	-0.07	-0.72	8.19
Degree of whiteness	Linear	6.48	0.13	Non-significant	9.85	0.02	Significant	1.60	0.34	0.20	-0.41	54.52
Viscocity	Linear	0.66	0.54	Non-significant	0.28	0.92	Non-significant	0.59	0.12	-0.06	-0.27	4.97
Purity	Linear	1.79	0.21	Non-significant	3.73	0.11	Non-significant	14.05	0.26	0.12	-0.50	4034.31
NaCl Content	Linear	1.79	0.22	Non-significant	3.73	0.11	Non-significant	14.05	0.26	0.12	0.50	4035.10

Table 5
Linear regression equations for the response variables

Response*	Regression equation
Yield	$Y = 25.43 + 1.88X_1 + 1.47X_2$
pH	$Y = 7.38 – 0.65\ X_1 – 0.21X_2$
Moisture content	$Y = 4.36 + 0.19X_1 – 0.16X_2$
Degree of whiteness	$Y = 76.25 + 1.18X_1 + 0.47X_2$
Viscosity	$Y = 6.33 – 0.18X_1 + 0.15X_2$
Purity	$Y = 75.71 – 9.11X_1 – 2.34X_2$
NaCl content	$Y = 24.29 + 9.10X_1 + 2.34X_2$

*Note: Y – NaCl content, X_1 – NaMCA concentration, X_2 – reaction time

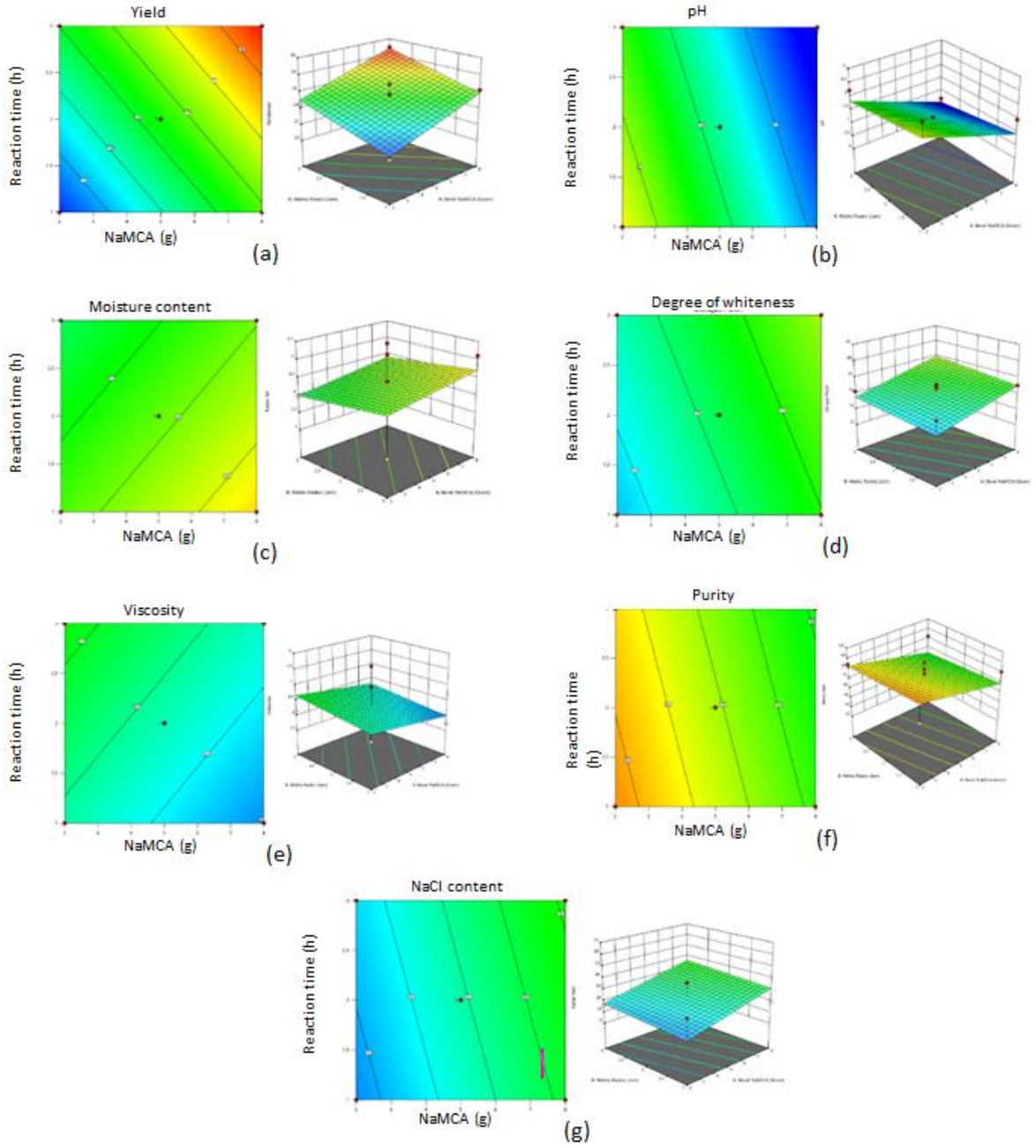


Figure 1: Response surface and contour plots for (a) yield, (b) pH, (c) moisture content, (d) degree of whiteness, (e) viscosity, (f) purity, and (g) NaCl content

The regression equation in Table 5 indicates that CMC moisture content increased with NaMCA dosage, but decreased with reaction time, as illustrated in Figure 2c. The positive relationship between NaMCA dosage and moisture content is consistent with previous findings,[19] in which 9 g NaMCA produced CMC with relatively high moisture content because of the water-binding capacity of CMC. Conversely, longer reaction times reduced moisture content, which has been related to changes in interactions between cellulose groups. Similar trends have been reported previously,[10,41] where prolonged reaction time increased the separation between cellulose groups and weakened intermolecular interactions, thereby reducing water-binding capacity. The moisture contents obtained in this study (3.21–5.14%; Table 3) were below 12% and therefore met the requirements of SNI[23] and FAO. Because CMC is hydrophilic and hygroscopic, controlling moisture content is important for minimizing physical changes during storage.[8,42]

NaMCA dosage and reaction time significantly affected the degree-of-whiteness response (Fig. 1d), with NaMCA exerting a stronger effect than reaction time (Table 5). Increasing both factors

increased the degree of whiteness. In Figure 1d, blue represents lower whiteness values and green represents higher values. Wijayani *et al*.[29] reported that CMC whiteness is influenced by browning reactions during processing and that residual lignin in cellulose may contribute to a yellowish appearance. The CMC produced in this study was light brown to yellowish with a cotton-like texture. The highest whiteness value, 80.34%, was obtained at 9.24264 g NaMCA and a reaction time of 2 h (Table 3). Whiteness is also influenced by bleaching with sodium hypochlorite (NaOCl) during cellulose preparation. Rachmawaty *et al*.[43] reported that hypochlorite ions are strong oxidizing agents capable of cleaving ether bonds in lignin, thereby increasing fiber whiteness. The CMC products obtained from the 13 experimental runs are shown in Figure 2.

Reaction time was positively associated with CMC viscosity (Table 5). Figure 1e shows only a slight color variation, indicating that the treatment effects on viscosity were not significant. Increasing NaMCA dosage decreased viscosity, consistent with previous observations[43] that higher NaMCA levels can reduce CMC viscosity. Conversely, increasing reaction time increased viscosity and produced a more homogeneous and transparent solution. This trend is consistent with previous findings,[44] indicating that longer carboxymethylation times can increase CMC viscosity, potentially in relation to ether solubility and the amount of suspended material in the solution.

Pujokaroni *et al*.[45] reported that viscosity is also influenced by the degree of polymerization; longer CMC molecular chains are associated with a higher degree of polymerization and, consequently, greater solution viscosity. A relatively low NaOH concentration may also contribute to low viscosity. In this study, the NaOH concentration was 10%, which may have limited the extent of the carboxymethylation reaction.[46] The Food Chemical Codex standard specifies a viscosity of ≥25 cP for CMC used as a food ingredient. The viscosity values obtained in this study (Table 3) were below 25 cP.

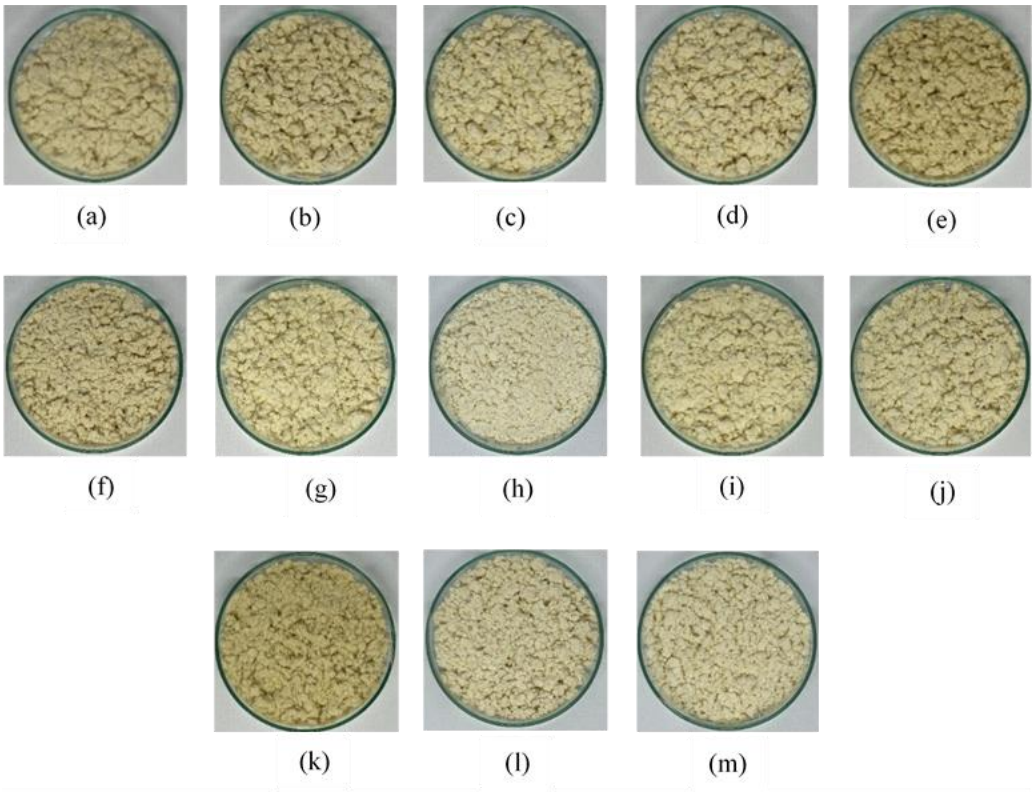


Figure 2: CMC products obtained from experimental runs (a) R1, (b) R2, (c) R3, (d) R4, (e) R5, (f) R6, (g) R7, (h) R8, (i) R9, (j) R10, (k) R11, (l) R12, and (m) R13

Increasing NaMCA dosage and reaction time decreased the predicted purity response (Table 5). Figure 1f shows only limited color variation, indicating that the effects of these factors on purity were not significant. Wijayani *et al*.[29] reported that CMC purity decreases with increasing NaMCA dosage because greater amounts of sodium chloride and sodium glycolate are formed. Pujokaroni *et al*.[45] also

reported that prolonged reaction time can reduce purity. Comparable results have been observed for CMC prepared from palm-fiber waste following ozone pretreatment.

This behavior has been associated with the use of 30% trichloroacetic acid, for which longer reaction times promote the formation of greater amounts of sodium chloride and sodium glyconate.[47] Pitaloka *et al*.[9] further suggested that low CMC purity may result from the formation of alternative crystalline structures in cellulose. The purity of rice-straw-derived CMC in this study ranged from 36.86% to 95.10%, which remained below the SNI Class I requirement of ≥99.5%,[27] but fell within the Class II quality threshold of 65.0%. The maximum purity obtained for rice-straw CMC was higher than the 90.9% purity reported for water-hyacinth-derived CMC.

NaMCA dosage and reaction time increased the NaCl-content response (Table 5). In Figure 1g, the NaCl response changes from blue, representing lower values, to green, representing higher values. Higher NaCl content reflects greater formation of by-products, such as sodium chloride and sodium glyconate, and is therefore associated with lower CMC purity. This finding is consistent with previous work[48] showing that the addition of 8–10 g NaMCA promotes the formation of side products, including NaCl. Pushpamalar *et al*.[13] reported that CMC synthesis under optimal reaction conditions generates NaCl as a by-product of the reaction between alkali-cellulose hydroxyl groups and the carboxymethyl groups of sodium monochloroacetate. NaCl and sodium glycolate can be reduced through washing after neutralization with glacial acetic acid, using methanol or ethanol as washing solvents. The NaCl contents obtained in this study did not meet the Class I CMC requirement of 0.25%, but remained within the Class II category, for which no NaCl limit is specified.

Optimal formulation based on CMC optimization variables

Numerical optimization was performed by assigning importance weights to the desired response targets. Importance was scored from 1 (+) to 5 (+++++), with higher scores indicating greater priority in the optimization procedure. The optimization criteria used to identify the best CMC formulation in Design-Expert are summarized in Table 6.

Yield was assigned a ‘maximize’ goal with the highest importance level, 5 (+++++), because the study sought to maximize CMC recovery. The pH and moisture-content responses were constrained within their specified ranges and were also assigned an importance level of 5 (+++++). The degree of whiteness and viscosity were assigned ‘maximize’ goals with an importance level of 3 (+++) to favor higher whiteness and viscosity. Purity and NaCl content were assigned an importance level of 5 (+++++), with the respective objectives of increasing product purity and reducing NaCl content.

Following numerical optimization, Design-Expert generated several candidate formulations with desirability values ranging from 0 to 1. Optimization seeks to maximize overall desirability by identifying an appropriate combination of the experimental factors.[32] The selected optimal formulation had a desirability of 0.59 and consisted of 4.40 g NaMCA with a reaction time of 3 h.

Table 6
Optimization criteria for the response variables

Response	Goal	Lower limit	Upper limit	Importance
NaMCA weight (g)	in range	2	8	+++++
Reaction time (h)	in range	1	3	+++++
Yield (%)	maximize	21.74	28.91	+++++
pH	in range	6.66	7.47	+++++
Moisture content (%)	in range	3.21	5.14	+++++
Degree of whiteness (%)	maximize	73.26	80.34	+++
Viscosity (cP)	maximize	5.73	7.60	+++
Purity (%)	maximize	36.84	95.10	+++++
NaCl content (%)	minimize	4.90	63.16	+++++

Table 7
Verification of the optimal formulation

Response	Measured value	95% PI lower	95% PI upper
Yield (%)	25.31	24.37	28.68
pH	7.58	6.21	8.38
Moisture content (%)	4.12	2.55	5.75
Degree of whiteness (%)	74.23	72.57	80.39
Viscosity (cP)	6.80	5.08	7.96
Purity (%)	94.09	40.79	109.58
NaCl content (%)	5.91	-9.58	59.21

Verification of optimized CMC formulation

The verification stage evaluated the predictive performance of the optimal formulation generated by Design-Expert. Predicted response values for the formulation containing 4.40 g NaMCA and a reaction time of 3 h are presented in Table 7. The measured verification values were consistent with the model predictions for all responses and fell within the corresponding 95% prediction-interval (PI) lower and upper bounds.[30]

Characterization of optimized CMC

The 13 experimental runs were evaluated in Design-Expert to select two samples for FTIR and SEM characterization. Samples were selected on the basis of the highest purity and the highest yield. Sample R5, produced with 0.78 g NaMCA and a reaction time of 2 h, exhibited the highest purity, whereas sample R2, produced with 8 g NaMCA and a reaction time of 3 h, exhibited the highest yield.

Functional-group analysis of optimized CMC

FTIR spectroscopy was used to characterize the organic components, chemical bonds, and functional groups of the CMC samples. The functional-group assignments for samples R5 and R2 are summarized in Table 8. The FTIR spectrum of commercial CMC (Fig. 3a) exhibited a band at 3281.23 cm^{-1} corresponding to the -OH group characteristic of cellulose. The -CH group was observed at 2925.32 cm^{-1}, while the C=O group appeared at 1589.99 cm^{-1}. A band at 1414.52 cm^{-1} indicated the presence of $-CH_2-$, whereas the -O- group was observed at 1017.72 cm^{-1}. A characteristic CMC fingerprint band was observed at 847.82 cm^{-1} and was associated with C-H vibration of the β-glycosidic linkage between glucose units in cellulose (Table 8). The spectra of samples R5 (Fig. 3b) and R2 (Fig. 3c) similarly exhibited -OH, -CH, C-O, $-CH_2-$, and -O- groups, as well as bands associated with β-glycosidic linkages characteristic of CMC.

The FTIR spectra of the rice straw CMC sample with the highest purity (R5; Fig. 3b) and the highest yield (R2; Fig. 3c) exhibited functional groups broadly comparable to those of commercial CMC (Fig. 3a). However, the commercial CMC spectrum showed stronger and sharper absorption bands. Hapsari[49] emphasized the importance of effective alkalization and carboxymethylation during CMC synthesis. Less distinct absorption bands may indicate incomplete reaction and lower product quality. Incomplete cellulose swelling may have restricted NaMCA substitution during carboxymethylation.

Table 8
Functional groups identified in CMC samples

Functional group	Wavenumber (cm^{-1})		
	Commercial CMC	CMC sample R5	CMC sample R2
-OH	3281.23	3328.84	3330.41
-CH	2925.32	2901.52	2892.35
C=O	1589.99	1643.07	1595.67
$-CH_2$	1414.52	1426.19	1424.96
-O-	1017.72	1026.69	1026.69
β-1,4 glycosidic bond	847.82	896.68	895.75

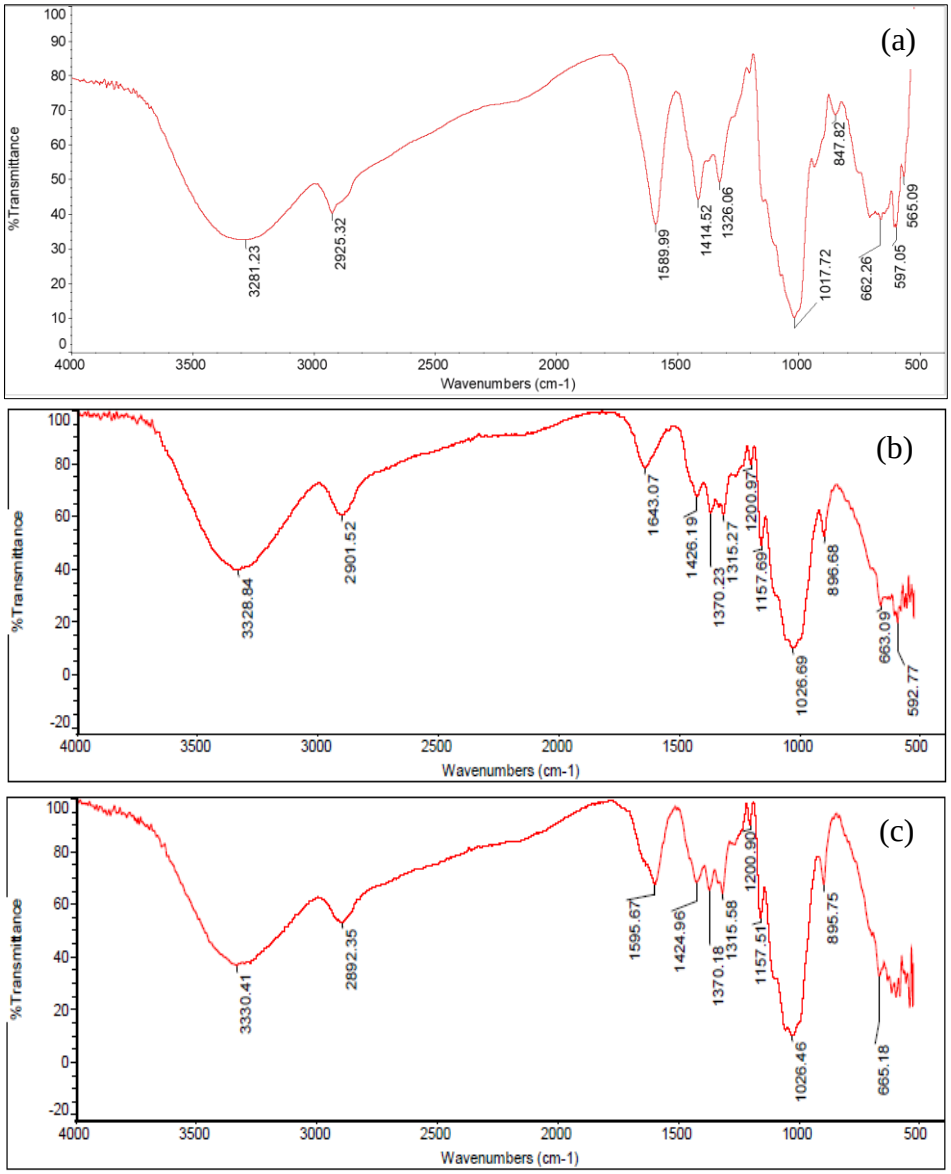
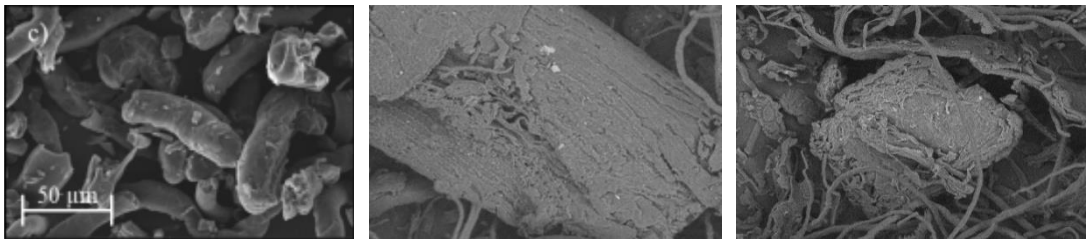


Figure 3: FTIR spectra of (a) commercial CMC, (b) sample R5, and (c) sample R2



(a) (b) (c)

Figure 4: SEM micrographs of (a) commercial CMC, (b) sample R5, and (c) sample R2 at 500× magnification

Morphological analysis of optimized CMC

SEM analysis was performed to examine CMC morphology and identify impurities that may have remained in the samples. Commercial CMC (Fig. 4a) consisted of relatively short fibrillar fragments with a size of 50 µm and exhibited finer particles than rice-straw-derived CMC. In contrast, the rice-straw CMC samples (Fig. 4b and 4c) observed at 500×magnification displayed rough fibrillar strands with fragmented regions. Individual particles could not be measured because the fibrils overlapped and

formed agglomerates. Klunklin *et al*.[50] attributed rough surfaces with irregularities and protrusions to less-developed and less-compact fiber structures.

Hastuti *et al*.[51] reported that the surface roughness of cellulose fibrils is commonly associated with extraction conditions involving strong chemical reagents and elevated temperatures. During etherification, cellulose reacts with the etherifying reagent to form CMC, and fibril surface roughness may decrease as the reaction proceeds. Changes in cellulose crystallinity may occur concurrently with this reduction in surface roughness.

Overall, this study contributes to the valorization of Ciherang rice straw, a local agricultural residue, as a cellulose source for CMC production and demonstrates the use of RSM-CCD to establish predictive relationships between synthesis variables and key physicochemical properties.

CONCLUSION

This study demonstrated the valorization of Ciherang rice straw, a local agricultural residue, into carboxymethyl cellulose (CMC) by optimizing sodium monochloroacetate (NaMCA) dosage and reaction time using response surface methodology with a central composite design. The optimal conditions were 4.40 g NaMCA and a reaction time of 3 h, producing CMC with a yield of 25.31%, pH of 7.58, whiteness of 74.23%, moisture content of 4.12%, purity of 94.09%, viscosity of 6.8 cP, and NaCl content of 5.91%, with an overall desirability of 0.58.

ANOVA indicated that NaMCA dosage and reaction time significantly affected yield and pH, whereas moisture content, whiteness, viscosity, purity, and NaCl content were less strongly influenced. FTIR analysis confirmed the presence of characteristic CMC functional groups, while SEM micrographs revealed a rough, fibrillar morphology distinct from that of commercial CMC.

Although the resulting CMC did not fully meet food-grade requirements for viscosity and NaCl content, the findings highlight the potential of Ciherang rice straw as a sustainable feedstock for cellulose derivatives. The predictive framework developed in this study provides a basis for further process refinement aimed at improving product quality and expanding the potential food and non-food applications of rice-straw-derived CMC.

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