

Optimization of bio-based cellulose-phosphate hydrogel production from rice husk waste using the Taguchi method

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Abstract: This work reports the development and statistical optimization of a fully Bio-based absorbent hydrogel synthesized from rice husk-derived cellulose via phosphoric acid crosslinking. Cellulose was extracted through sequential chemical treatments and subsequently converted into a phosphate-crosslinked hydrogel using a controlled synthesis process. A Taguchi L16 (4⁵) orthogonal array was employed to optimize four key synthesis parameters: cellulose content, reaction time, heating temperature, and phosphoric acid volume. Hydrogel structure and morphology were characterized using Fourier Transform Infrared Spectroscopy (FTIR) and Scanning Electron Microscopy (SEM). FTIR results confirmed the formation of phosphate ester linkages, indicating successful crosslinking, while SEM observations revealed a porous and interconnected network structure favorable for water absorption. The optimized hydrogel formulation achieved a maximum swelling ratio of 91.25 g/g, demonstrating effective absorbent performance despite the absence of synthetic monomers or grafting agents. These findings indicate that rice husk waste can be efficiently valorized into an environmentally benign absorbent material through a simple and statistically guided synthesis route, supporting sustainable hydrogel development and agricultural waste utilization.

Keywords: cellulose; extraction; hydrogel; rice husk; Taguchi method

1. Introduction

Bio-based absorbent hydrogels are three-dimensional crosslinked polymer networks capable of absorbing and retaining large amounts of water without dissolution [1]. Owing to their hydrophilic functional groups and interconnected porous structures, these materials have been widely explored for applications in agriculture, wastewater treatment, biomedical devices, and controlled-release systems [2]. Their ability to retain water and gradually release moisture makes them particularly attractive for sustainable agricultural and environmental applications. Despite their broad applicability, most commercially available absorbent hydrogels are synthesized from petrochemical-based polymers, such as polyacrylamide or acrylic acid derivatives [3][4]. Although these materials exhibit high swelling capacities, their limited biodegradability and reliance on non-renewable resources raise environmental and sustainability concerns [5]. Consequently, increasing research attention has been directed toward the development of environmentally benign hydrogels derived from renewable biomass sources [6][7][8].

Among various biomass feedstocks, agricultural residues have emerged as promising raw materials due to their abundance, low cost, and high cellulose content [9], Rice husk, a major by-product of

rice milling, is generated in large quantities worldwide and is often disposed of through open burning or landfilling, leading to environmental pollution[10]. However, rice husk contains a substantial amount of cellulose that can be isolated and chemically modified into value-added functional materials, including absorbent hydrogels [11][12]. Cellulose-based hydrogels derived from agricultural waste have demonstrated potential as sustainable alternatives to synthetic absorbents [13][14]. Nevertheless, many reported cellulose hydrogels still rely on synthetic grafting agents, multi-component crosslinkers, or complex modification routes to achieve high swelling performance [15]. Such approaches may compromise environmental compatibility, increase processing complexity, and limit large-scale applicability [16]. Moreover, several studies primarily focus on maximizing absorbency values without systematically investigating the influence of synthesis parameters on hydrogel structure–property relationships [17].

In this context, phosphoric acid has attracted interest as a single, effective, and environmentally compatible crosslinking agent for cellulose-based hydrogels [18]. Phosphate ester crosslinking can introduce hydrophilic functional groups while forming a stable three-dimensional network without the need for synthetic monomers [19]. However, limited studies have addressed the systematic optimization of synthesis conditions for phosphate-crosslinked cellulose hydrogels derived from agricultural waste. Therefore, this study presents the development and statistical optimization of a fully bio-based cellulose–phosphate hydrogel synthesized from rice husk waste. The Taguchi method combined with analysis of variance (ANOVA) was employed to evaluate the effects of cellulose content, reaction time, heating temperature, and phosphoric acid volume on the swelling behavior of the hydrogel. In addition, Fourier Transform Infrared Spectroscopy (FTIR) and Scanning Electron Microscopy (SEM) were used to elucidate the chemical structure and pore morphology of the synthesized material [20][21][22]. The novelty of this work lies in the utilization of rice husk-derived cellulose and phosphoric acid as a single crosslinking system, coupled with a statistically guided optimization approach. By avoiding synthetic monomers and complex grafting processes, this study provides a simple, scalable, and environmentally friendly route for converting agricultural waste into a functional absorbent hydrogel. The findings contribute to a deeper understanding of structure-property relationships in phosphate-crosslinked cellulose hydrogels and support the advancement of sustainable absorbent materials.

2. Material and methods

2.1 Research design

This study employed an experimental research design to synthesize and optimize a bio-based cellulose-phosphate hydrogel derived from rice husk waste. A Taguchi design of experiments using an L16 (4^5) orthogonal array was applied to systematically evaluate the effects of four synthesis parameters: cellulose content (A), reaction time (B), heating temperature (C), and phosphoric acid volume (D). The swelling ratio of the hydrogel was selected as the response variable. The overall experimental workflow is illustrated in Figure 1. Each control factor was investigated at four levels, as summarized in Table 1. The Taguchi approach enabled efficient exploration of parameter interactions while minimizing the number of experimental runs.

Table 1. Research design used in the experiment

Control Factor	Level 1	Level 2	Level 3	Level 4
Rice Husk Cellulose (A) (g)	1	2	3	4
Reaction Time (B) (min)	2	3	4	5
Heating Temperature (C) ($^{\circ}$ C)	30	40	50	60
Volume of Phosphoric Acid (D) (ml)	6	8	10	12

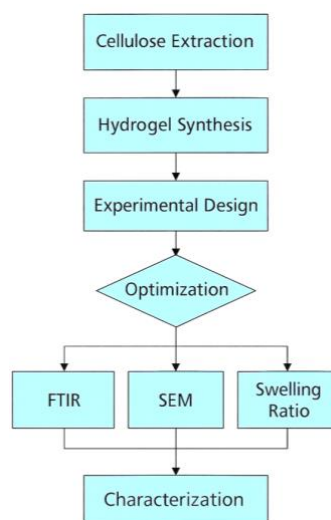


Figure 1. The flow of the research process

2.2 Materials

Rice husk was collected from a local rice milling facility in Palembang, Indonesia. Analytical-grade sodium hydroxide (NaOH), sodium sulfite (Na₂SO₃), nitric acid (HNO₃), acetic acid (CH₃COOH), and phosphoric acid (H₃PO₄, 85 wt%) were used as received without further purification. Distilled water was employed throughout all experimental procedures.

2.3 Cellulose extraction from rice husk

Cellulose was extracted from rice husk through sequential chemical treatments to remove non-cellulosic components. Initially, rice husk was treated with a mixture of nitric and acetic acids under controlled heating to remove hemicellulose and impurities. Delignification was subsequently carried out using an alkaline solution containing sodium hydroxide and sodium sulfite under continuous stirring. The resulting solid residue was thoroughly washed with distilled water and ethanol to remove residual chemicals and then dried in an oven at 60 °C for 19 h until constant weight was achieved [23]. The cellulose extraction process from rice husk was carried out based on the schematic shown in Figure 2.



Figure 2. The cellulose extraction process from rice husk

The cellulose yield was calculated according to Equation (1) [24].

$$Y (\%) = \left(\frac{W_c}{W_r} \right) \times 100\% \tag{1}$$

Where:

Y is the cellulose yield (%)

W_c is the weight of extracted cellulose (g)

W_r is the initial weight of rice husk (g)

2.4 Experimental design using Taguchi method

The Taguchi method uses an orthogonal array to evaluate multiple process parameters with fewer experiments. In this study, an L16 (4^5) orthogonal array was employed, comprising 16 experimental runs with four control factors at four levels each. The experimental matrix and factor assignments are presented in Table 2 [25][26].

Table 2. Orthogonal array $L_{16}(4^5)$

Experiment	Factor			
	A (g)	B (min)	C (°C)	D (ml)
1	1	20	30	6
2	1	30	40	8
3	1	40	50	10
4	1	50	60	12
5	2	20	40	10
6	2	30	30	12
7	2	40	60	6
8	2	50	50	8
9	3	20	50	12
10	3	30	60	10
11	3	40	30	8
12	3	50	40	6
13	4	20	60	8
14	4	30	50	6
15	4	40	40	12
16	4	50	30	10

Description:

A = Amount of Rice Husk Cellulose (g)

B = Reaction Time for Synthesis (minutes)

C = Heating Temperature (°C)

D = Volume of Phosphoric Acid (ml)

Therefore, this research conducted experiments using the $L_{16}(4^5)$ orthogonal array, where "L" denotes a Latin square design. The number "16" indicates the number of rows or experimental runs, the number "4" refers to the number of levels or variations of each control factor, and the number "5" indicates the number of columns or control parameters involved [27]. Signal-to-noise ratio (SNR) analysis was conducted using the "larger-the-better" criterion to identify optimal synthesis conditions that maximize swelling performance while minimizing variability.

2.5 Bio-based absorbent hydrogel synthesis process

Before crosslinking, the extracted cellulose was dispersed in 30 mL of distilled water and allowed to swell for 24 h. Phosphoric acid was then added as a crosslinking agent according to the experimental design [28].

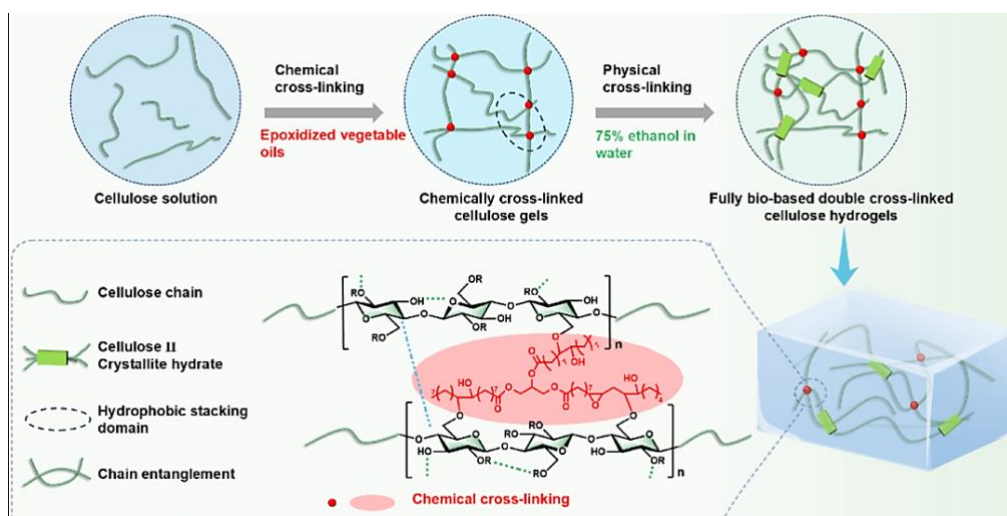


Figure 3. Schematic illustration of a bio-based double crosslinked cellulose hydrogel system

The schematic depicts the formation of a bio-based double cross-linked hydrogel derived from rice husk cellulose. Initially, rice husk cellulose chains are dissolved to form a homogeneous cellulose solution. Chemical cross-linking is introduced through reactions between hydroxyl groups of cellulose and epoxidized vegetable oils, resulting in a chemically cross-linked cellulose gel [29]. The mixture was subjected to microwave-assisted heating at a fixed power output, while reaction time and temperature were varied based on the Taguchi orthogonal array [30].

After completion of the reaction, the mixture was cooled to room temperature, filtered, and repeatedly washed with distilled water to remove unreacted species. The resulting hydrogel was dried to constant weight and stored for further characterization.



Figure 4. Bio-based absorbent hydrogel Synthesis Process

2.6 Swelling ratio measurement

The swelling behavior of the synthesized hydrogels was evaluated gravimetrically. Dried hydrogel samples were immersed in distilled water at room temperature until equilibrium swelling was achieved. Excess surface water was gently removed, and the swollen mass was recorded. Each measurement was conducted in triplicate to ensure reproducibility. The swelling ratio (SR) was calculated using Equation (2) [31].

$$SR = \frac{W_s - W_d}{W_d} \quad (2)$$

Where:

SR is the swelling ratio (g/g)

W_s is the weight of the swollen hydrogel (g)

W_d is the weight of the dry hydrogel (g)

2.7 Characterization techniques

Fourier Transform Infrared Spectroscopy (FTIR) was employed to identify functional groups and confirm chemical interactions between cellulose and phosphoric acid. Spectra were recorded in the range of 4000-400 cm^{-1} . Surface morphology and pore structure of the hydrogel were examined using Scanning Electron Microscopy (SEM). SEM analysis was performed on the hydrogel synthesized under optimal conditions to evaluate pore distribution and network architecture relevant to swelling behavior.

3. Results and discussion

This section presents the results obtained from synthesizing and characterizing Bio-based absorbent hydrogels derived from rice husk cellulose, followed by a comprehensive discussion. The optimization of synthesis parameters using the Taguchi method and relevant analyses, such as swelling performance, FTIR spectroscopy and SEM morphology images are also explained.

3.1 Swelling behavior of cellulose-phosphate hydrogels

The swelling behavior of the synthesized cellulose-phosphate hydrogels was evaluated to assess their water absorption capacity under different synthesis conditions. Table 3 summarizes the swelling ratios obtained from the 16 experimental runs based on the L16 (4^5) Taguchi orthogonal array. The swelling ratio values ranged from 42.75 to 91.25 g/g, indicating a strong dependence of absorbent performance on synthesis parameters. The highest swelling ratio (91.25 g/g) was achieved using 3 g of cellulose, a reaction time of 3 min, a heating temperature of 60 °C, and 10 mL of phosphoric acid. These results demonstrate that appropriate control of cellulose content and processing conditions is essential for achieving optimal network formation and water uptake. Although the swelling capacity obtained in this study is moderate compared with synthetic or grafted superabsorbent hydrogels reported in the literature, the performance is notable considering the fully bio-based composition and the absence of synthetic monomers or multi-component crosslinking systems. The swelling behavior observed reflects a balance between crosslink density and network porosity, which governs water diffusion and retention within the hydrogel structure.



Table 3. Swelling ratio results based on experimental design

Experiment	Factor				Swelling Ratio (g/g)
	A (g)	B (min)	C (°C)	D (ml)	
1	1	2	30	6	72.10
2	1	3	40	8	80.43
3	1	4	50	10	85.60
4	1	5	60	12	70.25
5	2	2	40	10	86.42
6	2	3	30	12	68.95
7	2	4	60	6	73.75
8	2	5	50	8	83.50
9	3	2	50	12	69.12
10	3	3	60	10	91.25
11	3	4	30	8	88.40
12	3	5	40	6	65.37
13	4	2	60	8	60.18
14	4	3	50	6	55.89
15	4	4	40	12	50.33
16	4	5	30	10	42.75

3.2 Signal-to-Noise Ratio (SNR)

To identify synthesis conditions that maximize swelling performance while minimizing experimental variability, signal-to-noise ratio (SNR) analysis was conducted using the “larger-the-better” criterion. The calculated SNR values for each experimental run are presented in Table 4. The signal-to-noise ratio was calculated according to Equation (3) [32].

$$SNR_{LTB} = -10 \cdot \log_{10} \left(\frac{1}{n} \sum_{i=1}^n \frac{1}{Y_i^2} \right) \quad (3)$$

Where: *SNR* denotes the signal-to-noise ratio (dB), *n* represents the number of replications, and *Y_i* represents the swelling ratio response value for the *i*-th experimental run. The signal-to-noise ratio (SNR) values calculated for each experimental condition are reported in Table 4.

Table 4. SNR values

Experiment	Swelling Ratio (g/g)	SNR
1	72.10	37.16
2	80.43	38.11
3	85.60	38.65
4	70.25	36.93
5	86.42	38.73
6	68.95	36.77
7	73.75	37.36
8	83.50	38.43
9	69.12	36.79
10	91.25	39.20
11	88.40	38.93
12	65.37	36.31
13	60.18	35.59
14	55.89	34.95
15	50.33	34.04
16	42.75	32.62

Among the experimental runs, Run 10 exhibited the highest SNR value (39.20 dB), indicating the most stable and consistent swelling performance. In contrast, Run 16 showed the lowest SNR value (32.62 dB), suggesting higher sensitivity to noise factors under those conditions.

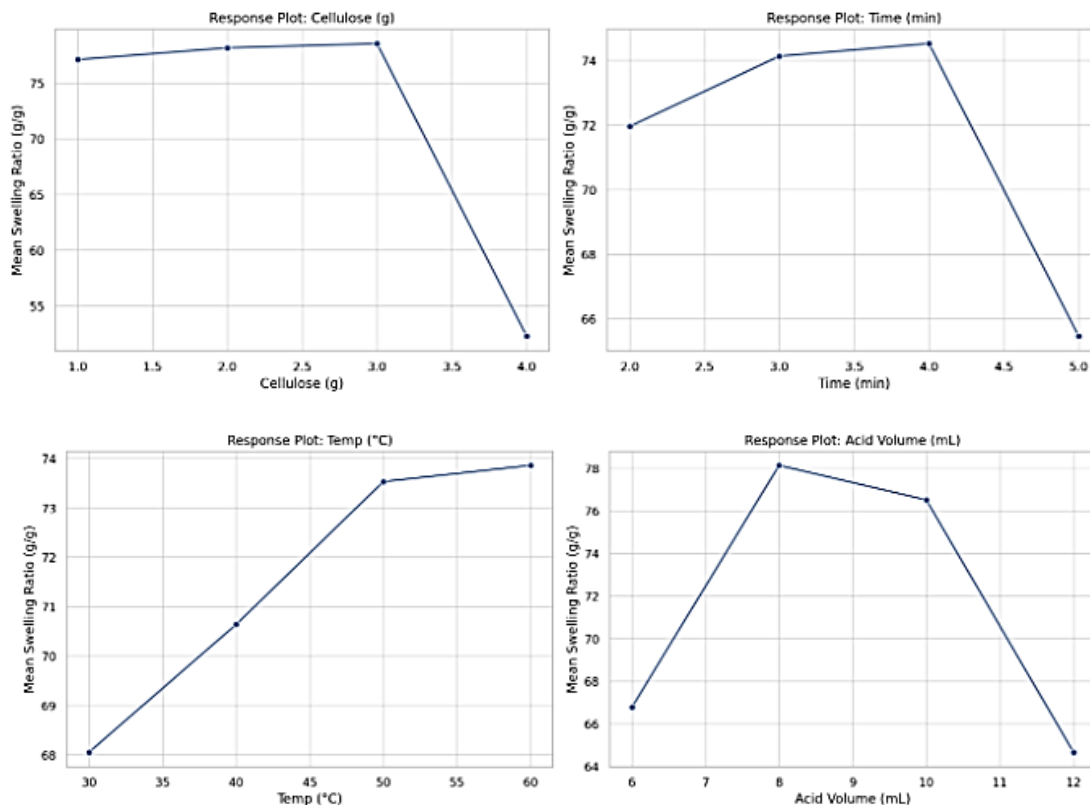


Figure 5. Means response graph for each parameter level

The main effects plot of SNR values revealed that phosphoric acid volume exerted the strongest influence on swelling performance, followed by cellulose content and heating temperature. Reaction time showed a comparatively smaller effect. These trends suggest that the extent of crosslinking and the availability of hydrophilic functional groups play a critical role in governing water absorption behavior.

3.3 Analysis of variance (ANOVA)

Analysis of variance (ANOVA) was employed to quantitatively evaluate the statistical significance of each synthesis parameter on the swelling ratio. The ANOVA results are summarized in Table 5.

Table 5. ANOVA summary for swelling ratio

Control Factor	Sums of Squares (SS)	DF	Mean Square (MS)	F-Value	P-Value
A: Rice Husk Cellulose (g)	1976.81	3	658.94	10.12	0.045
B: Reaction Time (min)	210.54	3	70.18	1.08	0.476
C: Heating Temperature (°C)	89.25	3	29.75	0.46	0.732
D: Volume of Phosphoric Acid (ml)	552.10	3	184.03	2.83	0.208
Error	195.40	3	65.13	-	-
Total	3024.10	15	-	-	-

The results indicate that cellulose content (Factor A) had a statistically significant effect on swelling behavior, with an F-value of 10.12 and a p-value of 0.045 ($p < 0.05$). This confirms that cellulose concentration is the primary factor influencing hydrogel absorbency, as it directly affects network density and the availability of hydrophilic sites. In contrast, reaction time (Factor B), heating temperature (Factor C), and phosphoric acid volume (Factor D) did not show statistically significant effects within the investigated ranges (p -values > 0.05). Although phosphoric acid volume showed a relatively higher F-value than the other non-significant factors, its effect was insufficient to reach statistical significance at the 95% confidence level. The relatively low error mean square value indicates good experimental control and supports the reliability of the Taguchi design and ANOVA results. Overall, the statistical analysis demonstrates that cellulose content governs the hydrogel's swelling performance, while the remaining parameters exert secondary effects.

3.4 FTIR analysis

Fourier Transform Infrared Spectroscopy (FTIR) was used to investigate chemical interactions and confirm crosslinking between rice husk cellulose and phosphoric acid. The FTIR spectrum of raw cellulose exhibited a broad absorption band at approximately $3340\text{--}3400\text{ cm}^{-1}$ corresponding to O-H stretching vibrations, as well as a characteristic peak near 1050 cm^{-1} associated with C-O stretching of the cellulose backbone.

Following crosslinking, notable spectral changes were observed in the hydrogel. The intensity of the O-H stretching band decreased and broadened, indicating the involvement of hydroxyl groups in esterification reactions. In addition, new absorption bands appeared in the regions of $1240\text{--}1260\text{ cm}^{-1}$ and $1020\text{--}1080\text{ cm}^{-1}$, which are characteristic of P=O stretching and P-O-C asymmetric stretching vibrations, respectively. The emergence of these phosphate-related bands provides clear evidence of phosphate ester bond formation between cellulose chains. These findings confirm successful crosslinking and the formation of a chemically stable cellulose-phosphate network, consistent with previously reported phosphate-crosslinked cellulose hydrogels.

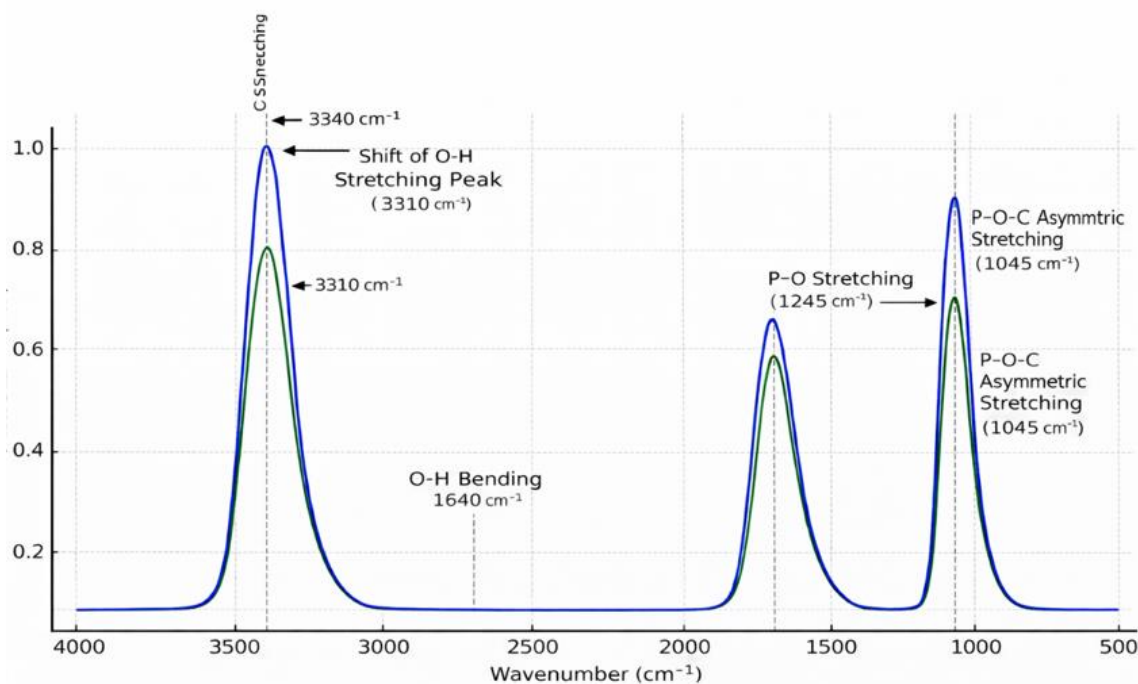


Figure 6. FTIR spectra of raw rice husk cellulose and phosphate-crosslinked cellulose hydrogel

3.5 SEM morphological analysis

Scanning Electron Microscopy (SEM) was employed to examine the surface morphology and pore structure of the cellulose-phosphate hydrogel synthesized under optimal conditions. The SEM micrographs revealed a porous, non-uniform surface architecture with an interconnected network.

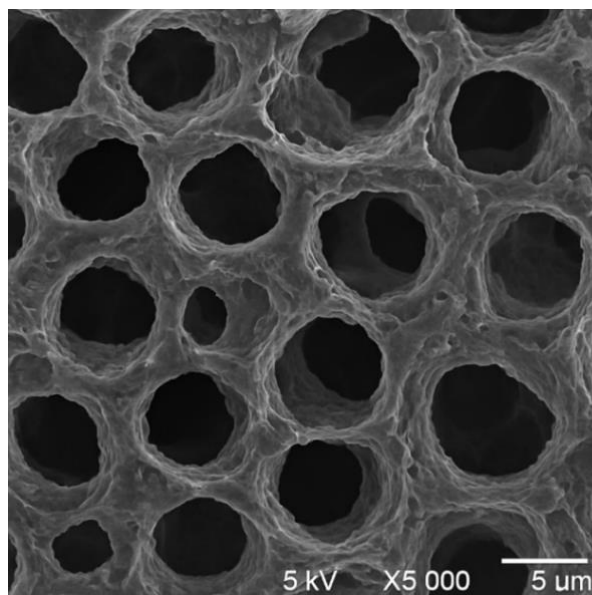


Figure 7. SEM micrograph of cellulose hydrogel at 5x magnification

Based on the scale bar and visual estimation, pore diameters ranged approximately from 2.0 to 6.5 μm , with the majority distributed in the 3–5 μm range. This porous morphology facilitates capillary-driven water diffusion and enhances swelling capacity by allowing efficient penetration of water molecules into the hydrogel matrix. The interconnected pore structure indicates effective phosphate ester crosslinking, which forms a three-dimensional network while preventing excessive chain packing. This morphological characteristic is consistent with the experimentally observed swelling behavior and supports the formation of a stable absorbent hydrogel network.

4. Conclusion

This study successfully demonstrated the synthesis and statistical optimization of a fully bio-based cellulose-phosphate hydrogel derived from rice husk waste using a Taguchi design of experiments. The hydrogel was produced through a simple crosslinking route, using phosphoric acid as a single crosslinking agent, without the use of synthetic monomers or complex grafting processes. The swelling performance of the hydrogel was strongly influenced by the synthesis conditions, with a maximum swelling ratio of 91.25 g/g under optimal conditions. Analysis of variance revealed that cellulose content was the only parameter with a statistically significant effect on swelling behavior, indicating its dominant role in governing network density and hydrophilic site availability. Other parameters, including reaction time, heating temperature, and phosphoric acid volume, contributed secondary effects within the investigated ranges. FTIR analysis confirmed the formation of phosphate ester linkages between cellulose chains, while SEM observations revealed a porous and interconnected network morphology that facilitates water diffusion and retention. These structural and chemical characteristics collectively explain the hydrogel's observed swelling behavior. Although the swelling capacity obtained is moderate compared with synthetic superabsorbent hydrogels, the achieved performance is notable given the fully bio-based composition, environmentally benign crosslinking strategy, and process simplicity. The findings highlight the potential of rice husk waste as a renewable feedstock for sustainable absorbent materials and provide

a reproducible framework for optimizing cellulose-based hydrogels using statistical design approaches. This work contributes to advancing environmentally friendly hydrogel systems for applications requiring water retention and to sustainable material development.

Author's declaration

Author contribution

The authors declare their respective contributions to this manuscript as follows: **Selvia Aprilyanti** conceptualized and designed the study framework. **Irnanda Pratiwi**, as the principal grant recipient, was responsible for material preparation, data analysis, and drafting the primary manuscript. **Winy Andalia** contributed to data acquisition and performed analytical procedures. **Tine Aprianti** critically reviewed the manuscript and provided substantive revisions. **Hariman Al Faritzie** participated in data collection, conducted analytical assessments, and assisted in the initial drafting of the manuscript. All authors have examined the final data interpretations and collectively approved the definitive version of the manuscript for publication.

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Data Availability

The primary dataset generated and analyzed during the current study is accessible upon request. Researchers interested in utilizing the data for subsequent investigations are encouraged to contact the corresponding author for further information.

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Competing interest

The authors declare that they have no competing financial or non-financial interests that could have appeared to influence the work reported in this manuscript.

Ethical clearance

This research does not involve humans as subjects.

AI statement

This scholarly work constitutes the authors' original research, with all textual and graphical elements produced independently and without recourse to artificial intelligence technologies.

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Nomenclature

°C	: Degree Celcius
ANOVA	: Analysis of Variance
DF	: Degree of Freedom
FTIR	: Fourier Transform Infrared
g	: gram
g/g	: gram per gram
MS	: Mean Square
ml	: mililiter
NaOH	: Sodium Hydroxide
Na ₂ SO ₃	: Sodium sulfite
SEM	: Scanning Electron Microscope
SNR	: Signal-to-noise ratio (dB)
SS	: Sums of Squares
TS	: Thickness Swelling
w/w	: Weight per Weight
wt%	: Percent Weight
μ_i	: mean value of the <i>i</i> -th experiment
y_i	: response value
<i>n</i>	: number of observations
<i>Y</i>	: Cellulose yield (%)
<i>SR</i>	: Swelling ratio (g/g)
$W_{(s)}$: Weight of swollen hydrogel (g)
$W_{(d)}$: Weight of dry hydrogel (g)