

Optimization of Phosphate Removal from Simulated Wastewater Using Corn (*Zea mays*) Husk-Derived Cellulose as a Bioadsorbent Using Full Factorial Design of Experiments

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Abstract—Bioadsorbents are extensively utilized in wastewater treatment due to their eco-friendly nature and effectiveness in removing contaminants. The properties of bioadsorbents can undergo significant alterations depending on preparation methods, raw material types, and chemical modifications. This study investigates the efficiency of Corn Husk-Derived Cellulose Bioadsorbent (CHDC-BA) in the removal of phosphate from simulated wastewater. Corn husks were treated with alkali and bleaching processes to obtain CHDC-BA. Batch adsorption experiments were conducted to examine the effects of pH, adsorbent dosage, and contact time on the Percent Phosphate Removal (PPR) efficiency. The experimental approach employed a Full Factorial Design of Experiments (DOE), and the data was processed using JMP® (SAS Institute). The data gathered were analyzed using p-value hypothesis testing, Summary of Fit data, and Prediction Profiler of JMP. Optimal conditions for maximum PPR were found at pH 13, 6 g adsorbent dosage, and 60 min contact time. Under these conditions, CHDC-BA achieved a 60.88% removal efficiency, reducing the phosphate concentration in the simulated wastewater from 9.8 ppm to 3.83 ppm.

Keywords—*Zea mays*, corn husk, cellulose bioadsorbent, full factorial DOE, phosphate removal, wastewater

I. INTRODUCTION

Wastewater consists of the community's water supply after it has been utilized for various purposes [1]. It can also originate from agricultural activities, surface runoff, transport and fuel depots, or rainwater. Its composition is highly variable and constantly changing, depending on the source from which it is released. Water scarcity is a pressing issue that is exacerbated by the persistent rise in untreated wastewater from agricultural, industrial, and domestic sources, alongside rapid urbanization, particularly in developing countries. With this, the demand for clean water has surged dramatically, even as the availability of freshwater resources has become increasingly constrained [2]. Elevated phosphate concentrations can cause eutrophication that stimulates harmful algal blooms and reduces dissolved oxygen levels, ultimately leading to the degradation of aquatic ecosystems [3].

In accordance with the United Nations Sustainable Development Goal (UN SDG) 6 – Clean Water and Sanitation, this study focuses on improving water quality by addressing the pervasive issue of wastewater contamination. The goal emphasizes the necessity of providing clean water and proper sanitation for everyone, which involves

implementing sustainable water management strategies [4]. By investigating the use of corn husk-derived cellulose bioadsorbent for phosphate removal, this research aims to offer a cost-effective and eco-friendly solution for treating wastewater. This approach not only addresses the challenges posed by agricultural and industrial water pollution but also contributes to the global mission of achieving sustainable water practices, thereby supporting the overarching aims of SDG 6.

II. LITERATURE REVIEW

A. Adsorption Process

Adsorption involves transferring a dissolved solid in a liquid onto the surface of another solid, which should have a minimum volume and maximum surface area [5]. Adsorption is considered one of the best and most practical technologies for removing pollutants from wastewater due to its high adsorption capacity, reduced sludge creation, low cost, and reusability. However, most synthetic adsorbents lack cost-effectiveness in their preparation [6].

B. Cellulose-Based Bioadsorbents

Cellulose is a prominent example of a natural bio-based material utilized in water treatment. Numerous review articles have highlighted the significance of cellulose-based adsorbents in addressing water pollution. However, most of these reviews tend to focus specifically on either the adsorbates or the adsorbents rather than providing a comprehensive overview [7]. Cellulose is a tough, fibrous, water-insoluble polysaccharide generally synthesized by plants and some bacteria [8]. Cellulose-based biomass materials possess a unique structure and offer numerous advantages, including environmental friendliness, biodegradability, and renewability. These materials also provide cost benefits due to their low energy requirements, making them an attractive option for various applications [9].

A study by [10] utilized a bioadsorbent derived from corn stalk cellulose to remove Congo red from wastewater. The adsorption mechanism revealed that Congo red is absorbed by amino groups through strong hydrogen bonds. This study presents an alternative method for treating corn stalk biowaste, demonstrating highly effective removal of Congo red from both acidic and alkaline wastewater.

Similarly, Kang *et al.* [11] reviewed the use of coffee waste as a bioadsorbent for removing pollutants such as heavy

metals and dyes from wastewater. Treatments of the coffee husk waste included acid pre-treatment, which hydrolyzes hemicellulose and lignin to increase surface area and porosity, and alkali pre-treatment, which creates more functional groups on the adsorbent surface and increases surface area and pore volume.

In addition, He *et al.* [12] stated that cellulose, being one of the significant components of the lignocellulosic biomass, is one of the most abundant biomasses in the world, and thus using them as bio-adsorbents not only contributes to proper recycling but also a way to reduce the environmental burdens in trash handling. In their study, they used a cellulose-based adsorbent produced from the processing waste of brewer's spent grain for efficient removal of Mn and Pb from contaminated water, which obtained a high maximum adsorption capacity of 272.5 mg/g and 52.9mg/g in Pb and Mn, respectively.

C. Corn (*Zea mays*) Husk

Corn (*Zea mays*), also known as maize, is a starchy vegetable surviving perfectly in nutrient-rich, well-drained soil [13]. A significant portion of corn processing, intended for human consumption, results in by-products such as cobs, husk leaves, discarded kernels, and portions of the stalks [14]. Given the extensive production of corn and its numerous applications, utilizing corn by-products for bioadsorbent production is economically viable and cost-effective. Corn husks offer a valuable and practical resource for environmental management, reducing the reliance on synthetic alternatives. Untreated corn husk consists of 29.3% cellulose, 39.7% hemicellulose, and 11.4% lignin [15]. In their assessment of several studies on the use of biochar made from rice husks for nutrient removal, [16] noted the high removal efficiencies (50–80%) of nitrogen and phosphate. Our research, on the other hand, looks at cellulose made from corn husks as a bioadsorbent. Although they both make use of agricultural waste, their adsorption mechanisms and production methods are very different. Cellulose-based adsorbents are made using lower-energy techniques than biochar, which needs high-energy processing. Verifying the viability of cellulose-based adsorbents for the same purpose is necessary because the results of the examined biochar experiments demonstrate the potential of materials derived from agricultural waste.

D. Novelty of Study

This study uniquely optimizes phosphate removal from simulated wastewater by using a low-energy, sustainable bioadsorbent derived from corn husk cellulose, employing a Full Factorial Design of Experiments (DOE) to systematically evaluate and optimize key operational parameters such as pH, adsorbent dosage, and contact time.

E. Limitation

The study was limited to evaluating the adsorption capacity of CHDC-BA without exploring its regeneration potential using mild chemical desorption techniques, such as treatment with diluted NaOH or HCl solutions. These approaches, as suggested by studies on similar adsorbent-adsorbate systems of Aktar *et al.* [17] and Olivito *et al.* [18], could enhance the bioadsorbent's lifespan and improve the economic feasibility of the process. Furthermore, the long-term fate of the

bioadsorbent after multiple regeneration cycles and the recovery and reuse of adsorbed phosphate were not investigated, leaving gaps in understanding the sustainability and scalability of this treatment method.

III. MATERIALS AND METHODS

A. Preparation of the Raw Material (Corn Husk)

Corn husks were collected from roadside corn vendors in Brgy. Santo Domingo, Sta. Rosa City, Laguna, Philippines. The collected husks were thoroughly washed with distilled water and then dried in an oven at 200°C until a constant mass was achieved, ensuring minimal moisture content. The dried husks were ground using a standard blender and further processed in a miller to obtain a powdered form. The resulting powder was screened using a 40-mesh sieve and stored at room temperature.

B. Extraction of the Cellulose from the Corn Husks

The researchers adapted and modified the corn husk cellulose preparation methods outlined in Kampeerapappun [15]. The powdered corn husks were treated with a 4 wt% sodium hydroxide solution at 80°C for 2 h. Theoretically, this treatment causes the hydrogen bonds between hydroxyl groups in the cellulose to break, leading to the defibrillation of the fibers. According to Gassan *et al.* [19], alkali treatment removes hemicelluloses, splits fibers into fibrils, and allows for closer packing of cellulose chains due to the release of internal strain, thereby enhancing the mechanical properties of the fibers. Defibrillation refers to the breaking down of fiber bundles into smaller fibers [20]. After the treatment, the solid was filtered, washed several times with distilled water, and neutralized using 1M HCl solution until a pH of 7 was achieved. This alkali treatment was applied twice. Following the alkali treatment, a bleaching process was conducted to remove remaining phenolic compounds, such as lignin and hemicellulose, ensuring the isolation of cellulose. The bleaching involved treating the fibers with a 1.7 %w/v sodium hypochlorite solution at 80°C for 4 hours. The mixture was then allowed to cool, filtered with distilled water, and neutralized using 1M NaOH solution until a pH of 7 was achieved. This bleaching process was also applied twice. Finally, the treated materials were dried in a laboratory oven at 100°C until a constant mass was reached. The resulting material is designated as Corn Husk-Derived Cellulose Bioadsorbent (CHDC-BA).

C. Preparation of Simulated Wastewater

The wastewater solution was prepared by dissolving 200 mg of Potassium dihydrogen phosphate (KH_2PO_4) in 1000 mL of distilled water, resulting in a stock solution with a concentration of 200 ppm. This stock solution was freshly prepared just before the adsorption experiments as it lacked preservatives. A 10 ppm KH_2PO_4 solution was diluted from the stock for subsequent usage.

D. Batch Bioadsorption Experiments

The bioadsorption experiment were conducted using a 2^{k+1} Full Factorial DOE to determine the PPR for this study. The 'k' represents the number of parameters or factors involved in the study, which in this case was three. Table 1 presents the low, center, and high values for the factors investigated: pH, adsorbent dosage, and contact time. In each

trial, a 100 mL aliquot of 10 ppm KH_2PO_4 simulated wastewater was used. The initial and final phosphate concentrations were measured using a Phosphate Low-Range Checker® HC-HI713. pH was assessed with a digital pH meter, and adjustments were made by adding drops of 1 M hydrochloric acid (HCl) or 1 M sodium hydroxide (NaOH). After adjusting the pH, the specified amount of CHDC-BA was added. A magnetic stirrer ensured thorough mixing in the beaker during the adsorption process, which was conducted at an agitation speed of 300 rpm for the specified time for each condition. The solution was filtered to separate the used biosorbent and the solution. The PPR can be computed using Eq. (1).

$$\%P \text{ removal} = \frac{(C_0 - C)}{C} \times 100 \quad (1)$$

where C_0 is the initial concentration of the phosphate ions, and C is the final concentration of the solution in mg/L.

Table 1. 2 Levels of the factors considered for the Full Factorial DOE

Level	pH	Adsorbent Dosage (g)	Contact Time (min)
Low (-)	7	2	20
High (+)	13	6	60

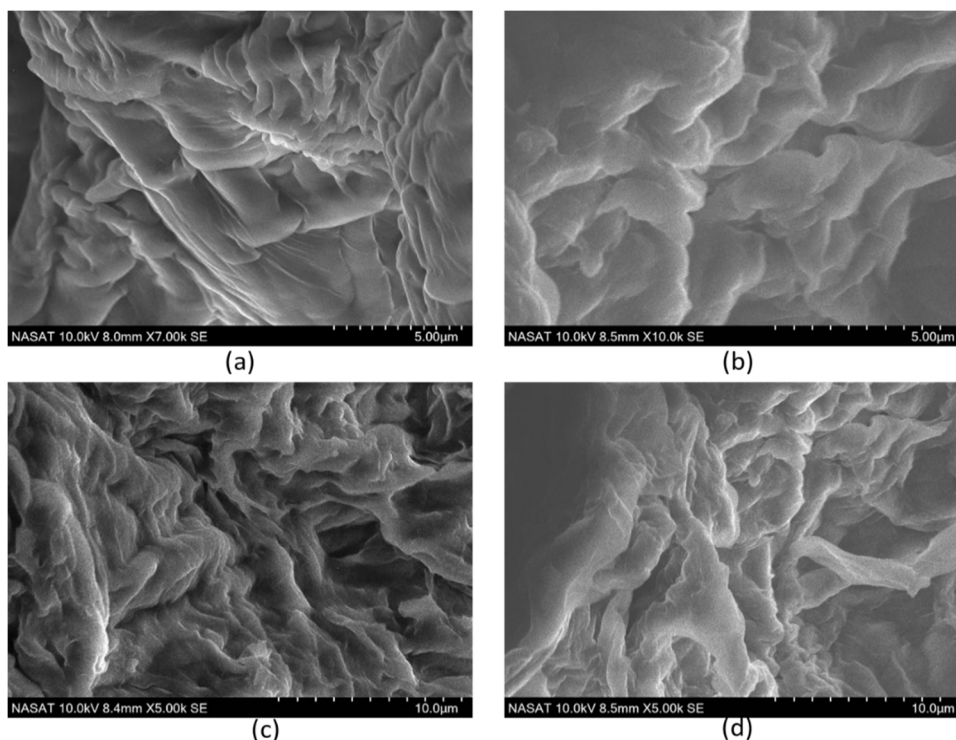


Fig. 1. SEM micrographs of CDHC-BA (a) before batch adsorption at 5 μm magnification, (b) before batch adsorption at 5 μm magnification, (C) after batch adsorption at 10 μm magnification, and (d) after batch adsorption at 15 μm magnification.

B. Bioadsorption Experiment Results

Table 2 summarizes the parameter combinations for each run and the corresponding PPR calculated using Eq. (1). The data were analyzed in JMP® software to identify the optimal parameter combination for maximum phosphate removal using the software's prediction profiler. The following observations highlight the individual and combined effects of each parameter. A study by Waweru *et al.* [21] observed that the PPR of their synthesized chitosan-cellulose acetate beads increased from 42% to approximately 75% as the pH was adjusted from 2 to 11, with maximum removal occurring near neutral pH. Their approach utilized a combination of two

E. Bioadsorbent Characterization

Scanning Electron Microscopy (SEM) – Energy Dispersive X-ray Spectroscopy (EDS) has become a central tool for material characterization, offering detailed insights into the microstructural features and surface morphology of materials. To effectively characterize the CHDC-BA, SEM-EDS was done before and after adsorption, with the latter done in association with the optimum PPR. The SEM-EDS services were from Nanotech Analytical Services and Training Corp (NASAT Labs).

IV. RESULTS AND DISCUSSION

A. SEM-EDS Results

The SEM-EDS micrographs in Fig. 1 illustrate the surface morphology of the CHDC-BA used in the adsorption experiments. While differences in contrast and brightness between the pre-and post-adsorption images may indicate changes in surface composition and morphology, the microstructural alterations or the presence of adsorbed phosphate may not be distinctly visible in the SEM images despite successful adsorption.

adsorbents, with the latter being a modified cellulose material. Notably, in our study, higher pH levels tend to result in higher PPR, except for Leg 6. For instance, when comparing similar adsorbent dosages and contact times, increasing the pH from 7 to 13 shows a significant increase in PPR. Specifically, at pH 13, the average PPR is consistently higher across all trials (Leg 7 through 9). This is in contrast with the findings of Panaligan *et al.* [22], which utilized algae-based adsorbents, which decreased PPR at higher pH levels. However, their adsorbent was mainly in low amounts considering our study. Similarly, our results differ from Manyatshe *et al.* [23] where PPR decreased inversely with pH. In their research, high removal was observed at pH 3 to 5, which they attributed to

the prevalence of H_2PO_4^- and HPO_4^{2-} species in this region, making these species more amenable to adsorption under acidic conditions.

An increase in adsorbent dosage generally leads to higher PPR for our study. For instance, at pH 13 and 20 min contact time, increasing the adsorbent dosage from 2g to 6g resulted in a significant increase in PPR (from 24.83% to 36.39% on average). However, the impact of adsorbent dosage is more pronounced at higher pH and longer contact times. This is consistent with findings from [22], which suggest that phosphate removal efficiency increases with adsorbent dosage, particularly for modified corn stalks.

Longer contact times tend to increase the PPR, which aligns with the research by [22], which observed similar trends where longer contact times resulted in better phosphate removal, though the adsorbent dosage used was relatively low. For example, at pH 13 and 2g adsorbent dosage, increasing the contact time from 20 to 60 min increased the PPR from 24.83% to 46.60% on average. The trend of increased removal efficiency with longer contact times is observed across different pH levels and adsorbent dosages. The combination of higher pH, higher adsorbent dosage, and longer contact time results in the highest PPR. The maximum average PPR of 60.88% was observed at Leg 9. Conversely, the lowest average PPR was observed at Leg 1, indicating that all three parameters play a crucial role in maximizing removal efficiency. The center point (Leg 5) also shows a relatively high average PPR of 32.65%, suggesting that even moderate settings can achieve substantial removal efficiency.

C. Statistical Treatment

Log worth is a transformed representation of the significance of effects in a factorial design derived by applying a logarithmic transformation to the p-value associated with each effect. This technique helps to visualize significance levels more clearly, especially when multiple factors are considered simultaneously. By rescaling experimental observations, log transformation improves the fit with the assumptions of specific statistical analyses [24]. Larger log-worth values indicate more significant effects, while smaller values represent less significant ones, with the p-value serving as the threshold for rejecting the null hypothesis and indicating statistical significance.

Loftus *et al.* [25] mentioned that different fields may have varying standards for acceptable α levels, but the most accepted significance level is $\alpha = 0.05$. A p-value below α signifies the statistical significance of both the main and interaction effects of variables. When a factor demonstrates a p-value equal to or less than α , it indicates significance, meaning there is less than a 5% probability that the observed results are due to random chance.

In the context of the study, this implies that the variables examined—including both their direct effects and possible interactions—are likely to have a significant impact rather than being merely the result of random variation. Therefore, it supports rejecting the null hypothesis and endorses the alternate hypothesis, underscoring the significance of the observed effects.

Table 2. DOE Matrix and %P removal result

Leg	Pattern	pH	Adsorbent Dosage (g)	Contact Time (min)	% P Removal			
					Trial 1	Trial 2	Trial 3	Average
1	---	7	2	20	23.47	20.41	25.51	23.13
2	--+	7	2	60	25.51	31.63	28.57	28.57
3	-+-	7	6	20	21.43	26.53	25.51	24.49
4	+++	7	6	60	32.65	35.71	36.73	35.03
5	000	10	4	40	27.55	33.67	36.73	32.65
6	+-	13	2	20	22.45	25.51	26.53	24.83
7	++	13	2	60	45.92	46.94	46.94	46.60
8	++-	13	6	20	33.67	37.76	37.76	36.39
9	+++	13	6	60	63.27	61.22	58.16	60.88

The analysis of the Log Worth and p-value, as summarized in Table 3, indicates that all the parameters and their interactions have significant effects on PPR. The Log Worth values, which range from 3.179 to 27.582, demonstrate high statistical significance. Specifically, contact time (log worth: 27.582) and pH (log worth: 26.925) are the most significant individual factors, followed by Adsorbent Dosage with a Log Worth of 22.527.

Additionally, the interaction effects between pH and contact time (Log Worth: 21.718), pH and adsorbent dosage (Log Worth: 17.387), and adsorbent dosage and contact time (Log Worth: 10.819) are all significant. The three-way interaction between pH, adsorbent dosage, and contact time is also substantial, with a Log Worth of 3.179 and a p-value of 0.00066.

Through the summary of the fit test, the R-squared value of the study is at 0.999398, which indicates that approximately 99.94% of the variability in PPR is explained

by the model, demonstrating an exceptionally close match between the observed and predicted data. The adjusted R-squared is 0.999176 and confirms that this strong association persists even after accounting for the number of predictors.

The Root Mean Square Error (RMSE) of 0.338435 further supports this, as it reflects a minimal average difference between observed and predicted values. According to Curran-Everett [24], the smaller the value of the RMSE indicates the accuracy of the predicted values. Additionally, RMSE values of between 0.2 and 0.5 show that the model can relatively predict a data set accurately. In Fig. 2, the data demonstrate that PPR reaches its maximum of 60.66% under conditions of pH 13, an adsorbent dosage of 6 grams, and a 60-min agitation time. These optimal conditions were precisely identified by the JMP desirability function, which predicted a phosphate removal efficiency of 60.88% and a desirability of 88.82%.

Table 3. Log Worth and p-Value results for JMP®

Parameter	Log Worth	p-Value
Contact Time	27.582	0.00000
pH	26.925	0.00000
Adsorbent Dosage	22.527	0.00000
pH * Contact Time	21.718	0.00000
pH * Adsorbent Dosage	17.387	0.00000
Adsorbent Dosage * Contact Time	10.819	0.00000
pH * Adsorbent Dosage * Contact Time	3.179	0.00066

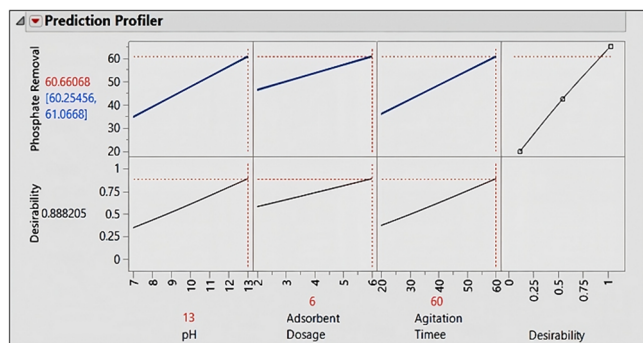


Fig. 2. JMP® Prediction Profiler Outcome.

The closeness between the predicted and actual values is evident, with a minimal percent difference (%diff) of 0.36%. The %diff quantitatively assesses the disparity or similarity between two numerical values. A small %diff value indicates an exceedingly minimal variation between the numbers, suggesting that the two quantities are nearly equivalent relative to their average.

V. CONCLUSION

This study emphasizes the significant potential of utilizing CHDC-BA for the effective removal of phosphate from wastewater. The comprehensive experimental approach, employing a Full Factorial DOE, revealed that factors such as pH, adsorbent dosage, and contact time, along with their interactions, significantly influence the phosphate removal efficiency. The research findings demonstrate that higher pH levels, increased adsorbent dosage, and prolonged contact times enhance phosphate adsorption, with the optimal conditions being pH 13, 6 g of adsorbent dosage, and 60 min of agitation time. The maximum phosphate removal efficiency achieved was 60.88%, closely aligning with the predicted value of 60.66% derived from data analysis. This suggests that 60.66% may be completed upon repeatability trials, demonstrating the robustness and reliability of the experimental design and the predictive model in its entirety. The use of corn husk as a bioadsorbent not only provides a cost-effective and sustainable alternative to synthetic adsorbents but also adds value to agricultural waste, promoting environmental sustainability. The SEM-EDS analyses confirmed the structural suitability of CHDC-BA for adsorption applications.

However, the adsorption of phosphate was not distinctly visible in the SEM micrographs. This suggests that other analytical methods may be necessary to better understand the adsorption process in future studies. Several additional analytical techniques could enhance the understanding of phosphate adsorption on CHDC-BA. Fourier Transform

Infrared Spectroscopy (FTIR) is highly recommended as it can identify functional groups and chemical bonds in the bioadsorbent, revealing an understanding of the interactions between phosphate ions and the material. Nuclear Magnetic Resonance (NMR) Spectroscopy is also valuable for investigating the molecular environment and interactions of phosphate within the bioadsorbent matrix.

CONFLICT OF INTEREST

The authors declare no conflict of interest.

AUTHOR CONTRIBUTIONS

DJB. Alpino, JB. Aquino, and AJA. Carta was instrumental throughout the research process, contributing significantly to material procurement, laboratory work, and statistical analysis, particularly with the statistical software. TRL. Panaligan and JR. Aquino offered essential guidance and direction to the team throughout the project.

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