

IRSTI 61.74.99

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<https://doi.org/10.55956/OHWP5394>

OPTIMIZATION OF BIOCHAR PRODUCTION FROM AGRO-INDUSTRIAL WASTE OF THE ZHAMBYL REGION USING RESPONSE SURFACE METHODOLOGY FOR APPLICATION IN PERMEABLE REACTIVE BARRIERS

Abstract. The escalating contamination of water resources by heavy metals and organic pollutants poses a significant environmental challenge, particularly in regions like Zhambyl (Kazakhstan), where water quality varies from moderately to extremely polluted. This study focuses on optimizing biochar production from agro-industrial waste, specifically rice husk, from the Zhambyl region, employing Response Surface Methodology (RSM) to enhance its applicability in Permeable Reactive Barriers (PRBs) for groundwater remediation. Through the Box-Behnken design, key pyrolysis parameters – temperature, activation time, and the ratio of rice husk to phosphoric acid (H_3PO_4) – were systematically varied to maximize biochar yield and adsorption capacity. Statistical analysis using ANOVA validated the quadratic model's significance ($p < 0.05$, $R^2 = 0.9662$), identifying optimal conditions at approximately 2.2 g H_3PO_4 and 540°C, yielding up to 84% biochar. These findings underscore the potential of optimized biochar as a sustainable, cost-effective material for environmental remediation, particularly in PRB systems.

Keywords: biochar, agro-industrial waste, response surface methodology (RSM), permeable reactive barriers (PRB), pyrolysis, surface morphology.



Kanatbek U.G., Karimbaeva Zh.E., Zhienbaeva A.S., Reimbayev Y.K., Kurtebayeva A.A., Jumbri Kh., Orynbayev S.A., Kalmakhanova M.S. Optimization of biochar production from agro-industrial waste of the Zhambyl region using Response Surface Methodology for application in permeable reactive barriers //Mechanics and Technology / Scientific journal. – 2025. – No.3(89). – P.167-180. <https://doi.org/10.55956/OHWP5394>

Introduction. The contamination of wastewater by heavy metals, organic compounds, and petroleum products has become a global environmental threat. The sources of heavy metal pollution in wastewater can be both natural – such as

geochemical, volcanic, and hydrothermal processes – and anthropogenic, including industrial activities, agriculture, domestic waste, and landfills [1].

The Zhambyl Region of Kazakhstan is also facing significant challenges related to the pollution of its water resources. According to recent studies, water quality indicators in the region range from moderately polluted to extremely high levels of contamination. For instance, the pollution level of the Bilikol River is classified as “extremely high,” while the Talas and Shu rivers are considered to have a “moderate level of pollution” [2].

Biochar is a carbon-rich, black solid material produced through a thermochemical process under limited oxygen conditions. It is typically synthesized from organic waste materials, particularly biomass, due to its availability and environmental sustainability. Biomass feedstock sources often include agricultural residues and forestry by-products. The conversion of such biomass into biochar is considered a valorization pathway that not only mitigates waste accumulation but also produces a functional carbonaceous material. Biochar has been successfully applied in diverse areas such as bioenergy production, wastewater treatment, and soil remediation, providing a sustainable alternative to conventional fossil-based carbon sources that are known to contribute to environmental degradation. These advantages support the ongoing interest in optimizing biochar synthesis from agro-industrial waste, especially for environmental remediation purposes [3].

In recent years, increasing attention has been directed toward the sustainable conversion of agricultural and agro-industrial wastes into value-added products. One such promising product is biochar, a carbon-rich solid produced through the thermal decomposition of biomass under limited oxygen conditions – a process known as pyrolysis. Pyrolysis is considered a cost-effective and energy-efficient method that contributes to environmental sustainability and offers multiple applications in soil improvement, water treatment, and pollution control [3,4].

Among the modern environmental technologies, Permeable Reactive Barriers (PRBs) have emerged as a passive, in-situ remediation system that effectively treats contaminated groundwater. These systems rely on various reactive materials – including biochar – to remove pollutants through mechanisms such as adsorption, ion exchange, reduction, and precipitation [5-7]. Biochar has become a material of interest for PRBs due to its large surface area, high porosity, and rich surface chemistry, which include functional groups such as hydroxyl, carboxyl, and aromatic structures [8].

Response Surface Methodology (RSM) is a powerful statistical tool used for modeling and optimizing processes influenced by multiple variables. It enables the development of predictive models by evaluating the relationship between input parameters and output responses such as biochar yield or adsorption capacity [9]. Design-Expert software facilitates this process with tools like power calculations, center points, and model validation features.

Several studies have demonstrated that temperature and heating rate are the most significant factors affecting biochar yield during pyrolysis, whereas particle size and gas flow rate have a lesser effect. For instance, RSM-based experiments have shown that increasing temperature positively correlates with fixed carbon content and yield, supporting the use of predictive modeling for optimization [10]. In one such study, Design-Expert 13 software was used with a Box-Behnken Design (BBD) to assess the influence of temperature, heating rate, and feedstock carbon content, with ANOVA confirming their statistical significance.

Recent work using cassava peels as feedstock further confirmed that temperature, heating rate, and reaction time are critical for both yield and quality, reinforcing the importance of RSM in scaling up sustainable biochar production [11].

Biochar from agro-industrial waste has great potential for environmental uses such as groundwater treatment in permeable reactive barriers (PRBs). Optimizing pyrolysis conditions is important to improve biochar quality and yield. This study aims to optimize biochar production from Zhambyl region waste using Response Surface Methodology (RSM) to enhance its use in PRBs.

Materials and methods. *Reagents and materials.* Rice husk, an agricultural waste material, was collected from the Shymkent region, Kazakhstan, and used as the carbon-containing raw material. Orthophosphoric acid (H_3PO_4 , 3 mol/L) served as the activating agent. Ultrapure (UP) water with a pH of 5.85 was used throughout the experiments. The pH of solutions was measured using a pH meter (model pH-009(1)A).

The physicochemical properties and functional groups of the activated samples were determined using FTIR (Fourier Transform Infrared) spectroscopy. FTIR spectra were recorded with an Infraspec spectrometer (model FSM 2202) at a resolution of 1 cm^{-1} and a scanning range of $5000\text{--}500\text{ cm}^{-1}$.

To determine the concentration of Ni in the solution after adsorption, an Agilent 4200 MP-AES microwave plasma atomic emission spectrometer equipped with an Agilent 4107 nitrogen generator was used. The sample introduction system included a cyclonic double-pass spray chamber, a OneNeb nebulizer, and a Solvaflex pump tubing. The analysis was performed using multicomponent calibration standards prepared in a 5% HNO_3 / 0.2% HF (v/v) medium.

To design the experiments and optimize the biochar activation process, Response Surface Methodology (RSM) was applied using Design-Expert software. The experimental plan was developed based on the Box-Behnken method, which included 17 runs. The considered factors were pyrolysis temperature, processing time, and the ratio of raw material to phosphoric acid (H_3PO_4). The responses were biochar yield and adsorption capacity. The results were expressed in percentages and used to build models for determining the optimal process conditions.

Synthesis of biochar. To produce biochar from agro-industrial waste of the Zhambyl region, rice husk was selected as the raw material. The rice husk was first washed with running water to remove impurities, then dried under direct sunlight.

The resulting samples were washed with distilled water to reach a stable pH value of the filtrate. The pH level was measured using a pH meter and maintained within the acceptable range of 5.80-6.00.



Fig. 1. Biochar after pyrolysis

Figure 1. showed about synthesis of biochar involved mixing the pre-washed husk with a phosphoric acid (H_3PO_4) solution at a ratio of 1:1.5 (g/g) and a concentration of 3 M. The mixture was left overnight to allow activation. Afterward, pyrolysis was carried out by thermally treating the material in a muffle furnace using a porcelain crucible.

Subsequently, the samples were dried in a vacuum drying chamber. As a result of these processes, two activated biochar samples with specific characteristics were obtained: as biochar derived from rice husk at BCHR 600°C and BCHR 550°C samples.

Research results and discussion. *Analysis of infrared (IR) spectra of samples.* As a result of the described processes, two samples of activated biochars with specified characteristics were obtained: As biochar derived from rice husk at BCHR 600°C and BCHR 550°C samples.

Fourier Transform Infrared (FTIR) spectroscopy and Scanning Electron Microscopy (SEM) confirmed the presence of functional groups (e.g., Si—O—Si, C=O, C=C) and a microporous structure, enhancing biochar's suitability for wastewater treatment.

The physicochemical properties and functional groups of the activated samples were determined using Fourier Transform Infrared (FT-IR) spectroscopy. The FT-IR spectra were recorded using an Infraspec FT-IR spectrometer (model FSM 2202) with a resolution of 1 cm^{-1} and a scanning range of 5000–500 cm^{-1} .

To thoroughly investigate the functional groups and their influence on the sorption characteristics, an analysis of the biochar FT-IR spectra was performed. Figures 1 and 2 present the spectra of biochar obtained from rice husk at temperatures of 600°C and 550°C, respectively.

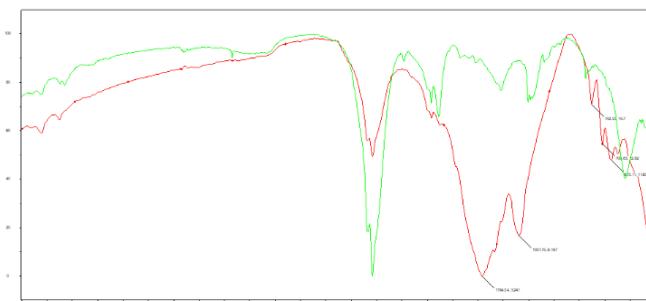


Fig. 2. FT-IR spectra of BCHR 600°C

FT-IR spectra analysis of BCHR 600°C in the range of 675–709 cm^{-1} (Fig. 2.), the observed vibrations correspond to the deformations of Si—O—Si bonds, which indicate the presence of silica in the composition of rice husk ash. In the range of 679–710 cm^{-1} , the vibrations are caused by the presence of C—H bonds in aromatic structures. In the range of 710–752 cm^{-1} , the deformational vibrations of C—H bonds are characteristic of aromatic hydrocarbons. In the range of 752–1034 cm^{-1} , intense absorption is associated with the valence vibrations of Si—O bonds in amorphous silicon dioxide, which is the primary component of rice husk after carbonization. In the range of 1037–1134 cm^{-1} , the vibrations correspond to C—O bonds, which are typical for complex esters, cellulose residues, or carbonized carbohydrates. In the range of 1385–1504 cm^{-1} , the vibrations of C=C bonds in aromatic systems suggest the presence of graphite-like structures in the biochar. In

the range of 1505–1616 cm⁻¹, the vibrations of C=O and C=C bonds are attributed to the presence of aromatic and carbonyl compounds.

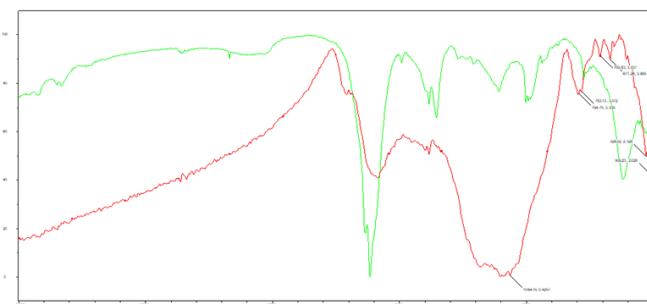


Fig. 3. FT-IR spectra of BCHR 550°C

According to Figure 3, the FT-IR spectrum of BCHR at 550 °C is shown. In all biochar samples, characteristic peaks are observed in the range of 1000–1100 cm⁻¹, corresponding to the asymmetric vibrations of Si–O–Si bonds, as well as a signal at 788 cm⁻¹ associated with Si–H groups. These findings confirm the presence of silica, which is resistant to high temperatures and persists after the carbonization of rice husk [12]. The results of the EDS spectra further validate its presence. Additionally, the region of 1030–1110 cm⁻¹ may be attributed to the vibrations of C–O–C groups present in the residues of cellulose and hemicellulose [12,13].

Following chemical activation, the structure of the biochar undergoes significant changes: bands appear in the range of 1450–1600 cm⁻¹, corresponding to aromatic C=C and C=O bonds, indicating the formation of phenolic and carboxyl groups from lignin. Deformational vibrations of aromatic C–H bonds are also observed in the region of 870–880 cm⁻¹.

The preparation process involves raw material pretreatment, pyrolysis under limited oxygen access, followed by washing and drying, resulting in a material suitable for use in reactive barriers.

Morphological analysis (SEM) of BCHR 600°C and BCHR 550°C materials. The SEM analyses of BCHR biochar samples pyrolyzed at 550°C and 600°C reveal progressive structural evolution with increasing temperature. At 550°C, the biochar exhibits a heterogeneous structure with a combination of micro- and macropores, partially preserved biomass anatomy, and disrupted cell wall frameworks. This suggests moderate devolatilization and structural reorganization.

At 600°C, the biochar undergoes more pronounced carbonization, leading to a well-developed porous network with uniformly distributed micropores and larger channels. These features result from intensified devolatilization and breakdown of organic constituents, promoting higher surface roughness and porosity.

Overall, the microstructural differences indicate that higher pyrolysis temperatures enhance pore development and carbonization degree. Both samples demonstrate promising characteristics for environmental applications such as adsorption, catalysis, and soil enhancement, with BCHR 600°C showing superior porosity and surface complexity.

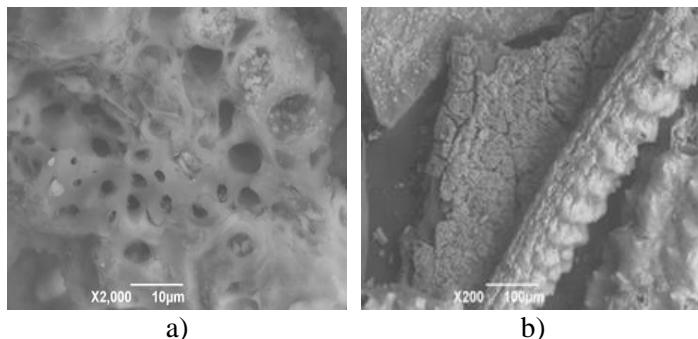


Fig. 4. SEM analys of BCHR 600°C

According to Figure 4, the SEM micrographs (images a and b) of BCHR biochar synthesized at 600°C show notable microstructural changes compared to lower pyrolysis temperatures. The surface morphology indicates a well-developed porous structure with varying pore sizes and visible surface fractures [14,15].

In image (a) (scale bar 10 μm), the surface reveals abundant spherical and oval-shaped micropores, several micrometers in diameter, which are distributed across the matrix. These pores suggest intense devolatilization and thermal degradation of volatile organic compounds, leading to the release of gases and formation of voids. The uniform distribution of such pores enhances the surface area and potential adsorption capacity of the biochar.

Image (b) (scale bar 100 μm) provides a broader view of the surface, showing interconnected channels and larger cracks. These may have originated from the collapse of biomass cell walls due to severe carbonization. The rough and fractured texture, combined with both micro- and macropores, indicates a transition from the original biomass structure to a highly carbonized, porous framework [14,15].

These structural features contribute significantly to the functional performance of the biochar, making BCHR 600°C a promising candidate for applications in water purification, gas adsorption, or as a soil amendment.

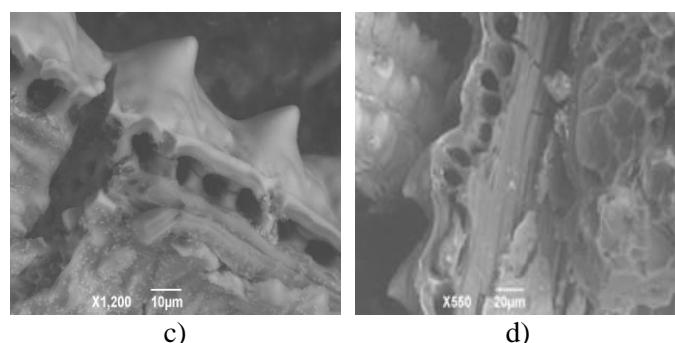


Fig. 5. SEM analys of BCHR 550°C

According to Figure 5, the SEM micrographs (images c and d) of BCHR at 550°C illustrate the structural and morphological transformations that take place during pyrolysis. The images reveal a heterogeneous microstructure characterized by a combination of compact and porous regions.

In image (c), taken at higher magnification (scale bar 10 μm), the surface shows well-defined porous channels and disrupted cell wall structures, indicating the degradation of the original biomass framework. The presence of microvoids and collapsed pore walls suggests significant thermal decomposition, likely driven by the breakdown of hemicellulose and cellulose components at this temperature.

Image (d), captured at lower magnification (scale bar 20 μm), demonstrates the overall structural integrity of the biochar matrix. It shows irregularly shaped particles with interconnected macropores and partially preserved anatomical features of the precursor material. These macropores are crucial for enhancing the surface area and adsorption properties of the biochar.

Overall, the observed porous architecture implies that pyrolysis at 550°C leads to a partially preserved but highly porous biochar structure, suitable for applications in adsorption, catalysis, or soil amendment, due to its favorable surface characteristics and porosity.

Elemental analysis of of BCHR 600°C and BCHR 550°C materials. To determine the quality and chemical composition of the biochar obtained from rice husk, an elemental analysis was carried out using a G4 ICARUS HF sulfur and carbon. This method is based on combusting the sample in a high-frequency furnace, followed by detection of carbon and sulfur content using infrared sensors.

The results of the two samples are presented in Table 1.

Table 1

Elemental analysis of of BCHR 600°C and BCHR 550°C materials.

Sample	Carbon content, %	Sulfur content, %
BCHR 600°C	7.3309	0.0027
BCHR 550°C	8.9576	0.0067

According to Table 1, elemental analysis revealed carbon contents ranging from 7.33% to 8.96% and sulfur contents from 0.0027% to 0.0067%, indicating enhanced carbonization at higher pyrolysis temperatures, which improves sorption properties. According to the results, the carbon content in BCHR 600°C is 7.33%, which indicates the beginning of the formation of a stable carbon structure. BCHR 550°C contains 8.96% carbon, which is significantly higher. This suggests more efficient carbonization, possibly due to a higher pyrolysis temperature or longer retention time. The higher the carbon content, the better the sorption and reductive properties of the material.

In BCHR 600°C, the sulfur content is 0.0027%, which can be considered a low level. In BCHR 550°C, the sulfur concentration increases to 0.0067%, which is still relatively low but indicates an increase in residual volatile compounds as a result of differences in processing conditions.

In conclusion, the increase in carbon content from 7.33% to 8.96% along with the simultaneous increase in sulfur from 0.0027% to 0.0067% proves that BCHR 550°C was obtained under more aggressive thermochemical conditions (at higher pyrolysis temperature and retention time). This contributed to deeper carbonization but also led to partial preservation or secondary formation of sulfur compounds.

Optimization of biochar production conditions using Response Surface Methodology (RSM). Biochar yield was selected as the first response variable to assess the efficiency of the production process. Yield is a critical factor because it reflects the amount of biochar produced from raw agro-industrial waste after thermal and chemical treatment. The experiments were designed using the Box-

Benken method in the Design-Expert program, which allowed three independent factors to be systematically varied: pyrolysis temperature, activation time, and rice husk impregnation ratio and phosphoric acid.

The experimental results showed that the yield varied from X% to Y% depending on the conditions. Analysis of variance (ANOVA) confirmed that the quadratic model used was statistically significant ($p < 0.05$) and the R^2 value was Z, indicating a strong correlation between the predicted and experimental results. The 3D surface plots showed that higher temperatures generally reduced the yield due to increased volatilization, while moderate impregnation ratio and activation time made a positive contribution.

Table 2
Experimental matrix and yield data for biochar production from rice husk via
RSM approach

Source	Sequential p-value	Lack of Fit p-value	Adjusted R ²	Predicted R ²	
Linear	0.0021	0.1554	0.6473	0.5294	
2FI	0.2931	0.1620	0.6877	0.5664	
Quadratic	0.0296	0.4165	0.9053	0.5991	Suggested
Cubic	0.4165		0.9285		Aliased

According to Table 2 Model fitting was evaluated through analysis of variance (ANOVA), and the results are presented in Table X. The sequential p-values, lack of fit p-values, adjusted R^2 , and predicted R^2 values were used to assess the suitability of different models (Linear, 2FI, Quadratic, and Cubic) for predicting the biochar yield.

The quadratic model was suggested as the most appropriate based on its statistically significant sequential p-value ($p = 0.0296$), acceptable lack of fit ($p = 0.4165$), and superior adjusted R^2 (0.9053), indicating a good fit to the experimental data. Although the predicted R^2 (0.5991) was slightly lower than the adjusted R^2 , the difference remained within acceptable limits, suggesting adequate predictive performance.

In contrast, the linear and 2FI models, while simpler, showed lower adjusted and predicted R^2 values, and their sequential p-values indicated weaker statistical significance. The cubic model was aliased, indicating overfitting and an insufficient number of degrees of freedom to estimate model coefficients reliably. Therefore, the quadratic model was selected for further analysis and optimization of the biochar production process.

Table 3
Statistical analysis (ANOVA) of the quadratic model for predicting biochar yield

Source	Sum of Squares	df	Mean Square	F-value	p-value	
1	2	3	4	5	6	7
Model	3745.40	9	416.16	15.87	0.0036	significant
A-Mass of H ₃ PO ₄	2483.36	1	2483.36	94.69	0.0002	
B-Temperature	252.45	1	252.45	9.63	0.0268	
C-Time	66.30	1	66.30	2.53	0.1727	
AB	207.65	1	207.65	7.92	0.0374	
AC	72.68	1	72.68	2.77	0.1569	
BC	102.21	1	102.21	3.90	0.1054	
A ²	161.51	1	161.51	6.16	0.0557	

Table 3 (continued)

1	2	3	4	5	6	7
B^2	193.61	1	193.61	7.38	0.0419	
C^2	289.44	1	289.44	11.04	0.0210	
Residual	131.13	5	26.23			
Lack of Fit	91.56	3	30.52	1.54	0.4165	not significant
Pure Error	39.57	2	19.79			
Cor Total	3876.53	14				

Table 3 presents the Model F-value of 15.87 implies the model is significant. There is only a 0.36% chance that an F-value this large could occur due to noise.

p-values less than 0.0500 indicate model terms are significant. In this case A, B, AB, B^2 , C^2 are significant model terms. Values greater than 0.1000 indicate the model terms are not significant. If there are many insignificant model terms (not counting those required to support hierarchy), model reduction may improve your model. The Lack of Fit F-value of 1.54 implies the Lack of Fit is not significant relative to the pure error. There is a 41.65% chance that a Lack of Fit F-value this large could occur due to noise. Non-significant lack of fit is good – it is necessary to fit a model to the research data.

Table 4

Model Fit Statistics for Response Surface Models of Biochar Yield

Std. Dev.	5.12	R^2	0.9662
Mean	64.51	Adjusted R^2	0.9053
C.V. %	7.94	Predicted R^2	0.5991
		Adeq Precision	11.8734

According to Table 4, the Predicted R^2 of 0.5991 is not as close to the Adjusted R^2 of 0.9053 as one might normally expect; i.e. the difference is more than 0.2. This may indicate a large block effect or a possible problem with your model and/or data. Things to consider are model reduction, response transformation, outliers, etc. All empirical models should be tested by doing confirmation runs.

Adeq Precision measures the signal to noise ratio. A ratio greater than 4 is desirable. Your ratio of 11.873 indicates an adequate signal. This model can be used to navigate the design space.

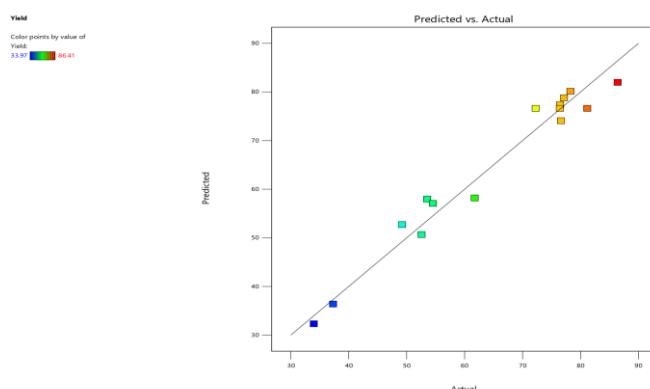


Fig. 8. Predicted vs. actual Response Plot for biochar yield based on the Quadratic Model

As illustrated in Figure 8, a strong correlation exists between experimental and predicted values of biochar yield, indicating the model's reliability. The data points are color-coded based on the yield values, ranging from low yields in blue (~33.97) to high yields in red (~86.41). Most data points lie close to the diagonal line, which represents perfect prediction (i.e., predicted = actual). This indicates a strong agreement between the experimental results and the model predictions. The color gradient (blue to red) also shows a good distribution of predicted responses across the yield range, suggesting that the model is effective across different levels of output. The slight deviations from the line in some points are normal and indicate minor prediction errors, which are acceptable given experimental variation. The plot confirms that the developed model provides a reliable prediction of biochar yield based on the selected parameters (temperature, time, and acid ratio). The high degree of alignment between predicted and actual values supports the accuracy and validity of the model.

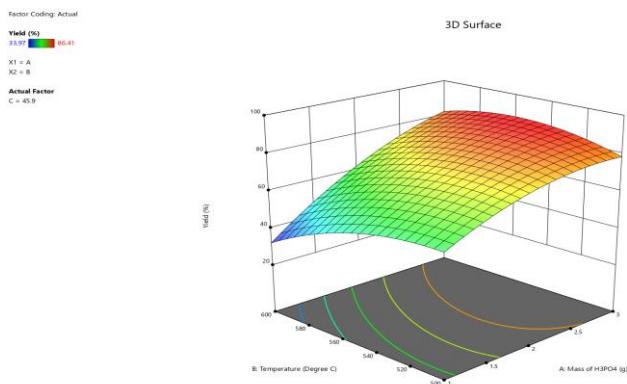


Fig. 9. 3D surface plot of biochar yield as a function of H₃PO₄ mass and pyrolysis temperature

A 3D surface plot in Figure 9 illustrates the combined effect of H₃PO₄ mass (A) and temperature (B) on the biochar yield (%) while keeping the third variable (C) constant at 45.9. The plot shows that increasing both H₃PO₄ mass and temperature generally leads to a higher yield, reaching a peak before slightly decreasing – indicating a non-linear relationship. The curved surface and contour lines at the base indicate significant interaction between the two factors. The maximum yield is observed in the region where both temperature and acid mass are high, but not at their absolute maximum values, which suggests the existence of an optimal point.

A contour plot in Figure 10 shows the effect of H₃PO₄ mass (A) and time (C) on the biochar yield (%), while temperature (B) is held constant at 500°C. As both the acid mass and time increase, the yield also increases, indicated by the shift from blue to orange regions. The highest yield values (close to 86%) are observed in the bottom-right region, where both H₃PO₄ mass and time are high. The concentric contour lines suggest a gradual and consistent improvement in yield with increasing values of both factors. The design points (marked with red dots) show where experiments were conducted.

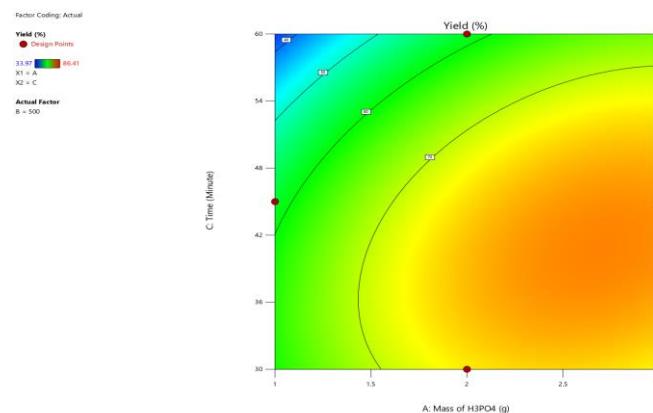


Fig. 10. Contour plot of biochar yield (%) as a function of H_3PO_4 mass and reaction time at constant temperature

Table 5

Optimization parameters including factor levels and experimental settings for maximizing biochar yield

Name	Goal	Lower Limit	Upper Limit	Lower Weight	Upper Weight	Importance
A:Mass of H_3PO_4	is in range	1	3	1	1	3
B:Temperature	is in range	500	600	1	1	3
C:Time	is in range	30	60	1	1	3
Yield	maximize	33.97	86.41	1	1	5

The optimization parameters for the process, including variables A, B, and C – which represent the mass of H_3PO_4 , temperature, and time respectively, each constrained within specified lower and upper limits – are presented in Table 5. All three factors have equal importance (3) and equal weights for their limits. The goal for the yield is to maximize it, with a wider importance value (5), indicating it is the most critical factor. The yield ranges from 33.97 to 86.41, reflecting the efficiency of the process under different conditions.

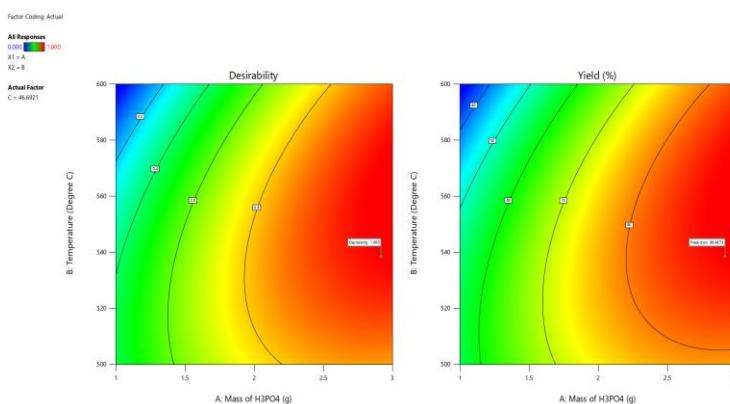


Fig. 11. Point prediction for (a) desirability and (b) yield within optimization range

Figure 11 presents contour plots used to optimize the influence of H_3PO_4 mass (A) and temperature (B) on two key responses: desirability and biochar yield

(%). The left plot depicts desirability, a composite metric ranging from 0 to 1, where higher values represent more favorable conditions. The optimal desirability (~0.7) is achieved near 2.2 g of H_3PO_4 and 540°C. The right plot illustrates biochar yield, with a maximum around 84% observed in the same region. Both plots indicate that increasing either H_3PO_4 mass or temperature beyond these optimal points results in decreased desirability and yield, as shown by the transition to lower-value regions. These findings, obtained through response surface methodology (RSM), highlight that the optimal process parameters to maximize biochar production efficiency and overall desirability are approximately 2.2 g of H_3PO_4 and 540°C.

Conclusion. This study successfully demonstrated the optimization of biochar production from rice husk, an abundant agro-industrial waste in the Zhambyl region, using Response Surface Methodology (RSM) to enhance its efficacy in Permeable Reactive Barriers (PRBs) for groundwater remediation. The application of the Box-Behnken design facilitated the identification of optimal pyrolysis conditions – specifically, a temperature of 540°C and an H_3PO_4 mass of 2.2 g – resulting in a maximum biochar yield of approximately 84% with enhanced adsorption properties. FTIR and SEM analyses confirmed the development of a porous structure and functional groups conducive to pollutant removal, while elemental analysis indicated improved carbonization at higher temperatures, with carbon content reaching 8.96% in BCHR 550°C samples. The quadratic model's statistical significance ($p = 0.0036$, $R^2 = 0.9662$) and adequate precision (11.8734) validated its reliability for predicting biochar yield. These results highlight the potential of rice husk-derived biochar as a sustainable, high-performance material for environmental applications, particularly in addressing groundwater contamination. Future research should focus on scaling up production and evaluating long-term performance in real-world PRB systems to further validate its practical utility.

References

1. Murzagaliyeva M.G., Ashimkhan N.S., Sapiyeva A.O., Tanybayeva A.K., Daribayeva G.T. Research of chemical processes of wastewater treatment // Bulletin of Kazakh University of Technology and Business. – 2024. – P. 166-175.
2. Jetimov M.A., Ybraimzhanova L.K., Bektenev N.A., Kambarova E.A., Mamanova S.A. Wastewater treatment using modified natural sorbents // Bulletin of Kazakh University of Technology and Business. – 2024. – P. 119-129.
3. Ong C.K., Ghazali N.F., Hasbullah H., Ismail A.F., Rahman S.A., Kusworo T.D., Lee C.T. Biomass-based biochar as adsorbent: A mini review of production methods, characterization, and sustainable applications // Chemical Engineering Transactions. – 2024. – Vol. 113. – P. 499-504.
4. Nadarajah K., Asharp T., Jeganathan Y. Biochar from waste biomass, its fundamentals, engineering aspects, and potential applications: An overview // Water Science and Technology. – 2024. – Vol. 89. – No. 5. – P. 1211-1239.
5. Li S., Wu Y., Nie F., Tu W., Li X., Luo X., Luo Y., Fan H., Song T. Remediation of nitrate contaminated groundwater using a simulated PRB system with an La-CTAC-modified biochar filler // Frontiers in Environmental Science. – 2022. – Vol. 10. – P. Article 986866.
6. Sakr M., El Agamawi H., Klammler H., Mohamed M.M. A review on the use of permeable reactive barriers as an effective technique for groundwater remediation // Groundwater for Sustainable Development. – 2023. – Vol. 21. – P. Article 100914.

7. Obiri-Nyarko F., Grajales-Mesa S.J., Malina G. An overview of permeable reactive barriers for in situ sustainable groundwater remediation // Chemosphere. – 2014. – Vol. 111. – P. 243-259.
8. Kraber. RSM helps to determine not only the main effects but also the interaction effects between factors. – [?]. – 2023.
9. Mariyam S., Alherbawi M., Pradhan S., Al-Ansari T., McKay G. Biochar yield prediction using response surface methodology: Effect of fixed carbon and pyrolysis operating conditions // Biomass Conversion and Biorefinery. – 2024. – Vol. 14. – P. 28879-28892.
10. Design-Expert software [Electronic resource]. – Available at: <https://www.statease.com/software/design-expert/>.
11. Pellera F.M., Regkouzas P., Manolikaki I., Diamadopoulos E. Biochar production from waste biomass: characterization and evaluation for agronomic and environmental applications // Detritus. – 2021. – Vol. 17. – P. 15-29.
12. Qiu M., Liu L., Ling Q., Cai Y., Yu S., Wang S., Fu D., Hu B., Wang X. Biochar for the removal of contaminants from soil and water: A review // Biochar. – 2022. – Vol. 4(19).
13. Hossain N., Nizamuddin S., Griffin G., Selvakannan P., Mubarak N.M., Mahlia T.M.I. Synthesis and characterization of rice husk biochar via hydrothermal carbonization for wastewater treatment and biofuel production // Scientific Reports. – 2020. – Vol. 10. – P. Article 18851.
14. Palniandy L.K., Yoon L.W., Wong W.Y., Yong S.T., Pang M.M. Application of biochar derived from different types of biomass and treatment methods as a fuel source for direct carbon fuel cells // Energies. – 2019. – Vol. 12. – P. Article 2477.

This research is funded by the Committee of Science of the Ministry of Science and Higher Education of the Republic of Kazakhstan (Grant No. BR24992867).

Received: 29 August 2025

Accepted: 15 September 2025

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ЖАМБЫЛ ӨҢІРІНІҢ АГРОӨНЕРКЕСІПТІК ҚАЛДЫҚТАРЫНАН АЛЫНГАН БИОЧАР ӨНДІРІСІН ҚӨПФАКТОРЛЫ МОДЕЛЬДЕУ АРҚЫЛЫ ОҢТАЙЛАНДЫРУ ЖӘНЕ ОНЫ ӨТКІЗГІШ РЕАКТИВТІ ТОСҚАУЫЛДАРДА ПАЙДАЛАНУ

Аннотация. Ауыз су ресурстарының ауыр металдармен және органикалық ластағыштармен ластануы қоршаған орта үшін елеулі проблемаға айналуда, әсіресе Жамбыл облысы сияқты өнірлерде, мұндағы су сапасы орташа ластанғаннан бастап жоғары деңгейде ластануға дейін ауытқиды. Бұл зерттеу Жамбыл облысындағы күріш қауызынан алынған биочар өндірісін оңтайландыруға бағытталған және оны жер асты суларының биологиялық тазартылуына арналған өткізгіш реактивті барьерлерде (ӨРБ) қолдану мүмкіндігін арттыру мақсатында жауап беретінің әдісі (Response Surface Methodology, RSM) пайдаланылды. Box-Behnken дизайны негізінде пиролиз процесінің негізгі параметрлері – температура, активация үақыты және күріш қауызымен ортофосфор қышқылының (H_3PO_4) арақатынасы жүйелі түрде өзгертілді. Мақсат – биочардың шығымын және адсорбциялық қабілетін арттыру. Дисперсиялық талдау (ANOVA) нәтижелері квадраттектік модельдің статистикалық

маңыздылығын көрсетті ($p < 0,05$, $R^2 = 0,9662$). Оңтайлы жағдайлар шамамен 2,2 г H_3PO_4 және 540°C температурада анықталып, био-көмірдің шығымы 84%-ға дейін жетті. Зерттеу нәтижелері оңтайландырылған биочардың қоршаған ортаны қалпына келтіруде, атап айтқанда ΘРБ жүйелерінде, тұрақты және үнемді материал ретінде қолданылу әлеуетін дәлелдейді.

Тірек сөздер: биочар, агропромышленные отходы, методология отклика поверхности (МОП), проницаемые реактивные барьеры (ПРБ), пиролиз, морфология поверхности.

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ОПТИМИЗАЦИЯ ПРОИЗВОДСТВА БИОУГЛЯ ИЗ АГРОПРОМЫШЛЕННЫХ ОТХОДОВ ЖАМБЫЛСКОГО РЕГИОНА ПУТЕМ МНОГОФАКТОРНОГО МОДЕЛИРОВАНИЯ И ЕГО ИСПОЛЬЗОВАНИЕ В ПРОНИЦАЕМЫХ РЕАКТИВНЫХ БАРЬЕРАХ

Аннотация. Усиливающееся загрязнение водных ресурсов тяжёлыми металлами и органическими поллютантами представляет собой серьёзную экологическую проблему, особенно в таких регионах, как Жамбылская область Казахстана, где качество воды варьируется от умеренно до крайне загрязнённого. Настоящее исследование направлено на оптимизацию производства биочара из агропромышленных отходов – в частности, рисовой шелухи, собранной в Жамбылском регионе – с применением метода ответной поверхности (Response Surface Methodology, RSM) с целью повышения эффективности материала в составе проницаемых реактивных барьеров (ПРБ) для очистки подземных вод. С использованием экспериментального плана Box-Behnken были систематически варьированы ключевые параметры пиролиза: температура, время активации и соотношение рисовой шелухи к ортофосфорной кислоте (H_3PO_4), чтобы максимизировать выход биочара и его адсорбционную способность. Статистический анализ с применением ANOVA подтвердил значимость квадратичной модели ($p < 0,05$, $R^2 = 0,9662$), при этом оптимальные условия составили около 2,2 г H_3PO_4 и 540°C, что позволило достичь выхода до 84%. Полученные результаты подтверждают потенциал оптимизированного биочара как устойчивого и экономически эффективного материала для экологической ремедиации, особенно в системах ПРБ.

Ключевые слова: биоуголь, агропромышленные отходы, методология отклика поверхности (МОП), проницаемые реактивные барьеры (ПРБ), пиролиз, морфология поверхности.