

Research Article

Optimization of Activated Charcoal Synthesis from Sugar Palm Shells for the Adsorption of Iron Ions in Batik Waste

Optimasi Pembuatan Arang Aktif dari Kulit Kolang-Kaling sebagai Adsorben Ion Logam Fe Limbah Batik

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Abstract

Efforts have been made to improve the synthesis of activated charcoal from sugar palm shells (*Arenga pinnata*). Therefore, the purpose of this study was to use the activated charcoal from sugar palm shells to adsorb Iron (Fe) ions contained in batik waste. Charcoal was activated with a 20-30% phosphoric acid solution (H_3PO_4), dried at 110 °C for 3 hours, and then calcined at a range of 700-900 °C for 3-5 hours. The results demonstrated that the optimal adsorption of Fe ions was achieved under conditions of 25% H_3PO_4 concentration, calcination at 800 °C for 4 hours, yielding 46.94% adsorbed Fe, and the largest surface area of 45.087 m²/g.

Keywords: adsorbent; activated charcoal; batik waste; Fe ions; sugar palm shell

Abstrak

Telah dilakukan penelitian tentang optimasi pembuatan arang aktif dari kulit kolang-kaling (*Arenga pinnata*). Tujuan dari penelitian ini adalah untuk menyerap ion logam Fe yang terkandung pada limbah batik dengan menggunakan arang aktif dari kulit kolang-kaling. Arang diaktifkan dengan menggunakan larutan asam fosfat dengan rentang konsentrasi 20-30%, kemudian dikeringkan pada suhu 110°C selama 1 jam, dan dikalsinasi dengan rentang suhu 700-900 °C untuk rentang waktu 3-5 jam. Hasil menunjukkan bahwa kondisi adsorpsi optimal dihasilkan pada penggunaan konsentrasi 25% asam fosfat, suhu kalsinasi 800 °C, dengan lama kalsinasi 4 jam dimana ion Fe yang terserap sebesar 46,94%. Pada suhu kalsinasi 800 °C menghasilkan adsorben dengan luas permukaan tertinggi yaitu 45,087 m²/g.

Kata kunci: adsorben; arang aktif; ion Fe; kulit kolang-kaling; limbah batik

1. Introduction

In Indonesia, the batik industry is classified into large, medium, small, and even home scale industries. The pollution caused by the batik industry not only occurs in industrial areas but also in densely populated settlements.

Furthermore, one of the Small Medium Enterprises (SMEs) in Yogyakarta produces around 125 L of liquid waste per kg of batik [1] and this liquid waste contains 4.23 mg/L COD, 5.47 mg/L ammonia, 535 mg/L phenol, 535 mg/L TSS, 2.0587 mg/L Fe, and other metals [2].

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The presence of heavy metals in water or wastewater with concentrations exceeding the threshold can damage normal biological cycles in the environment. Among the metal ions polluting the environment are cadmium, lead, zinc, mercury, copper, and iron [3][4]. Filtration, chemical precipitation, ion exchange adsorption, electrodeposition, and membrane systems are techniques that have been developed for the extraction of heavy metals from water [5]. One technique that has been widely developed is the principle of solid-phase extraction utilizing specific adsorbents because it does not require hazardous solvents. The effort to control metal ion waste has recently increased, prompting the search for novel methods that are cheap, effective, and efficient. One method is by using activated charcoal as a medium to adsorb metals.

Silica and bentonite can adsorb heavy metal Fe in batik waste by 49.48% and 2.9%, respectively [2]. Another study conducted metal adsorption on batik waste using corn husk cellulose as the adsorbent, and it was able to reduce Cr metal content by 28.94% [6]. This indicates that natural materials and waste containing cellulose can be processed into adsorbents to adsorb metals in waste.

In the Limbangan sub-district, Kendal district, sugar palms are quite abundant and 50 kg of their shells are produced as waste from 100 kg of the palms [7]. This waste is usually only disposed of and used as natural compost, but composting takes a long time. The shell is relatively strong and contains lignin, cellulose, and hemicellulose [8]. Furthermore, it contains porous fibers that have the potential to be used as activated carbon [9]. Therefore, using this shell as a

metal adsorbent makes it more beneficial for adsorbing bulk metals than being composted.

The purpose of this study was to convert sugar palm shells into activated charcoal and obtain optimal conditions for the synthesis of activated charcoal from the shells to be used as an adsorbent for Fe ions in batik liquid waste.

2. Material and Method

This experiment was held in the Agricultural Technology Laboratory, Stiper Agricultural University, Yogyakarta. The materials used are sugar palm shells obtained from Tercel village, Limbangan District, Kendal regency, pH meter, filter paper, phosphoric acid (H_3PO_4) and aquadest obtained from Chem-Mix Pratama Yogyakarta.

2.1. Semi-carbonized sugar palm shell

A 2 kg sugar palm shell was collected and placed in a barrel to be semi-carbonized in an oven at 110 °C before being analyzed for cellulose, lignin, and hemicellulose. The charcoal was ground and sieved using a Tyler sieve with a mesh size of 60 [10].

2.2. Charcoal activation process

A 10 g charcoal was dissolved in a 20-30% H_3PO_4 solution then soaked for 24 hours. It was dried in an oven at 110 °C for 1 hour. The mixture was then calcined at 700-900 °C for 3-5 hours.

2.3. Fe ions adsorption process

Activated charcoal and batik liquid waste were stirred for 40 minutes and each sample was analyzed by Atomic Absorption spectrophotometer (AAS) AA-6200 Shimadzu.

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2.4. Experimental design

Design expert 12.0 software was used in processing the data. The method used was the Response surface methodology (RSM), which is a simple and efficient method that simplifies process optimization[11]. In addition, the experimental design of activated charcoal synthesis was determined by using important parameters namely, phosphoric acid concentration, temperature, and calcination time, and central composite design was chosen as the experimental design. The RSM is an experimental strategy that is used when the response is influenced by multiple factors with the objective of this study being to determine the optimum response. RSM problems include selecting a suitable design for optimization and a factor space search method to reach the optimum quickly. This experiment consisted of 16 trial runs and the polynomial equation is shown in equation 1 [12][13][14][15].

$$Y = \beta_0 + \sum_{i=1}^3 \beta_i X_i + \sum_{i=1}^3 \beta_{ii} X_i^2 + \dots + \sum_{i < j} \beta_{ij} X_i X_j \dots \dots \dots (1)$$

3. Result and Discussion

Table 1 shows the results of the adsorbed Fe ions and Table 2 shows the significance of each factor by testing the F-value and p-value. A high F-value and a low p-value < 0.05 indicate variables that have a significant effect on the observed response.

Based on the results of ANOVA for adsorbed Fe ions, factor B is the most significant factor, with a low p-value (0.0835) and a high F-value (4.30). The objective function of the results of this test is used to determine the optimal H₃PO₄ concentration (X₁), calcination temperature (X₂), and calcination time (X₃). Subsequently, the interaction between the

operating variables and the adsorbed Fe ions can be expressed mathematically in a 2nd order polynomial equation based on equation 2.

$$Y = -817.03972 + 5.25828X_1 + 1.90092X_2 + 14.23310X_3 - 0.00102X_1X_2 - 0.0342X_1X_3 - 0.0017X_2X_3 - 0.088166X_1^2 - 0.001156X_2^2 - 1.52414X_3^2 \dots \dots \dots (2)$$

The coefficient of determination is used for order selection, with the recommended model being that with the highest value. The coefficient of determination (R²) for the 2nd order polynomial equation in this study is 0.9395, indicating a 93.95% match between the experimental and predicted data. In addition, the model can be declared accurate if the value of R² exceeds 70% implying that the value estimated by the model is close to the value obtained from the experimental results [16].

3.1. Interaction between H₃PO₄ concentration and calcination temperature to adsorb Fe ions

Figures 1 and 2 showed a contour graph between H₃PO₄ concentration and calcination temperature, and a 3D optimization chart between H₃PO₄ concentration and calcination temperature respectively. The higher the H₃PO₄ concentration, the more concentrated the activator used. Furthermore, there will be blockages in the pores of the activated charcoal due to the high ash content. The higher the ash content in activated charcoal, the more pores will become clogged [17]. However, the higher the calcination temperature employed, the greater the pore surface area of the adsorbent, and the greater the adsorption of the adsorbent [18].

Optimization of Activated Charcoal Synthesis from Sugar Palm Shells for the adsorption of Iron Ions in Batik Waste**Table 1.** Adsorbed Metal Ions Results

Run	H ₃ PO ₄ Conc. (%)	Temperature (°C)	Time (h)	Adsorbed Fe (%)
1	20	700	3	26,53
2	20	700	5	27,21
3	20	800	4	39,12
4	20	900	3	31,63
5	20	900	5	30,95
6	25	700	4	27,55
7	25	800	3	40,14
8	25	800	4	46,6
9	25	800	4	46,94
10	25	800	5	39,8
11	25	900	4	32,31
12	30	700	3	26,87
13	30	700	5	26,19
14	30	800	4	39,46
15	30	900	3	29,25
16	30	900	5	28,57

Table 2. ANOVA Results

Source	Coeff.	Df	F-value	p-value
Model	-817.03972	9	10.35	0.0051
X ₁ -H ₃ PO ₄ Conc (%)	5.25828	1	0.3318	0.5855
X ₂ -Temp (°C)	1.90092	1	4.30	0.0835
X ₃ -Time (h)	14.23310	1	0.0369	0.8541
X ₁ X ₂	-0.001020	1	0.2655	0.6248
X ₁ X ₃	-0.034000	1	0.0295	0.8693
X ₂ X ₃	-0.001700	1	0.0295	0.8693
X ₁ ²	-0.088166	1	1.63	0.2484
X ₂ ²	-0.001156	1	44.98	0.0005
X ₃ ²	-1.52414	1	0.7813	0.4108

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Factor Coding: Actual
 ● Design Points
 26.19 46.94

X1 = A: H₃PO₄ Concentration
 X2 = B: Temperature

Actual Factor
 C: Time = 4

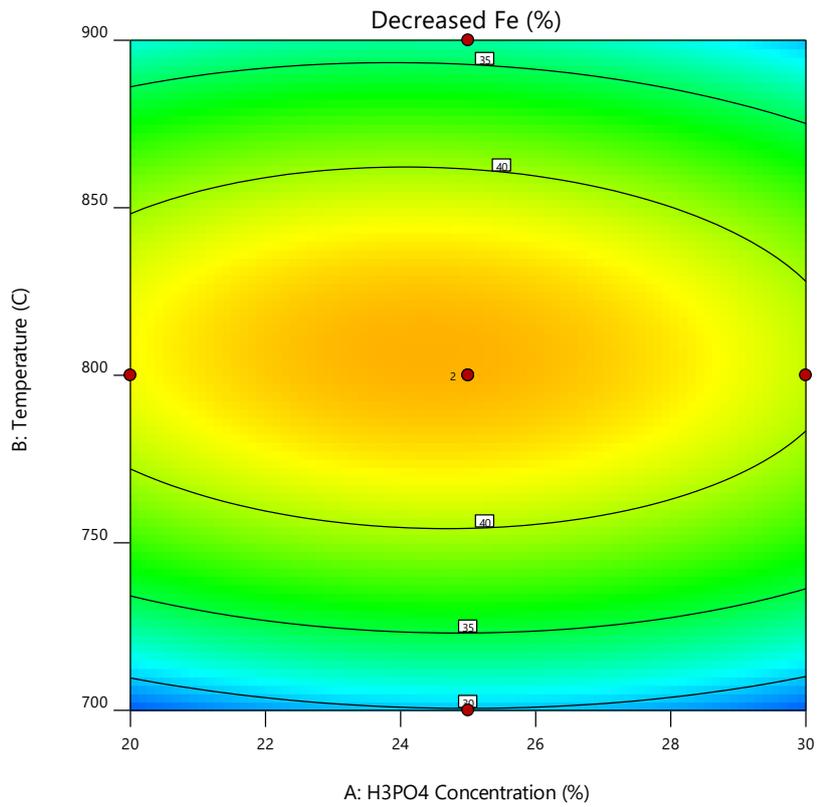


Figure 1. Contour graph of H₃PO₄ concentration vs calcination temperature

Factor Coding: Actual
 Design Points:
 ● Above Surface
 ○ Below Surface
 26.19 46.94

X1 = A: H₃PO₄ Concentration
 X2 = B: Temperature

Actual Factor
 C: Time = 4

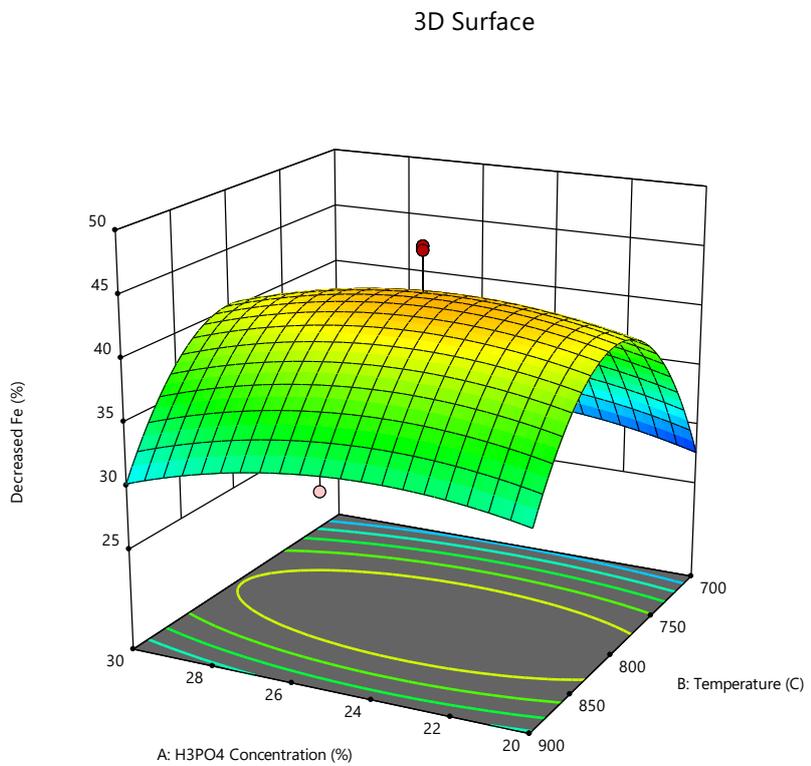


Figure 2. 3D Optimization chart of H₃PO₄ concentration vs calcination temperature

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3.2. Interaction between H_3PO_4 concentration and calcination time to adsorb Fe ions

Figures 3 and 4 showed the contour graph between H_3PO_4 concentration and calcination time, and a 3D optimization chart of H_3PO_4 concentration against calcination time respectively. It was discovered that increasing the calcination time did not significantly affect the adsorbed Fe ions. These ions adsorbed by

the activated charcoal were less than optimal due to the increased concentration of H_3PO_4 used. Subsequently, this resulted in a large number of activators that were not activated by the sugar palm shell charcoal, causing a pile of dirt in the pores. activated carbon pores become damaged if the activator is too large, resulting in a decreased pore volume and low adsorption [18].

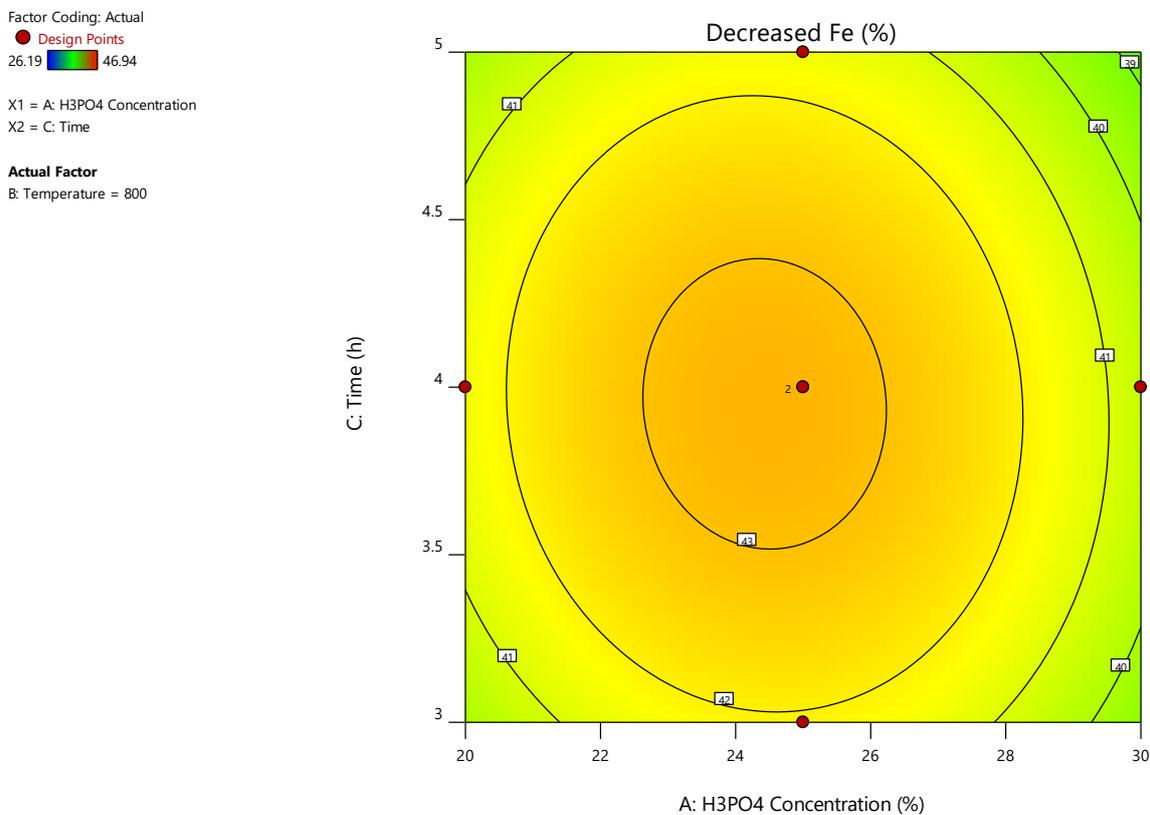


Figure 3. Contour graph of H_3PO_4 concentration vs time of calcination

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Factor Coding: Actual
 Design Points:
 ● Above Surface
 ○ Below Surface
 26.19  46.94

X1 = A: H3PO4 Concentration
 X2 = C: Time

Actual Factor
 B: Temperature = 800

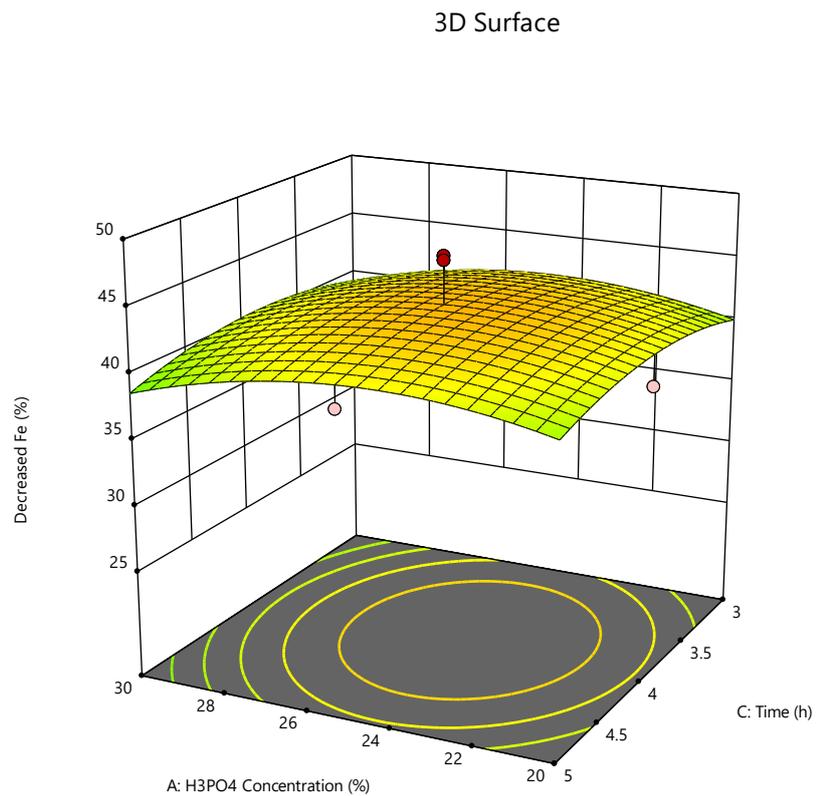


Figure 4. 3D Optimization chart of H₃PO₄ concentration vs time of calcination

3.3 Effect of temperature and calcination time on adsorption of Fe ions

Figure 5 and 6 shows the interaction between temperature and calcination time and the resulting adsorbed Fe ions. Calcination temperature is a sensitive factor in the formation of an activated charcoal surface area environment. As the calcination temperature increases, the surface area will increase and decrease as it reaches the optimum point [17].

As indicated in Table 3, the BET results were only applied on three samples with a concentration of 25% H₃PO₄ and 4

hours calcination time at different temperatures. The surface area of the adsorbent produced is still significantly below the standard surface area of activated charcoal, which is roughly 300 - 3500 m²/g [19].

Table 3. BET Results of Activated Charcoal at Different Calcination Temperature

No	Temp (°C)	Surface area (m ² /g)
1	700	28.925
2	800	45.087
3	900	32.172

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Factor Coding: Actual
 ● Design Points
 26.19 46.94
 X1 = B: Temperature
 X2 = C: Time
Actual Factor
 A: H3PO4 Concentration = 25

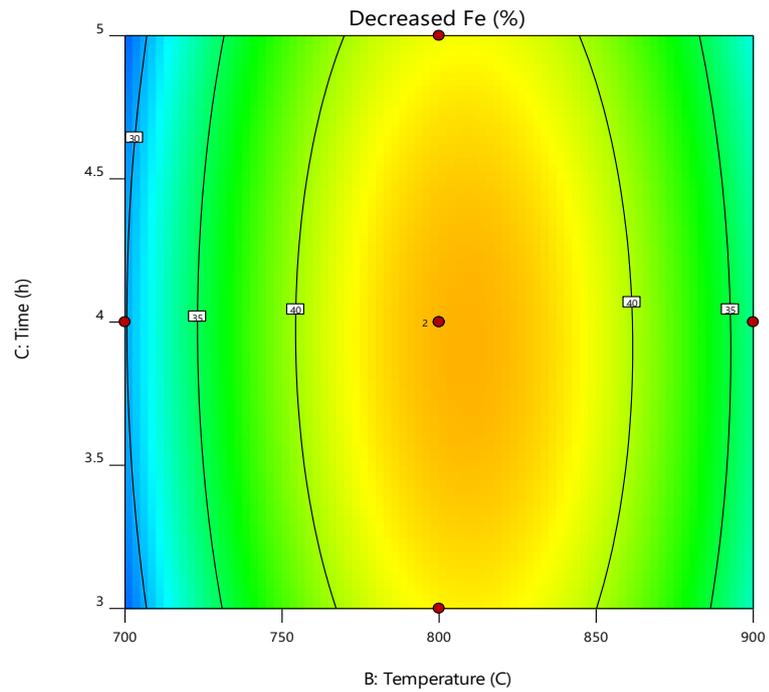


Figure 5. Contour graph of temperature vs calcination time

Factor Coding: Actual
 Design Points:
 ● Above Surface
 ○ Below Surface
 26.19 46.94
 X1 = B: Temperature
 X2 = C: Time
Actual Factor
 A: H3PO4 Concentration = 25

3D Surface

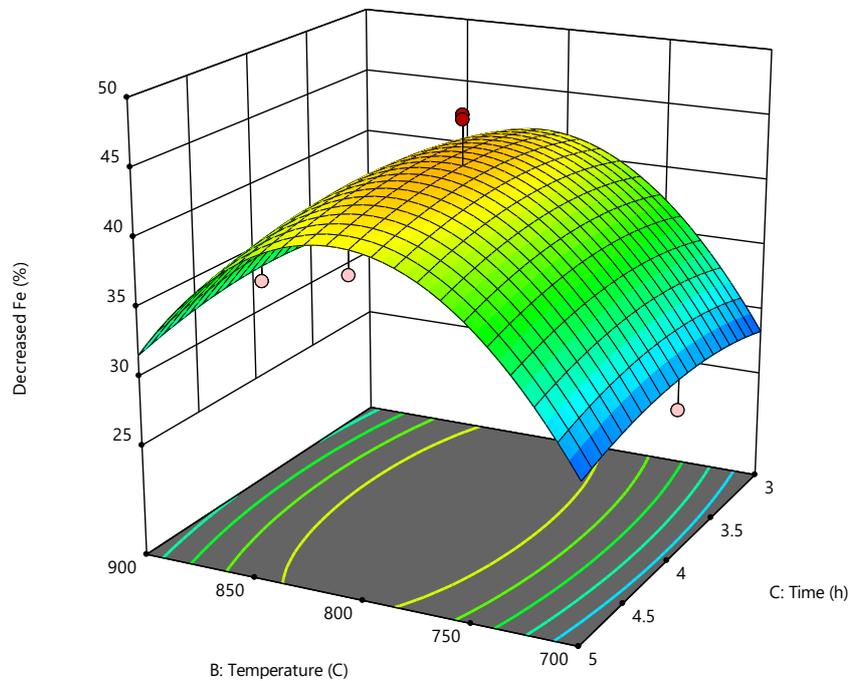


Figure 6. 3D Optimization chart of temperature and calcination time

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4. Conclusion

This study demonstrated the optimization of activated charcoal synthesis from sugar palm shell using design expert 12.0 software, where the variables used in the activation process include phosphoric acid concentration, calcination temperature, and time. The results showed that the optimal adsorption of Fe ions was obtained under conditions of 25% H₃PO₄ concentration, 800 °C

calcination temperature, and 4 hours calcination time, yielding 46.94% adsorbed Fe. The largest surface area of 45.087% was attained at a calcination temperature of 800 °C. Moreover, further study is required to increase the surface area and adsorption capacity of the adsorbent by varying the activator used.

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