Scavenging of caffeine from aqueous medium through optimized H₃PO₄-activated Acacia mangium wood activated carbon: Statistical data of optimization

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A B S T R A C T

The optimization data presented here are part of the study planned to remove the caffeine from aqueous solution through the large surface area optimized H₃PO₄-activated Acacia mangium wood activated carbon (OAMW-AC). The maximum adsorption capacity of the OAMW-AC for caffeine adsorption was achieved (30.3 mg/g) through optimized independent variables such as, OAMW-AC dosage (3.0 g/L), initial caffeine concentration (100 mg/L), contact time (60 min), and solution pH (7.7). The adsorption capacity of OAMW-AC was optimized with the help of rotatable central composite design of response surface methodology. Under the stated optimized conditions for maximum adsorption capacity, the removal efficiency was calculated to be 93%. The statistical significance of the data set was tested through the analysis of variance (ANOVA) study. Data confirmed the statistical model for caffeine adsorption was significant. The regression coefficient (R²) of curve fitting through the
quadratic model was found to be 0.9832, and the adjusted regression coefficient was observed to be 0.9675.

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1. Data

Based on the earlier reported results on caffeine adsorption [5–8], it was observed that caffeine adsorption parameters such as contact time, adsorbent dosage, initial concentration, and solution pH were not optimized by the previous researchers. In this data article, the optimized parameters with their statistical significance are reported. The experimental variables and their response with ranges and standard deviations are illustrated in Table 1. The dataset contains results of rotatable central composite design of design of experiment software version 6. The experiments were conducted in batch mode, after each experiment the residual caffeine
concentrations were calculated using UV–Vis spectroscopy (Hitachi U2000) at $\lambda_{\text{max}}$ 274 nm. Table 2 describes the experimental plan for different combinations of independent variables and their corresponding results on adsorption capacity. As a result, the adsorption capacity varied from 3.7 to 40.0 mg/g with a standard deviation of 8.8 mg/g. Fig. 1 contains six contour plot, each plot depicts the change in adsorption capacity of OAMW-AC when two independent variables changes simultaneously, while other two independent variables kept constant. The adsorption capacity lines shown in the contour plot is above and below of the optimized independent variables, therefore, the values are less than the optimized response (adsorption capacity 30.3 mg/g).

A correlation matrix of regression coefficient and a correlation matrix of factors (Pearson’s r) were generated and displayed in Table 3 and Table 4; ‘A’ is the contact time (min), ‘B’ is the adsorbent dose (g/L), ‘C’ is the adsorbate concentration (mg/L), and ‘D’ is the pH of the solution.

### Table 1
Variables, ranges, standard deviation, and response design summary.

| Name                  | Units | Type    | Std. Dev. | Low | High |
|-----------------------|-------|---------|-----------|-----|------|
| Contact time          | min   | Factor  | 1         | 60  | 175  |
| OAMW-AC dosage        | g/L   | Factor  | 0.6066    | 3   | 7    |
| Initial caffeine concentration | mg/L | Factor  | 8.7       | 50  | 100  |
| pH                    | Factor|         | 0.38      | 4   | 8    |
| Adsorption capacity   | mg/g  | Response| 8.7861    | 4.9 | 40.3 |

### Table 2
Parameters and design layout for planned design of experiments.

| Sdt | Run | Variables          | Adsorbent dose (g/L) | Adsorbate concentration (mg/L) | pH | Response Adsorption capacity (mg/g) |
|-----|-----|--------------------|----------------------|-------------------------------|----|-------------------------------------|
| 15  | 1   | 60.0               | 7.0                  | 100                           | 8.0| 12.6                                |
| 9   | 2   | 60.0               | 3.0                  | 50                            | 8.0| 14.4                                |
| 17  | 3   | 2.5                | 5.0                  | 75                            | 6.0| 13.3                                |
| 28  | 4   | 117.5              | 5.0                  | 75                            | 6.0| 11.1                                |
| 30  | 5   | 117.5              | 5.0                  | 75                            | 6.0| 11.4                                |
| 13  | 6   | 60.0               | 3.0                  | 100                           | 8.0| 30.1                                |
| 7   | 7   | 60.0               | 7.0                  | 100                           | 4.0| 12.3                                |
| 10  | 8   | 175.0              | 3.0                  | 50                            | 8.0| 14.0                                |
| 22  | 9   | 117.5              | 5.0                  | 125                           | 6.0| 21.9                                |
| 24  | 10  | 117.5              | 5.0                  | 75                            | 6.0| 13.3                                |
| 20  | 11  | 117.5              | 9.0                  | 75                            | 6.0| 7.4                                 |
| 29  | 12  | 117.5              | 5.0                  | 75                            | 6.0| 13.7                                |
| 25  | 13  | 117.5              | 5.0                  | 75                            | 6.0| 13.5                                |
| 3   | 14  | 60.0               | 7.0                  | 50                            | 4.0| 6.4                                 |
| 21  | 15  | 117.5              | 5.0                  | 25                            | 6.0| 4.9                                 |
| 14  | 16  | 117.5              | 3.0                  | 100                           | 8.0| 29.5                                |
| 1   | 17  | 60.0               | 3.0                  | 50                            | 4.0| 14.4                                |
| 11  | 18  | 60.0               | 7.0                  | 50                            | 8.0| 6.3                                 |
| 26  | 19  | 117.5              | 5.0                  | 75                            | 6.0| 13.5                                |
| 27  | 20  | 117.5              | 5.0                  | 75                            | 6.0| 13.3                                |
| 8   | 21  | 175.0              | 7.0                  | 100                           | 4.0| 13.1                                |
| 23  | 22  | 117.5              | 5.0                  | 75                            | 2.0| 12.0                                |
| 4   | 23  | 175.0              | 7.0                  | 50                            | 4.0| 6.4                                 |
| 2   | 24  | 175.0              | 3.0                  | 50                            | 4.0| 14.8                                |
| 12  | 25  | 175.0              | 7.0                  | 50                            | 8.0| 6.4                                 |
| 6   | 26  | 175.0              | 3.0                  | 100                           | 4.0| 30.0                                |
| 18  | 27  | 232.5              | 5.0                  | 75                            | 6.0| 13.6                                |
| 19  | 28  | 175.0              | 1.0                  | 75                            | 6.0| 40.3                                |
| 5   | 29  | 60.0               | 3.0                  | 100                           | 4.0| 30.2                                |
| 16  | 30  | 175.0              | 7.0                  | 100                           | 8.0| 12.9                                |
Fig. 1. Contour plots showing change in the adsorption capacity of OAMW-AC with changing two variables simultaneously.
Furthermore, the variance inflation factor (VIF) and the power at 5% alpha level for effect of $\frac{1}{2}$, 1, and 2 standard deviations were determined (Table 5). The degrees of freedom can be found in Table 6. Additionally, the leverages derived from the $(XX)^{-1}$ are stated in Table 7. Fig. 2 shows the perturbation of the StdErr of design.

Table 3
Correlation matrix of the regression coefficient.

|       | Intercept | A     | B     | C     | D     | A²    | B²    | C²    |
|-------|-----------|-------|-------|-------|-------|-------|-------|-------|
| Intercept | 1.000    |       |       |       |       |       |       |       |
| A     | 1.000     | −0.000 | 1.000 |       |       |       |       |       |
| B     | 1.000     | −0.000 | −0.000| 1.000 |       |       |       |       |
| C     | 1.000     |       | −0.000| −0.000| 1.000 |       |       |       |
| D     | 1.000     |       |       | −0.000| −0.000| 1.000 |       |       |
| A²    | 1.000     |       |       |       | −0.000| −0.000| −0.000| 1.000 |
| B²    | 1.000     |       |       |       |       | −0.000| −0.000| −0.000|
| C²    | 1.000     |       |       |       |       |       | −0.000| −0.000|
| D²    | 1.000     |       |       |       |       |       |       | −0.000|

Table 4
Correlation matrix of factors.

|       | A     | B     | C     | D     | A²    | B²    | C²    |
|-------|-------|-------|-------|-------|-------|-------|-------|
| A     | 1.000 |       |       |       |       |       |       |
| B     | 1.000 |       |       |       |       |       |       |
| C     | 1.000 |       |       |       |       |       |       |
| D     | 1.000 |       |       |       |       |       |       |
| A²    | 1.000 |       |       |       |       |       |       |
| B²    | 1.000 |       |       |       |       |       |       |
| C²    | 1.000 |       |       |       |       |       |       |
| D²    | 1.000 |       |       |       |       |       |       |

Table 5

Table 6

Correlation matrix of factors.
The model was analyzed through a sequential model sum of squares (Table 8), a lack of fit test (Table 9) and model summary statistics (Table 10). The data of the analysis of variance is described in Table 11. There is a 0.01% chance that this model could occur due to noise and an 21.5% chance that the F-value of lack of fit occurs due to noise. The adeq. Precision for the design of experiment is 31.6. Table 12 shows the factors for the equation to predict the adsorption capacity and Table 13 represented the diagnostics case in statistical design. In addition to the normal plot of residuals. Fig. 3 illustrate the studentized residuals [a] depending on the predicted [b], run number [c], contact time [d], OAMW-AC dosage [e], initial caffeine concentration [f] and pH [g]. Fig. 4 shows the Outlier t [a], Cook's Distance [b] and leverage [c] against run number and the predicted against actual [d]. The box-cox plot for power transforms can be seen in Fig. 5.

Finally, the optimum independent variables for caffeine adsorption, outcome response as adsorption capacity, and propagation of error in the results due to deviations in the independent variables are represented in Fig. 6. The descriptive plot for propagation error in the adsorption capacity owing to deviations in the independent variables, considering two variables at a time, is represented through six plots as shown in Fig. 7.

2. Experimental design, materials, and methods

The Experimental Design was calculated through the software Design Expert (version 6.0.6 Stat-Ease Inc. Minneapolis, USA). The activated carbon was produced from wood sawdust of Acacia mangium by the method described by Danish et al., 2014 [9]. The flow diagram of the experiment conducted to generate this data set is shown in Fig. 8. Effect of contact time on the caffeine adsorption was studied at the time interval of 2.5 min, 60 min, 117.5 min, 175 min, and 232.5 min. The initial concentration of caffeine varies at 25.00 (±0.35) mg/L, 50.00 (±1.92) mg/L, 75.00 (±2.73) mg/L, 100.00 (±1.71) mg/L, and 125.00 (±3.99) mg/L; and the effect of pH on OAMW-AC were studied at five different pH levels: 2.0 (±0.08), 4.0 (±0.15), 6.0 (±0.11), 8.0 (±0.08), and 10.0 (±0.10) mg/L.
Table 7
Measures derived from \((X'X)^{-1}\) matrix.

| Std | Leverage | Point Type |
|-----|----------|------------|
| 1   | 0.5833   | Fact       |
| 2   | 0.5833   | Fact       |
| 3   | 0.5833   | Fact       |
| 4   | 0.5833   | Fact       |
| 5   | 0.5833   | Fact       |
| 6   | 0.5833   | Fact       |
| 7   | 0.5833   | Fact       |
| 8   | 0.5833   | Fact       |
| 9   | 0.5833   | Fact       |
| 10  | 0.5833   | Fact       |
| 11  | 0.5833   | Fact       |
| 12  | 0.5833   | Fact       |
| 13  | 0.5833   | Fact       |
| 14  | 0.5833   | Fact       |
| 15  | 0.5833   | Fact       |
| 16  | 0.5833   | Fact       |
| 17  | 0.5833   | Axial      |
| 18  | 0.5833   | Axial      |
| 19  | 0.5833   | Axial      |
| 20  | 0.5833   | Axial      |
| 21  | 0.5833   | Axial      |
| 22  | 0.5833   | Axial      |
| 23  | 0.5833   | Axial      |
| 24  | 0.5833   | Axial      |
| 25  | 0.1667   | Center     |
| 26  | 0.1667   | Center     |
| 27  | 0.1667   | Center     |
| 28  | 0.1667   | Center     |
| 29  | 0.1667   | Center     |
| 30  | 0.1667   | Center     |
| Average | 0.5000 |           |

Fig. 2. Perturbation plots for the statistical design.
Table 8
Sequential model sum of squares.

| Source   | Sum of Squares | DF | Mean Square | F Value | Prob > F |
|----------|----------------|----|-------------|---------|----------|
| Mean     | 6961.63        | 1  | 6961.63     |         |          |
| Linear   | 1775.47        | 4  | 443.87      | 32.73   | <0.0001  |
| 2FI      | 85.18          | 6  | 14.20       | 1.06    | 0.4183   |
| Quadratic| 218.26         | 4  | 54.56       | 22.98   | <0.0001  |
| Cubic    | 28.68          | 8  | 3.58        | 3.62    | 0.0538   |
| Residual | 6.94           | 7  | 0.99        |         |          |
| Total    | 9076.16        | 30 | 302.54      |         |          |

Table 9
Lack of fit tests.

| Source   | Sum of Squares | DF | Mean Square | F Value | Prob > F |
|----------|----------------|----|-------------|---------|----------|
| Linear   | 332.18         | 20 | 16.61       | 12.08   | 0.0058   |
| 2FI      | 247.00         | 14 | 17.64       | 12.83   | 0.0054   |
| Quadratic| 28.74          | 10 | 2.87        | 2.09    | 0.2151   |
| Cubic    | 0.066          | 2  | 0.033       | 0.024   | 0.9765   |
| Pure Error| 6.88          | 5  | 1.38        |         |          |

Table 10
Model summary statistics.

| Source   | Std. Dev. | R-Squared | Adjusted R-Squared | Predicted R-Squared | PRESS |
|----------|-----------|-----------|--------------------|---------------------|-------|
| Linear   | 3.68      | 0.8397    | 0.8140             | 0.7597              | 508.18|
| 2FI      | 3.68      | 0.8799    | 0.8167             | 0.7923              | 439.13|
| Quadratic| 1.54      | 0.9832    | 0.9674             | 0.9170              | 175.46|
| Cubic    | 1.00      | 0.9967    | 0.9864             | 0.9908              | 19.38 |

Table 11
Analysis of variance (ANOVA).

| Source    | Sum of Squares | DF | Mean Square | F value | Prob > F |
|-----------|----------------|----|-------------|---------|----------|
| Model     | 2078.91        | 14 | 148.49      | 62.54   | <0.0001  |
| A         | 0.042          | 1  | 0.042       | 0.018   | 0.8964   |
| B         | 1159.26        | 1  | 1159.26     | 488.21  | <0.0001  |
| C         | 616.11         | 1  | 616.11      | 259.47  | <0.0001  |
| D         | 0.060          | 1  | 0.060       | 0.025   | 0.8758   |
| A²        | 0.88           | 1  | 0.88        | 0.37    | 0.5517   |
| B²        | 211.85         | 1  | 211.85      | 89.22   | <0.0001  |
| C²        | 0.76           | 1  | 0.76        | 0.32    | 0.5795   |
| D²        | 0.012          | 1  | 0.012       | 0.005   | 0.9445   |
| AB        | 0.25           | 1  | 0.25        | 0.11    | 0.7501   |
| AC        | 0.000          | 1  | 0.000       | 0.000   | 0.9745   |
| AD        | 0.16           | 1  | 0.16        | 0.067   | 0.7987   |
| BC        | 84.64          | 1  | 84.64       | 35.65   | <0.0001  |
| BD        | 0.12           | 1  | 0.12        | 0.052   | 0.8234   |
| CD        | 0.010          | 1  | 0.010       | 0.000   | 0.9491   |
| Residual  | 35.62          | 15 | 2.37        |         |          |
| Lack of Fit| 28.74         | 10 | 2.87        | 2.09    | 0.2151   |
| Pure Error| 6.88           | 5  | 1.38        |         |          |
| Cor Total | 2114.53        | 29 |             |         |          |
For caffeine; by using 50 mg, 150 mg, 250 mg, 350 mg, and 450 mg in 50 mL of caffeine solution. The solutions of caffeine were prepared by diluting a stock solution (0.5 g in 1 L flask). Each solution was measured by a UV–Vis spectrometer at \( \lambda_{\text{max}} \) (maximum wavelength) 274 nm before the adsorption of caffeine to determine the exact initial concentration. Thirty experiments were conducted under the conditions which are shown in Table 2, after the adsorption had occurred, the

| Table 12 |
| Factors for the equation. |

| Factor | Coefficient Estimate | DF | Standard Error | 95% Cl Low | 95% Cl High | VIF |
|--------|----------------------|----|----------------|------------|-------------|-----|
| Intercept | 12.75 | 1 | 0.63 | 11.41 | 14.09 | |
| A | 0.042 | 1 | 0.31 | -0.63 | 0.71 | 1.00 |
| B | -6.95 | 1 | 0.31 | -7.62 | -6.28 | 1.00 |
| C | 5.07 | 1 | 0.31 | 4.40 | 5.74 | 1.00 |
| D | 0.050 | 1 | 0.31 | -0.62 | 0.72 | 1.00 |
| A^2 | 0.18 | 1 | 0.29 | -0.45 | 0.81 | 1.05 |
| B^2 | 2.78 | 1 | 0.29 | 2.15 | 3.41 | 1.05 |
| C^2 | 0.17 | 1 | 0.29 | -0.65 | 0.61 | 1.05 |
| D^2 | -0.021 | 1 | 0.29 | -0.70 | 0.95 | 1.00 |
| AB | 0.012 | 1 | 0.39 | -0.81 | 0.83 | 1.00 |
| AC | -0.10 | 1 | 0.39 | -0.92 | 0.72 | 1.00 |
| BC | 0.088 | 1 | 0.39 | -3.12 | -1.48 | 1.00 |
| CD | 0.025 | 1 | 0.30 | -0.80 | 0.85 | 1.00 |

| Table 13 |
| Diagnostics case statistics. |

| Standard Order | Actual Value | Predicted Value | Residual | Leverage | Student Residual | Cook’s Distance | Outlier t | Run order |
|----------------|--------------|-----------------|----------|----------|------------------|-----------------|-----------|-----------|
| 1              | 14.40        | 15.50           | -1.10    | 0.583    | -1.102           | 0.113           | -1.110    | 17        |
| 2              | 14.80        | 15.50           | -0.70    | 0.583    | -0.708           | 0.047           | -0.696    | 24        |
| 3              | 6.40         | 5.77            | 0.63     | 0.583    | 0.633            | 0.037           | 0.619     | 14        |
| 4              | 6.40         | 6.28            | 0.12     | 0.583    | 0.121            | 0.001           | 0.117     | 23        |
| 5              | 30.20        | 30.15           | 0.046    | 0.583    | 0.046            | 0.000           | 0.045     | 29        |
| 6              | 30.00        | 30.21           | -0.21    | 0.583    | -0.214           | 0.004           | -0.207    | 26        |
| 7              | 12.30        | 11.23           | 1.07     | 0.583    | 1.077            | 0.108           | 1.083     | 7         |
| 8              | 13.10        | 11.79           | 1.31     | 0.583    | 1.320            | 0.163           | 1.356     | 21        |
| 9              | 14.40        | 15.57           | -1.17    | 0.583    | -1.177           | 0.129           | -1.194    | 2         |
| 10             | 14.00        | 15.18           | -1.18    | 0.583    | -1.185           | 0.131           | -1.203    | 8         |
| 11             | 6.30         | 6.20            | 0.10     | 0.583    | 0.105            | 0.001           | 0.101     | 18        |
| 12             | 6.40         | 6.30            | 0.096    | 0.583    | 0.096            | 0.001           | 0.093     | 25        |
| 13             | 30.10        | 30.33           | -0.23    | 0.583    | -0.230           | 0.005           | -0.223    | 6         |
| 14             | 29.50        | 29.99           | -0.49    | 0.583    | -0.490           | 0.022           | -0.477    | 16        |
| 15             | 12.60        | 11.75           | 0.85     | 0.583    | 0.850            | 0.067           | 0.842     | 1         |
| 16             | 12.90        | 11.91           | 0.99     | 0.583    | 0.993            | 0.092           | 0.992     | 30        |
| 17             | 13.30        | 13.38           | -0.083   | 0.583    | -0.084           | 0.001           | -0.081    | 3         |
| 18             | 13.60        | 13.55           | 0.050    | 0.583    | 0.050            | 0.000           | 0.049     | 27        |
| 19             | 40.30        | 37.77           | 2.53     | 0.583    | 2.547            | 0.605           | 3.266     | 28        |
| 20             | 7.40         | 9.97            | -2.57    | 0.583    | -2.580           | 0.621           | -3.343    | 11        |
| 21             | 4.90         | 3.28            | 1.62     | 0.583    | 1.625            | 0.247           | 1.730     | 15        |
| 22             | 21.90        | 23.55           | -1.65    | 0.583    | -1.659           | 0.257           | -1.773    | 9         |
| 23             | 12.00        | 12.57           | -0.57    | 0.583    | -0.570           | 0.030           | -0.556    | 22        |
| 24             | 13.30        | 12.77           | 0.53     | 0.583    | 0.536            | 0.027           | 0.523     | 10        |
| 25             | 13.50        | 12.75           | 0.75     | 0.167    | 0.533            | 0.004           | 0.520     | 13        |
| 26             | 13.50        | 12.75           | 0.75     | 0.167    | 0.391            | 0.002           | 0.380     | 20        |
| 27             | 11.10        | 12.75           | -1.65    | 0.167    | -1.173           | 0.018           | -1.189    | 4         |
| 28             | 13.70        | 12.75           | 0.95     | 0.167    | 0.675            | 0.006           | 0.663     | 12        |
| 29             | 11.40        | 12.75           | -1.35    | 0.167    | -0.960           | 0.012           | -0.957    | 5         |
Fig. 3. Plot of the studentized residuals [a] depending on, predicted value of adsorption capacity [b], run number [c], contact time [d], OAMW-AC dosage [e], initial caffeine concentration [f] and solution pH [g].
OAMW-AC was filtrated, and the caffeine concentration was determined again. The adsorption capacity \( q_e (\text{mg/g}) \) was calculated using the following equation [10–12]:

\[
q_e = \frac{(C_i - C_e)}{C_{AC}}
\]

Fig. 4. Outlier t [a], Cook’s Distance [b] and leverage [c] against run number and the predicted against actual [d].

Fig. 5. Box-Cox plot for power transforms.
Fig. 6. Adsorption capacity optimization output for selected parameters taken within the range.

Fig. 7. The propagation of error in the adsorption capacity of OAMW-AC.
where, \( C_i \) is the initial concentration of caffeine (mg/L), \( C_e \) the concentration of caffeine after adsorption (mg/L) and \( C_{AC} \) the dosage of added OAMW-AC (g/L). For the calibration, five standards were measured within the linear range of 0.1–0.8 at the same wavelength. The average of the linear regression coefficient for all conducted calibrations was 0.999.

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**Conflict of Interest**

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

**Appendix A. Supplementary data**

Supplementary data to this article can be found online at https://doi.org/10.1016/j.dib.2019.105045.

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