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MODELLING THE EFFECTS OF CARBONISATION TEMPERATURE AND TIME ON ADSORPTION PERFORMANCE METRICS OF RICE HUSK ASH AND COCONUT SHELL ASH

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Abstract: The emissions from diesel engines continue to be a major environmental and health concern, especially in developing economies, where the use of outdated engines and generators is high and the availability of after-treatment technology is limited. The rationale behind this study is the need for affordable, locally available, and sustainable substitutes for exhaust emissions control. This study modelled the effects of temperature and time of carbonisation in combination with the chemical activation on the adsorption characteristics of Rice Husk Ash (RHA) and Coconut Shell Ash (CSA) while mechanically controlling diesel engine emissions. The process variables were carbonisation temperature (500–700 °C), carbonisation time (2–10 h) and activating reagent (phosphoric acid). Response surface methodology was used and the performance measures in the adsorption of diesel exhaust were Adsorption efficiency (%) and breakthrough time (min), which were measured based on diesel exhaust adsorption experiments. ANOVA statistical analysis revealed that the model quadrants developed were significant ($p < 0.0001$), and the coefficients of determination were high ($R^2 > 0.98$). The maximum adsorption capacity of 76% and the breakthrough duration of 138 minutes were attained at increased carbonisation temperatures and chemical activation. The findings showed that chemical activation and temperature of carbonisation are the prevailing factors that affect adsorption performance. The research finds that agro-waste-derived adsorbents, when optimised properly, can offer an efficient, sustainable, and long-lasting diesel emission control solution.

Keywords: Adsorption, Coconut Shell Ash, Emission, Phosphoric Acid, and Response Surface Methodology

BACKGROUND TO THE STUDY

The exhaust emission problem of diesel engines has become a significant concern worldwide, as more economies rely on diesel-powered systems for transportation, agriculture, and power generation, especially in developing countries (Razek *et al.*, 2025). Diesel engines have been found popular due to their durability, thermal efficiency and fuel economy; They are also primary contributors to harmful pollutants, including carbon monoxide (CO), nitrogen oxides (NO_x), unburned hydrocarbons (HC), particulate matter (PM), smoke and its emissions (Khanna *et al.*, 2025). Excessive exposure to such emissions is strongly linked to respiratory diseases, cardiovascular disorders, environmental degradation and climate change. The scenario is exacerbated by the fact that in most low- and middle-income countries, such as Nigeria, there is widespread use of older diesel engines and standby generators, lax emission standards and high prices for more modern after-treatment equipment, including diesel particulate filters and selective catalytic reduction systems (Bebetidoh, 2022). These limitations require the development of economically efficient, locally available and environmentally friendly alternatives to emission control.

Adsorption-based emission control has developed as an attractive mechanical method for overcoming diesel emissions, as it is straightforward, flexible, and relatively inexpensive to operate (Festus *et al.*, 2025). The success of this method will greatly depend on the physicochemical characteristics of the adsorbent material used, including surface area, porosity, surface functional groups, and thermal stability. In this regard, agro-waste materials such as rice husk ash and coconut shell ash are highly favourable, as they are abundant, renewable, inexpensive, and contain carbon and silica constituents that are beneficial for adsorption. Nigeria ranks among the top producers of rice and coconuts, and the production of husks and shells in large quantities that are not managed or openly burned pollutes the environment secondarily (Ahmad *et al.*, 2021; Adeleke *et al.*, 2025). These wastes can be converted into value-added adsorbents as a way to solve the emission control issue, as well as waste-to-wealth and Circular Economy approaches.

Although the combined use of agro-waste as an adsorbent in bioremediation is currently gaining prominence, one of the greatest constraints to its application in practice is the lack of knowledge on how production conditions affect adsorption

performance under conditions that closely mimic real engine exhaust conditions (Singh *et al.*, 2025). Some of the most important processing parameters which determine the formation of pore structure, surface chemistry, and adsorption capacity of ash-based adsorbent include carbonisation temperature and carbonisation time. Moreover, chemical activation by reagents such as phosphoric acid or potassium hydroxide can have a significant impact on the surface functionality and adsorption behaviour of the obtained materials. Nevertheless, the available literature tends to report inconsistent results because processing conditions were not optimised, gas tests carried out on a laboratory scale fail to represent real diesel exhaust compositions, or a lack of breakthrough analysis, which is necessary to analyse adsorbent durability and saturation behaviour under continuous-run conditions.

This research aims to systematically optimise the carbonisation temperature and duration, as well as the chemical activation conditions, of rice husk ash and coconut shell ash to maximise their adsorption efficiency and breakthrough time during mechanical use in diesel engine control systems for emission reduction. The research fills the knowledge gap regarding the combined and interactive effects of these parameters on adsorption performance metrics using a response surface approach and stringent statistical analysis. The results of this effort are anticipated to provide scientifically based design principles for creating high-performance and low-cost adsorbents suitable for real-world diesel emission control, particularly in environments with limited resources. The objective of this research is therefore to determine the influence of temperature and time of carbonisation in the presence of chemical activation on the adsorption properties and breakthrough time of rice husk ash and coconut shell ash as a mechanical method of controlling diesel engine emissions.

METHODOLOGY

In this study, the scientific research method of experimental and statistical optimization was used to determine the effect of carbonization temperature, carbonization duration, and chemical activity of rice husk ash (RHA) and coconut shell ash (CSA) on the adsorption properties of these materials in mechanical control of diesel engine emissions. The purpose of this method is to define optimal process conditions that will maximize the efficiency and breakthrough time of the adsorption process under realistic exposure to exhaust gases. The experimental variables were chosen based on the literature on the influence of pore growth, surface chemistry and adsorption behavior of carbonaceous materials. Agro-waste resources, i.e. rice husk and coconut husk, were collected from

local processing plants and treated through cleaning, drying, carbonization and chemical activation.

Table 1 shows the experimental factors and the levels as follows: the temperature of carbonisation (500-700 °C) and time of carbonisation (2-10 hours) were treated as numerical factors, whereas the activating reagent (phosphoric acid or potassium hydroxide) was treated as a categorical factor (coded as B). The RSM design was employed to systematically test the individual and interactive effects of these parameters, with fewer experimental runs.

The prepared adsorbents were tested in a mechanically integrated fixed-bed adsorption system attached to a diesel engine exhaust system of a mikano generator (MK-VII 60 kVA) and a heavy truck (Sinotruk HOWO 8×4 Tipper (12-wheel); Engine: WD615.47, Euro II diesel; Power: 371 HP (273 kW) at ~2000 rpm; Torque: ~1560 Nm at 1200-1500 rpm) as stated by Sinotruk (2025) The exhaust gas upstream and downstream of the adsorption chamber from engines were recorded using Gas Analyser (Kane (K) Automobile gas analyser, Aeroqual series 500 model gas analyser and H8 plus model gas analyser).

Table 1: Levels and Parameters

Numerical Factors		Levels			
Carbonisation temperature (°C)	500	550	600	650	700
Carbonisation Time (hours)	2	4	6	8	10
Categorical factors					
Reagent	Code		Level		
Phosphoric acid (H ₃ PO ₄)	B		1		2
Responses					
1	Adsorption Efficiency (%)				
2	Breakthrough Time (mins)				

The variables of response that were selected include adsorption efficiency (percentage) and breakthrough time (minutes) (Table 1), which are a combination of the effectiveness and stability of the adsorbents in the area of emission control.

■ Adsorption Efficiency

The performance measurement was based on adsorption efficiency to determine the capability of RHA and CSA to eliminate gaseous pollutants in diesel exhausts. This parameter represents the ratio of the decline in pollutant concentration caused by the adsorbent when the engine is under constant operating conditions. At the beginning of every experimental run, the concentration of pollutants at the inlet and outlet of the adsorption chamber was recorded once the engine had stabilised. The percentage of adsorption was determined by Equation 1 as the percentage of the inlet pollutant concentration and the outlet adsorption proportion:

$$\text{Adsorption Efficiency} = \frac{C_{in} - C_{out}}{C_{in}} \times 100\% \quad (1)$$

where: C_{in} is the Initial concentration of pollutant before adsorption (e.g., CO, NO_x, etc.) and C_{out} is the Final concentration of pollutant after adsorption.

Higher values of adsorption efficiency indicate high-performing pollutant removal. The physicochemical properties of the adsorbents that determine adsorption efficiency highly depend on carbonisation temperature, carbonisation time, and chemical activation.

Increased carbonisation temperatures tend to favour the formation of micropores and surface area whereas too high temperatures can result in the collapse of the pores and loss of adsorption sites. On the same note, phosphoric acid or potassium hydroxide chemical activation alters surface functional groups, which increases the degree of interaction between gases and solid, and boosts the activity of adsorption.

■ Breakthrough Time

The adsorption capacity as well as the working life of the adsorbents under continuous exposure to diesel exhaust was measured as the breakthrough time. It is a measure of the time within which the adsorbent remains active before saturation. In this experiment, the definition of breakthrough was the stage at which the level of outlet pollutant concentration became noticeable and steady, as a result of which the adsorbent ceased to absorb additional pollutants. It was recorded using a stopwatch

In every experimental run, continuous analysis was done on the outlet pollutant concentrations as the exhaust gas passed through the adsorption chamber. The breakthrough time, in minutes, was noted as the elapsed time since the initiation of adsorption to the beginning of the increase in concentration. Increased breakthrough times indicate increased adsorption capacity, better utilisation of the pores, and a higher adsorbent position, facilitating a more extended application in emission control.

Breakthrough behaviour is especially essential to real-life diesel emission control systems, as it defines the maintenance and the frequency of replacement for the adsorbent material (Oparanti *et al.*, 2025). It was anticipated that carbonisation conditions that would form well-developed networks of pores and chemically active surfaces would have longer breakthrough times.

The analysis of adsorption efficiency and breakthrough time, combined, thus provides a balanced evaluation of both the short-term and long-term performance of the adsorbent (Nurhayati *et al.*, 2026).

■ Data Analysis

Statistical analysis of experimental data from adsorption tests was performed to determine the effects of carbonisation temperature, carbonisation time, and chemical activation on adsorption efficiency and breakthrough time using response surface methodology (RSM). Analysis of variance (ANOVA) was used to identify the significance of each factor, quadratic and interaction at the 95% confidence level. The results of ANOVA, including F values and P values, were used to determine statistically significant terms in the model.

A quadratic response surface model with additional terms was fitted to the experimental data to establish predicted relationships between independent variables and responses. Model adequacy was assessed using coefficient of determination (R^2), adjusted R^2 and goodness-of-fit tests. Only significant terms were retained in the final model to increase predictive accuracy and reduce model complexity.

The optimisation was carried out using the desirability function method, with the aim of simultaneously maximising the adsorption efficiency and breakthrough time.

The optimal operating conditions predicted by the model were then validated through confirmatory experimental runs. This statistical framework ensured reliable interpretation of the results and robust optimization of the agricultural waste adsorbent for diesel engine emission control.

RESULTS AND DISCUSSION

This section summarises and explains the experimental findings of the response surface design research on how carbonisation temperature, carbonisation time, and chemical activation affect the adsorption behaviour of rice husk ash (RHA) and coconut shell ash (CSA).

The data obtained for the performance metrics, based on adsorption efficiency and breakthrough time, are presented in Table 2, which shows a Model F-value of 293.20, indicating that the model is statistically significant.

The A, B, C, A^2 are significant model terms at 95% level of confidence. The "Lack of Fit F-value" of 10.68 indicates that the Lack of Fit is statistically significant. There is only a 0.14% chance that a "Lack of Fit F-value" this large could occur due to noise.

■ Adsorption Efficiency

The adsorption efficiency values ranged between 38 and 76 per cent. The minimum efficiency of 38 was observed at 500 °C using Level 1 of reagent B, but the maximum efficiency of 76 was observed at 700 °C using Level 2 of reagent B. This broad spread indicates the high reliance of adsorption

efficiency on the conditions of carbonisation and chemical activation.

Table 2: Experimental Design

Carbonisation Temperature (°C)	Carbonisation time (hrs)	Phosphoric Acid	Adsorption Eff (%)	Breakthrough time (min)
1	650	6	Level 1 of B	58
2	600	5	Level 1 of B	52
3	600	3	Level 2 of B	60
4	600	5	Level 2 of B	65
5	550	4	Level 1 of B	45
6	600	3	Level 1 of B	48
7	650	4	Level 1 of B	56
8	700	5	Level 1 of B	62
9	600	5	Level 1 of B	52
10	550	6	Level 2 of B	59
11	600	5	Level 1 of B	52
12	600	5	Level 1 of B	52
13	600	5	Level 2 of B	66
14	600	7	Level 2 of B	69
15	600	5	Level 2 of B	65
16	550	4	Level 2 of B	57
17	650	6	Level 2 of B	73
18	500	5	Level 1 of B	38
19	600	5	Level 2 of B	66
20	550	6	Level 1 of B	46
21	600	7	Level 1 of B	58
22	600	5	Level 2 of B	65
23	500	5	Level 2 of B	50
24	650	4	Level 2 of B	70
25	600	5	Level 1 of B	52
26	700	5	Level 2 of B	76

Table 3: ANOVA of the Adsorption Efficiency

Source	Sum of Squares	df	Mean Square	F Value	p-value	
Model	2155.76	8	269.47	293.20	< 0.0001	significant
A–Carbonisation temp	937.50	1	937.50	1020.07	< 0.0001	
B–Carbonisation Time	88.17	1	88.17	95.93	< 0.0001	
C–Phosphoric acid (H ₂ PO ₄)	1111.54	1	1111.54	1209.44	< 0.0001	
AB	0.50	1	0.50	0.54	0.4708	
AC	2.67	1	2.67	2.90	0.1067	
BC	0.000	1	0.000	0.000	1.0000	
A ²	14.00	1	14.00	15.24	0.0011	
B ²	4.310E–003	1	4.310E–003	4.690E–003	0.9462	
Residual	15.62	17	0.92			
Lack of Fit	14.42	9	1.60	10.68	0.0014	significant
Pure Error	1.20	8	0.15			
Cor Total	2171.38	25				

The ANOVA results presented in Table 3 confirm that the model developed is statistically significant on adsorption efficiency. The value of model F293.20 and the p-value of less than 0.0001 indicate that the quadratic response surface model is appropriate for describing the experimental

results (Liu *et al.*, 2022). All of the individual linear terms, including carbonisation temperature (A), carbonisation time (B), and phosphoric acid activation (C) were significantly important ($p < 0.0001$), which indicates that the effect of each of the parameters alone is very strong on adsorption efficiency. The largest amount of squares was produced by chemical activation (factor C) and carbonisation temperature (factor A), indicating that surface chemistry modification and thermal treatment were the most significant factors determining adsorption performance.

This was also found to be significant ($p = 0.0011$) in the quadratic term of carbonisation temperature (A²), which indicated the relationship between temperature and adsorption efficiency was non-linear. This means that as the temperature increases and adsorption increases due to the better development of pores or an increase in surface area, excessively high temperatures could result in partial pore collapse or sintering, hence, reaching a limiting efficiency increase. Conversely, the interaction terms (AB, AC, and BC) t-tests were not significant; hence, the combined effects of the factors were less significant, as the factors had a significant effect on their own. These observations are further supported by the regression coefficients and confidence intervals presented in Table 4.

The coefficients A (+6.25), B (+1.92), and C (+6.54) are positive, which indicates that the temperature of carbonisation, the time of carbonisation, and the utilisation of chemical activation are all beneficial to the adsorption efficiency. The observed curvature in the response surface is a result of the negative coefficient of the quadratic temperature term (–0.55).

Table 4: Analysis of the Adsorption Efficiency

Factor	Coefficient Estimate	df	Standard Error	95% CI Low	95% CI High	VIF
Intercept	58.66	1	0.28	58.06	59.25	
A–Carbonisation temp	6.25	1	0.20	5.84	6.66	1.00
B–Carbonisation Time	1.92	1	0.20	1.50	2.33	1.00
C–Phosphoric acid (H ₂ PO ₄)	6.54	1	0.19	6.14	6.94	1.00
AB	0.25	1	0.34	–0.47	0.97	1.00
AC	0.33	1	0.20	–0.080	0.75	1.00
BC	0.000	1	0.20	–0.41	0.41	1.00
A ²	–0.55	1	0.14	–0.85	–0.25	1.09
B ²	9.698E–003	1	0.14	–0.29	0.31	1.09

The predictive model of adsorption efficiency developed after using a coded form and shown using Equation 2 has excellent goodness-of-fit statistics ($R^2 = 0.9928$, Adj $R^2 = 0.9894$, Pred $R^2 = 0.9720$), which means that it has a high predictive reliability.

The sufficiency of the model is graphically satisfied in Figure 1 in which the predicted values of adsorption efficiency are similar to the real values in the experiment, there is very little scatter in and around the parity line. This supports the fact that the model is healthy in predicting adsorption efficiency in the design space studied.

$$AE = 58.66 + 6.25A + 1.92B + 6.54C + 0.25AB + 0.33AC - 0.55A^2 + 0.009.69B^2 \quad (2)$$

(SD - 0.96, R2 - 0.9928,
Adj R2 - 0.9894, Pred R2 - 0.9720)

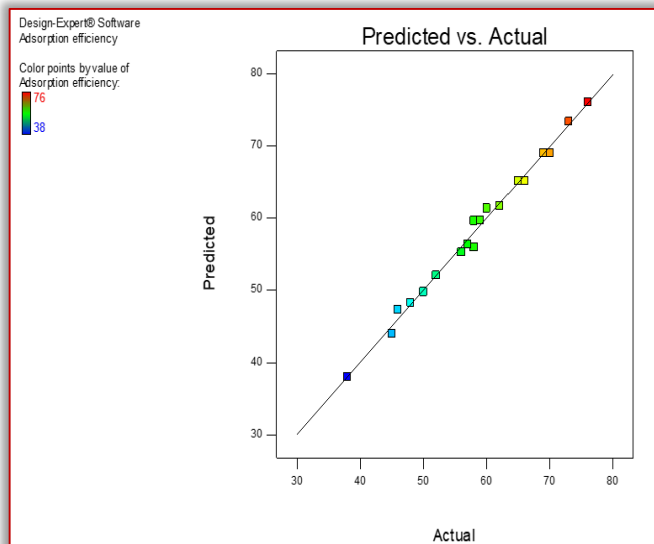


Figure 1: Predicted VS Actual Values of Adsorption Efficiency

Moreover, Figure 2 illustrates the combined effect of carbonisation time and temperature on the adsorption efficiency. The figure clearly indicates that the adsorption efficiency rises with the rise in temperature to the upper range, especially when it is coupled with moderate-moderate carbonisation times. This tendency can be attributed to the improvement of the pore structure, the increase in surface area, and the enhanced accessibility of adsorption sites.

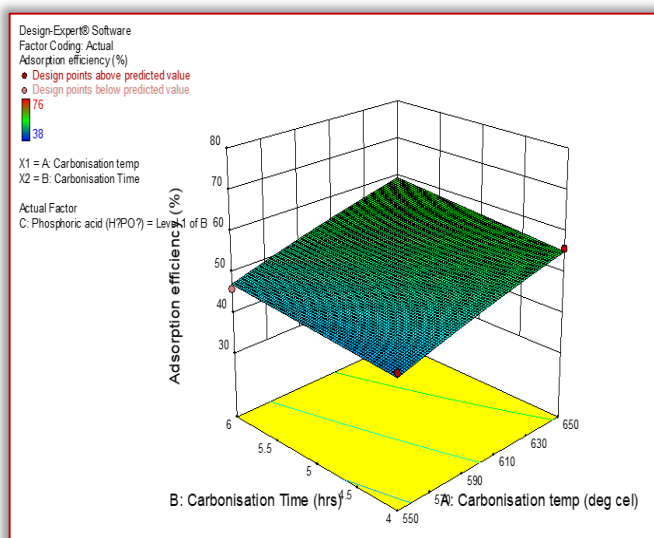


Figure 2: Effect of Carbonisation Temperature and Time on Adsorption Efficiency

The adsorption efficiency outcomes indicate that the adsorption was best optimised with an increased carbonisation temperature, a moderate to long carbonisation duration, and chemical activation with Level 2 of Phosphoric Acid (Barakat *et al.*, 2024; Hong *et al.*, 2025; Adegoke *et al.*, 2025). These results affirm that with appropriate optimisation, agro-waste-derived adsorbents are suitable in the control of diesel emissions (Subudhi *et al.*, 2025).

Breakthrough Time

The breakthrough time obtained in the experimental design ranged from 78 to 138 minutes. The shortest breakthrough time was recorded at a 500 °C carbonisation temperature using Level 1 of reagent B, indicating that the adsorbent is easily saturated under non-optimal processing conditions. The shortest breakthrough time (138 minutes) was achieved at 700 °C and Level 2 of reagent B, indicating increased adsorption capacity and long-term efficiency when exposed to continuous exhaust.

Table 5: ANOVA of the Breakthrough Time

Source	Sum of Squares	df	Mean Square	F Value	p-value Prob > F	
Model	12.24	8	1.53	202.84	< 0.0001	significant
A-Carbonisation temp	6.58	1	6.58	872.04	< 0.0001	
B-Carbonisation Time	0.80	1	0.80	105.75	< 0.0001	
C-Phosphoric acid (H ₃ PO ₄)	4.82	1	4.82	638.49	< 0.0001	
AB	4.423E-003	1	4.423E-003	0.59	0.4544	
AC	3.995E-003	1	3.995E-003	0.53	0.4767	
BC	0.018	1	0.018	2.43	0.1376	
A ²	0.017	1	0.017	2.25	0.1517	
B ²	8.654E-004	1	8.654E-004	0.11	0.7390	
Residual	0.13	17	7.545E-003			
Lack of Fit	0.11	9	0.012	4.34	0.0252	significant
Pure Error	0.022	8	2.723E-003			
Cor Total	12.37	25				

In Table 5, the ANOVA result for the breakthrough time demonstrates that the response surface model is statistically significant, as indicated by the model F-value and p-value of 202.84 and 0.0001, respectively. As with adsorption efficiency, the carbonisation temperature (A), carbonisation time (B), and chemical activation (C) linear terms were also found to be statistically significant, indicating that they had a strong impact on the adsorbent durability. The F-value of carbonisation temperature was the highest, implying that thermal treatment is a highly important factor in determining pore volume and adsorption capacity, which has a direct impact on breakthrough behaviour (Odeh *et al.*, 2025).

The breakthrough time model lack-of-fit was also significant ($p = 0.0252$) and there could be some variability unexplained, but the model is still reliable because of its high R^2 (0.9896), adjusted R^2 (0.9848) and predicted R^2 (0.9632). Such statistics indicate that the model accurately reflects the prevailing trends in the data and can be used with considerable certainty in prediction and optimisation (Yusuf *et al.*, 2025).

Table 6 presents regression coefficients indicating a positive linear dependence of temperature of carbonisation (+0.52), carbonisation time (+0.18), and chemical activation (+0.43) on breakthrough time. The findings support the hypothesis that an increase in these parameters delays adsorbent saturation by increasing pore volume, enhancing surface reactivity, and improving gas-solid contact. The carbonisation temperature's negative quadratic coefficient (-0.019) indicates a decrease in returns at excessively high temperatures, which is attributed to the trend of decreasing adsorption efficiency (Ibrahim *et al.*, 2025).

Table 6: Analysis of the Breakthrough Time

Factor	Coefficient Estimate	df	Standard Error	95% CI Low	95% CI High	VIF
Intercept	10.35	1	0.026	10.30	10.41	
A–Carbonisation temp	0.52	1	0.018	0.49	0.56	1.00
B–Carbonisation Time	0.18	1	0.018	0.14	0.22	1.00
C–Phosphoric acid (H ₃ PO ₄)	0.43	1	0.017	0.39	0.47	1.00
AB	-0.024	1	0.031	-0.088	0.041	1.00
AC	0.013	1	0.018	-0.025	0.050	1.00
BC	0.028	1	0.018	-9.783E-003	0.065	1.00
A ²	-0.019	1	0.013	-0.046	7.810E-003	1.09
B ²	4.346E-003	1	0.013	-0.023	0.031	1.09

The predictive breakthrough model (Equation 3) exhibits a good match between the predicted and experimental values, as illustrated in Figure 3. The close deviation of the data points near the parity line verifies that there is a low prediction error and the accuracy of the model is high.

$$BT = 10.35 + 0.52A + 0.18B + 0.43C - 0.024AB + 0.013AC + 0.028BC - 0.019A^2 + 0.004346B^2 \quad (3)$$

(SD - 0.087, R² - 0.9896,
Adj R² - 0.9848, Pred R² - 0.9632)

Figure 4 depicts the overall effect of carbonisation temperature and time on the breakthrough time with a clear indication of the duration of the breakthrough increasing with the increase in temperatures and with the increase of carbonisation time.

The findings in breakthrough times show that optimised carbonisation and activation conditions can be important in prolonging the adsorbent's service life (Ogedengbe *et al.*, 2025). On the one hand, in combination with adsorption efficiency, the results will confirm that agro-waste-derived RHA and CSA can be applied as viable and sustainable adsorbents to mechanically control diesel engine emissions (Ravi, 2023; Vanshpati and Tiwari, 2025).

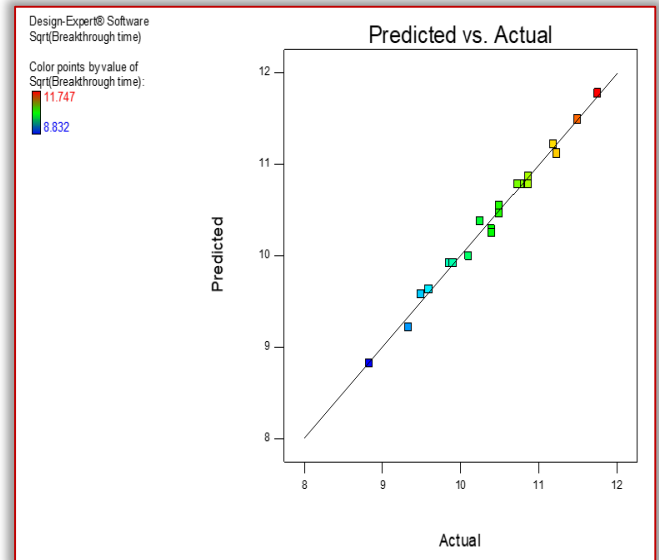


Figure 3: Predicted Vs Actual Values of the Breakthrough Time

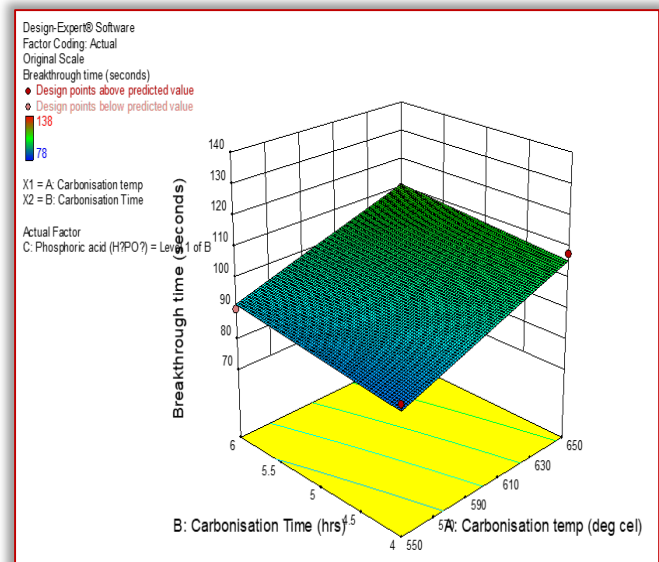


Figure 4: Effect of Carbonisation Temperature and Time on Breakthrough Time

CONCLUSION

This study investigated the effects of carbonisation temperature, carbonisation time, and chemical activation on the adsorption characteristics of rice husk ash and coconut shell ash, aiming to mechanically control the emission of diesel engines. The results have shown beyond a reasonable doubt that the processing environment of agro-waste-based adsorbents is a determining factor in the efficiency of agro-waste-based

adsorbents in pollutant removal and their operational lifespan.

The experimental findings indicated that the adsorption efficiency and breakthrough time increased with an increase in the temperature of carbonisation as well as chemical activation (especially at high temperatures). High carbonisation temperatures and Level 2 of the activating reagent yielded the highest adsorption efficiency of 76 per cent and the longest breakthrough time of 138 minutes, demonstrating the feasibility of surface modification and improved pore growth. Carbonisation time was also found to be positive, albeit to a lesser extent, compared to temperature and chemical activation. The non-linear nature of behaviour with an increase in temperature means that there is an optimum level of carbonisation where an additional increase in temperature may not result in performance increases which are proportional.

The soundness of the developed models was statistically verified using response surface methodology and ANOVA; the coefficients of determination were high, indicating that the models are sufficiently predictive. The fact that the predicted and the experimental values are close also confirms the reliability of the optimisation method used in this paper. The prevalence of linear effects as compared to interaction terms indicates that the performance of adsorption can be easily manipulated by the appropriate choice of the individual processing parameters.

The research as a whole identifies rice husk ash and coconut shell ash as potentially feasible, inexpensive, and sustainable adsorbents for managing diesel emissions in the proper state. These agro-wastes are also useful in the sense that their utilisation helps to reduce emissions and also facilitates waste valorisation and environmental sustainability. The study gave a technical foundation for the development of effective adsorption-based emission control systems that can be applicable in resource-constrained environments.

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