



Adsorption of methylene blue from aqueous solutions using activated carbon derived from sand box seed shell (*Hura crepitans*): Isothermal, thermodynamic, and kinetic studies

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Received: 19 September 2025 / Accepted: 3 December 2025 / Published online: 26 January 2026
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Abstract

This study explores the use of activated carbon derived from sandbox seed shell (SSS) for the removal of methylene blue (MB⁺) from aqueous solutions. The SSS was impregnated individually with KOH and H₃PO₄ at 85% of the biomass weight using a 1:3 solution ratio to enhance porosity and surface functionality. Carbonisation was performed at 700 °C under nitrogen following a 24-h soaking period. Characterisation using SEM, FTIR, EDS, BET, XRD, TGA, XRF, and PZC verified notable chemical and structural changes. BET surface areas increased from 209.454 m²/g (RSSS) to 543.378 m²/g (SSS-KOHAC) and 433.493 m²/g (SSS-H₃PO₄AC), with corresponding iodine numbers of 482.334, 977.361, and 1142.37 mg/g, respectively. Batch adsorption experiments demonstrated that the removal efficiency of MB⁺ increased with rising pH and peaked near neutral values, consistent with PZC measurements of 6.9, 7.12, and 7.83 for SSS-KOHAC, SSS-H₃PO₄AC, and RSSS, respectively. Thermodynamic modelling indicated spontaneous adsorption for activated samples ($\Delta G^\circ < 0$), whereas RSSS exhibited non-spontaneous behaviour ($\Delta G^\circ > 0$) at elevated temperatures. SSS-H₃PO₄AC showed the highest adsorption energy and selectivity, attributed to phosphate-derived surface groups. Adsorption followed favourable isotherm and kinetic models, confirming the potential of chemically activated biomass as an efficient, low-cost adsorbent for dye removal in water treatment applications.

Keywords Adsorption · Biomass · Wastewater · Sandbox seed shell · Activated carbon · Methylene blue

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Abbreviations

AC	Activated carbon
MB	Methylene blue
SSS	Sandbox seed shell
RSSS	Raw sandbox seed shell
FTIR	Fourier transform infrared spectroscopy
SEM	Scanning electron microscopy
MC	Moisture content
X_{ash}	Ash content
FC	Fixed carbon
Y_{AC}	Yield of activated carbon
XRD	X-ray diffraction
XRF	X-ray fluorescence
TGA	Thermogravimetric Analysis
BET	Brunauer Emmett–Teller
PZC	Point of zero charge
A	Adsorption potentials
KOH	Potassium hydroxide
H_3PO_4	Phosphoric acid
EDS	Energy-dispersive X-ray spectroscopy
ΔH_X	Isosteric heat of adsorption
R	Universal gas constant
ΔH°	Change in enthalpy
ΔS°	Change in entropy
ΔG°	Change in Gibbs free energy
FO	First order
PSO	Pseudo-second-order model
IPD	Intra-particle diffusion model
TI	Temkin isotherm
EI	Elovich isotherm
DRI	Dubinin Radushkevich
FI	Freundlich isotherm
LI	Langmuir isotherm
SSS- H_3PO_4 AC	Sandbox seed shell phosphoric acid activated carbon
SSS-KOHAC	Sandbox seed shell potassium hydroxide activated carbon
R^2	Correlation coefficient

1 Introduction

Industrial effluent released by processes such as paper, leather, rubber, food and medicine, textile, and printing is characterised by the presence of numerous synthetic dye constituents (Alsukaibi 2022). Commercial wastewater can contain many different chemicals, biochemicals, and molecules that require oxygen. The oxygen demand of dyed wastewater is high, both chemically and biologically (Chaturvedi et al. 2025). The disposal of wastewater containing dyes can lead to several illnesses, including skin cancer, in addition to contaminating the environment and even the ocean (Periyasamy 2025; Sudarshan et al. 2023). The reuse

of such wastewater reduces manufacturing efficiency, contaminates products, and diminishes material quality. Membrane filtration, ozonation, advanced oxidation, biosorption, coagulation and flocculation, adsorption, and liquid–liquid extraction are among the physical, chemical, and biological treatment techniques used to treat dye-contaminated wastewater. Adsorption is considered the most effective treatment method for wastewater containing dyes because of its many advantages, including low cost, ease of use, simplicity in design, and resistance to harmful substances (Hu & Hao 2025). The aim of research is to provide an adsorption material that enables the most efficient adsorption process at the lowest possible production cost (Topare et al. 2024). The desire to improve the accessibility, availability, and affordability of materials has been stimulated by the use of inexpensive, renewable sorbents (Aragaw & Bogale 2021; Liu et al. 2024). These sorbents are derived from natural resources and agricultural waste, thereby reducing pollution and waste generation in the environment. Activated carbon (AC) is a solid carbonaceous material manufactured through thermal or thermochemical processes involving coal or biomass (Ukanwa et al. 2019). AC is distinguished by a highly developed pore structure, large surface area, amphoteric properties that enable electron conduction, and high adsorption capacity (Ganjoo et al. 2023). AC is particularly effective in addressing undesirable air and water pollution. Such environmental concerns arise from activities including coal combustion and improper disposal of household and commercial waste (Spencer et al. 2024). The food processing, brewing, textile, and chemical industries also emit effluents (Sathya et al. 2022). Wastewater can be treated using AC due to its broad scope of application and adsorbate production (Raheel et al. 2024). AC is a versatile adsorbent substance comprising up to 90% carbon (Heidarinejad et al. 2020). Two of its greatest attributes are its high surface area and porosity. The carbon structure contains crucial functional groups that play a significant role in pollutant adsorption, including carboxyl, carbonyl, phenol, lactone, and quinone (Yang et al. 2023). Other chemical atoms or functional groups such as nitrogen, sulphur, oxygen, and hydrogen are also present in AC (Ganjoo et al. 2023). These functional groups are introduced through activation, precursor selection, and thermal purification, which confer the unique adsorption properties of activated carbon. AC contains smaller pores with low volumes that enable adsorption processes distinct from other chemical reactions (Serafin & Dziejarski 2023a, b). Unactivated charcoal has a surface area of between 2.0 and 5.0 m²/g, which can increase to 1000 m²/g upon activation (Ganjoo et al. 2023). The high surface area of activated carbon enables it to capture and retain liquid and gaseous molecules, making it a highly efficient adsorbent (Abd et al. 2021). The material can be produced from carbon-rich sources such as coal, wood, coconut shells, and agricultural waste.

Carbonisation and activation processes allow producers to tailor the surface chemistry and pore structure of activated carbon to industrial requirements (de Oliveira et al. 2021; Thommes et al. 2011). There are two common methods of producing activated carbon: physical activation and chemical activation. Physical activation has two major disadvantages compared with chemical activation. It requires higher temperatures, whereas chemical activation is conducted at lower temperatures. Chemical activation also produces excellent yields because it does not require char burn-off. Common chemicals used in chemical activation include ZnCl_2 , H_3PO_4 , K_2CO_3 , KOH , CaCl_2 , Na_2SO_4 , and H_2SO_4 (Üner & Bayrak 2018). Numerous studies have examined how chemical agents influence the characteristics of porous carbon. The use of phosphoric acid (H_3PO_4) and potassium hydroxide (KOH), an acid and a base that promote pore incorporation and carbon formation, respectively, provides a notable example of the chemical activation process for creating porous carbon. Agricultural wastes are the most readily available bio-adsorbents because they are renewable, environmentally friendly, and highly effective at removing colour from aqueous solutions. The adsorption process has not yet been carried out using sandbox seed shell (SSS), even though many adsorbents have recently been employed to create more affordable and accessible alternatives. The granular form, excellent mechanical integrity, and good chemical stability of SSS make it suitable for adsorption-based methylene blue removal from water. Sandbox seed shells represent a reasonably priced option for the production of activated carbon, as they are a free and inexpensive raw material (Ganjoo et al. 2023). The increased surface area of activated carbon derived from seed shells generally enhances its adsorption capacity (Heidarinejad et al. 2020). The utilisation of agricultural waste, such as crushed seed shells, to produce activated carbon promotes environmental sustainability by reducing waste quantities and recycling biomass (Ahmad et al. 2021; Ukanwa et al. 2019). Sandbox seed shells can be used to produce activated carbon that is beneficial for air filtration and water purification due to its ability to adsorb contaminants in both media (Fatima et al. 2023; Jaya Rajan & Indira Anish 2024; Nguyen et al. 2023). Activated carbon derived from sandbox seed shells is applied across various industries (William et al. 2019; Pagketanang et al. 2015; Mawlood et al. 2024).

The novel feature of this work is the use of sandbox seed shell, an underutilised waste biomaterial, to produce activated carbon, which is subsequently employed to remove methylene blue from aqueous solution. This study utilises both base (KOH) and acid (H_3PO_4) activators for the adsorption of methylene blue. The properties of the generated activated carbon were evaluated using proximate analysis. The effects of various factors on the removal of a basic dye from aqueous solution were assessed through batch testing.

Process variables included temperature, agitation speed, contact time, adsorbent dosage, pH, and initial dye concentration. Adsorption isotherms, kinetics, thermodynamics, and characterisation were also established and examined.

2 Materials and methods

2.1 Raw material, equipment and apparatus used

Sandbox seed shells were collected from the area around Federal Polytechnic Nekede in Imo State, Nigeria. Potassium hydroxide (KOH), phosphoric acid (H_3PO_4), and methylene blue were supplied by the Chemical Engineering Laboratory at Ahmadu Bello University, Zaria. The necessary solutions were prepared using distilled water, and all chemicals employed in the study were of analytical grade. The point of zero charge (PZC) was determined using HCl and NaCl , with pH values adjusted between 2 and 10. The tools and equipment used in this study included a weighing balance (OHAUS DV215CD), analytical balance (DRAWELL), planetary ball mill (OVERUFLD S/No: 810), jaw crusher (RETSCH BB2), magnetic stirrer, analytical hot plate/stirrer, volumetric flasks, funnels, measuring flasks, spatulas, droppers, crucibles, plastic brushes, and a vertical reactor.

2.2 Impregnating agent [potassium hydroxide (KOH) and Phosphoric acid (H_3PO_4)]

The efficacy of the activation process is greatly influenced by the choice of impregnating agent, which is crucial in the production of activated carbon (Kiani et al. 2021). Sandbox seed shell was used as a raw material to prepare activated carbon. KOH and H_3PO_4 were purchased and employed as activators for the adsorption of methylene blue in aqueous solution. The activators were supplied by the Chemical Engineering Laboratory at Ahmadu Bello University, Zaria.

According to Sopanrao et al. (2024), H_3PO_4 is comparatively less toxic than other chemical activators owing to its high surface area, porosity, which enhances adsorption capacity, and good chemical stability. H_3PO_4 and KOH have been utilised as impregnation agents in various agricultural wastes to address environmental degradation, particularly water rehabilitation, and both have demonstrated outstanding adsorption capacities. KOH effectively activates carbon material when used alone at low temperatures (Wang & Kaskel 2012). It has been applied in the adsorption of methylene blue due to its high porosity, which also boosts its adsorption capacity (Jaya Rajan & Indira Anish, 2024; Khu Leo Van et al. 2019; Srenscek-Nazzal et al. 2016).

An enhanced activated carbon surface area has also been shown to improve the effectiveness of KOH , thereby contributing to adsorption (Zhao et al. 2023). The activation

temperature required is typically lower than the 1327 °C boiling point of KOH (Heidarinejad et al. 2020). Moreover, KOH saturates its precursor and does not evaporate completely, which qualifies it as an excellent activator (Sun et al. 2015). KOH is also less expensive, less corrosive, and has a smaller environmental impact.

2.3 Adsorbate (methylene blue)

The main dye used in this study was methylene blue (MB), which was obtained from the Chemical Engineering Laboratory of Ahmadu Bello University, Zaria, Nigeria. Methylene blue was selected owing to its high affinity for solid materials and its effective application in the characterisation of adsorptive materials (Hameed 2009; Amode et al. 2016). Table 1 presents the physicochemical characteristics of methylene blue, and Fig. 1 illustrates its chemical structure.

2.4 Preparation and carbonization of sandbox seed shell

The sandbox seed shell (SSS) used in this study was collected from the area around Federal Polytechnic Nekede in Imo State, Nigeria. The SSS was first washed with tap water to remove dust, debris, and other contaminants, then thoroughly cleaned with hot distilled water and dehydrated for 24 h at 105 °C to eliminate impurities (Fito et al. 2023). The dried SSS was crushed with a jaw crusher for 30 min to break it into smaller pieces. The finely crushed particles were sun-dried for six hours to further reduce moisture content. These particles were then sieved through a 6 mm mesh to obtain fine particles of uniform size (King et al. 2024). The sieved SSS was stored in an airtight container to prevent moisture absorption and fungal growth, which could reduce particle performance.

The SSS was impregnated separately with KOH and H₃PO₄, each at 85% of the biomass weight, using a 1:3 solution ratio, and soaked for 24 h (Yirdaw et al. 2023). The impregnated samples were air-dried at room

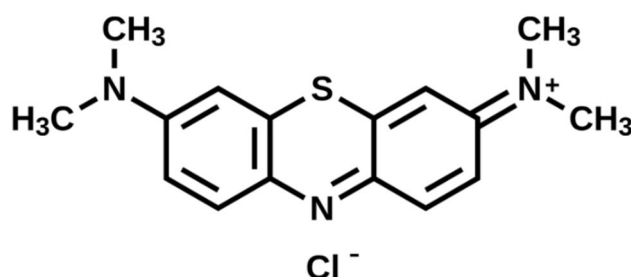


Fig. 1 Chemical Structure of methylene blue dye (Amode et al. 2016)

temperature before being transferred to a reactor and placed in a tube furnace. Carbonisation was carried out at 700 °C in a nitrogen atmosphere for one hour (heating rate: 10 °C/min; nitrogen flow rate: 1000 cm³/min). The dried samples at room temperature ensured uniform dispersion of the impregnating agents, preventing clumping and enabling consistent activation. This drying step enhanced the porous structure, maximising adsorption efficiency by ensuring complete interaction of the agents during high-temperature activation.

After heating, the samples were cooled to room temperature and thoroughly washed with distilled water to remove residual salts and contaminants, yielding a neutral pH of 7. Activated carbon was produced using the same procedure for both base and acid activators.

2.5 Proximate analysis

Proximate analysis enables the evaluation of activated carbon's moisture content, ash content, volatile matter, and fixed carbon. The analysis was conducted in accordance with ASTM International standards for moisture content (ASTM D5142), ash content (ASTM D3174), volatile matter (ASTM D872), and fixed carbon (ASTM D3175) (Asadu et al. 2021a, b; Ndecky et al. 2022).

Table 1 Physio-chemical properties of Methylene blue

Parameter	Value
Molecular Formula	$C_{16}H_{18}ClN_3S$
Water Solubility	43 g/L at 25 °C
Molecular Weight of Methylene blue	319.85 g/mol
Melting Point of Methylene blue	100 to 110 °C
Adsorption Maximum	663 nm (visible spectrum) in water; shifts in different solvents
Molar Absorptivity (ϵ) ($Lmol^{-1}cm^{-1}$)	8.7×10^{-4}
pK_a	Approx. 3.8
Log P (octanol/water)	-1.85, indicating hydrophilicity
Reduction Potential	+0.011 V vs. standard hydrogen electrode (SHE)

2.5.1 Moisture content

The moisture content of the SSS activated carbon was determined by heating one gram of the sample in a crucible for 24 h at 105 °C in accordance with ASTM D5142. The sample was then cooled in a desiccator at room temperature. The mass loss was recorded, and the moisture content was calculated using Eq. 1.

$$\text{Moisture content (MC)} = \frac{W_1 - W_2}{W_1} \times 100\% \quad (1)$$

The weights of the crucible and sample before and after drying are denoted by W_1 and W_2 , respectively, and the moisture content of the adsorbent is denoted by MC.

2.5.2 Volatile matter

The moisture content of the activated carbon was determined by heating one gram of the adsorbent in a crucible at 800 °C for eight minutes in a muffle furnace, in accordance with ASTM D872. The volatile matter content of the SSS adsorbent was then calculated using Eq. 2.

$$\text{Volatile matter (VM)} = \frac{W_1 - W_2}{W_1} \quad (2)$$

The weights of the crucible and adsorbent before and after ignition are denoted by W_1 and W_2 , respectively, and the volatile matter content of the adsorbent is denoted by VM.

2.5.3 The ash content

The ash content of the activated carbon was determined by heating a crucible containing one gram of SSS activated carbon at 750 °C for four hours in a muffle furnace to remove all organic matter. The sample was then cooled in a desiccator

at room temperature, and the ash content was calculated using Eq. 3.

$$\text{Ash content (X}_{\text{ash}}) = \frac{\text{Weight before ignition (W}_2)}{\text{Weight after ignition (W}_1)} \times 100\% \quad (3)$$

X_{ash} is ash content of the adsorbent, W_1 is the weight before ignition, and W_2 indicates the weight after ignition.

2.5.4 Fixed carbon

Fixed carbon (FC) represents the proportion of carbon present in activated carbon. The surface area and pore structure suitable for adsorption increase with the amount of fixed carbon. The fixed carbon content was calculated using Eq. 4 (He et al. 2023).

$$\text{Fixed carbon FC (\%)} = 100 - (\text{MC} + \text{VM} + X_{\text{ash}}) \quad (4)$$

FC is the fixed carbon, MC is the moisture content, VM is the volatile matter and.

X_{ash} is the ash content.

2.6 Adsorbent characterization

The main objective of adsorbent characterisation is to understand the surface chemistry that governs interactions with adsorbates. Characterisation identifies the functional groups, surface charge behaviour, elemental composition, and chemical stability that influence adsorption processes such as complexation, electrostatic attraction, and ion exchange. The categories, techniques, and equipment models employed for characterisation are presented in Table 2.

The iodine number was determined using the ASTM D4607 method as shown in Eq. 5.

Table 2 Characterization categories and technique used

Categories	Characterization technique	Model, Manufacturer & Country
Surface chemistry analysis	Fourier transform infrared spectroscopy	Cary 630 FTIR, Agilent Technologies, USA
	X-ray diffraction studies	Rigaku Mini-Flex X-ray diffraction (XRD) system, Rigaku Corporation, Japan
Thermal stability analysis	Thermogravimetric analysis	TGA 4000 by Perkin Elmer Netherlands
Structural analysis	Scanning electron microscopy	Zeiss Sigma 300 VP, Carl Zeiss Microscopy GmbH, Germany
	Brunauer–Emmett–Teller analysis	Quantachrome NOVA (e-series) gas sorption analyzer. 4-station NOVA 4200e running NovaWin + v11.03, Anton Paar QuantaTec Inc. USA
Elemental analysis	Energy-dispersive X-ray spectroscopy	Zeiss Sigma 300 VP, Carl Zeiss Microscopy GmbH, Germany
	X-ray fluorescence	Xenometrix Genius IF X-ray fluorescence (XRF) spectrometer, Xenometrix Ltd, Israel
Solution chemistry analysis	pH measurement	Hannah Pocket-sized pH Meter

$$\frac{X}{M}(\text{mg/g}) = \frac{(V_b - V_s) \times N \times 126.93}{W} \quad (5)$$

V_b is the titration value of blank (mL), V_s is the titration value of sample (mL), w is the weight of sample, N is normality of sodium thiosulfate solution, 126.9 is the equivalent weight of iodine (g/mol).

2.7 Batch adsorption

The effects of contact time, temperature, pH, dosage, and concentration on the adsorption of MB were examined in batch adsorption experiments using a stock solution of 3000 mg/L MB, prepared by dissolving the supplied dye in an appropriate volume of distilled water (Baloo et al. 2021). The experiments involved agitating a pre-weighed amount of adsorbent (0.4 g/L of SSS activator) at 200 rpm in a 500 mL beaker. The extent of adsorption depends on the mixing and interactions between the adsorbent and the adsorbate, and effective adsorption requires adequate mixing and contact, achieved by maintaining a constant agitation speed. Neutral pH was used as a reference point to control the adsorption process. All experiments were conducted at room temperature to ensure consistency and reproducibility. After agitation, the samples were immediately filtered using 0.45 μm Whatman filters and analysed spectrophotometrically at 663 nm (the absorption maximum of MB) with a Hach UV–Visible spectrophotometer (model DR 2800) to determine the residual dye concentration (Zazouli et al. 2016; Reçber 2022). Laboratory-prepared MB solutions in distilled water were used for all thermodynamic and temperature-effect adsorption experiments. The temperature range of 30–70 °C was selected to encompass both normal laboratory conditions and moderately elevated temperatures typical of industrial operations or warm effluents (Pathania et al. 2017; Ahmad et al. 2022). This range allowed investigation of adsorption behaviour under different thermal conditions, reliable determination of thermodynamic parameters (ΔH° , ΔS° , ΔG°), and assessment of adsorbent stability and pore accessibility under thermal stress (Tseng et al. 2022). Experiments conducted within these limits enabled differentiation between endothermic and exothermic processes and determination of whether changes in removal efficiency were due to increased diffusion, altered surface energetics, or desorption effects (Plaza et al. 2016). The parameters and values used for the batch adsorption experiments are presented in Table 3.

The adsorption capacity (q_e) was calculated using Eq. 6, and the percentage of methylene blue absorbed by the adsorbents was determined using Eq. 7.

Table 3 Parameters and values used for Batch adsorption

Parameters	Values				
Adsorption dosage (g/L)	0.2	0.4	0.6	0.8	1
Initial concentration of adsorbate (mg/L)	200	400	600	800	1000
Contact time (mins)	20	30	40	50	60
pH solution	2	4	6	8	10
Temperature (°C)	30	40	50	60	70

$$q_e = \frac{(C_0 - C_e) \times V}{m} \quad (6)$$

$$\% \text{ removal } (\%R) = \frac{(C_0 - C_t)}{C_0} \times 100 \quad (7)$$

q_e is the adsorption capacity (mg/g), C_0 is the initial concentration of MB at start time (mg/L), C_e is the equilibrium concentration of MB or pollutant concentration at a required time (mg/L), V is the volume of the MB solution (L), m is the mass of activated carbon (g).

2.8 Point zero charge analysis (PZC)

The method developed by Ogbodo et al. (2021) was employed to determine the point of zero charge (PZC) of activated carbon derived from SSS. A series of 0.1 mol/dm³ NaCl solutions were prepared in Erlenmeyer flasks, and 0.1 g of activated carbon was added to each 50 mL NaCl solution (Jedynak & Charnas 2024). The initial pH of the mixtures was recorded without agitation, after which each batch was adjusted with 0.1 mol/dm³ HCl or 0.1 mol/dm³ NaOH to obtain pH values between 2 and 10 (Nwosu et al. 2018). The mixtures were shaken for 24 h at 500 rpm using a rotating shaker (BA 200N) to ensure equilibrium (Ogbodo et al. 2021). The shaking rate of 500 rpm, recommended by Usman et al. (2012) and Asadu et al. (2021a, b), ensured complete suspension of carbon particles and rapid equilibration of the suspension pH. This confirmed that the computed PZC represented a true equilibrium point without mass-transfer limitations. Preliminary kinetic studies indicated that the pH stabilised after approximately 20 h; therefore, a contact time of 24 h at 500 rpm was adopted to guarantee equilibrium (Liyanaarachchi et al. 2023). The final pH of each solution was measured once equilibrium was reached. The pH at which the initial and final pH values are equal is known as the PZC. An initial pH graph was plotted against the initial and final pH difference [pH_i vs ($pH_f - pH_i$)] (Lim et al. 2013). The PZC is the point at which the curve crosses the line where the initial and final pH values are equal. The

point of zero charge was determined for both the raw and activated samples.

2.9 Adsorption thermodynamic

The evaluation of thermodynamic parameters is facilitated by the temperature dependence of adsorption. Adsorption tests employ thermodynamics to assess the feasibility and spontaneity of adsorption processes. Consequently, thermodynamic properties are derived from experimental adsorption data. The thermodynamic evaluation was carried out using the methodology outlined by Thompson et al. (2020) and Rauf et al. (2022). Data generated from the effects of temperature on MB adsorption onto RSSS, SSS-KOHAC, and SSS-H₃PO₄AC were used for thermodynamic modelling. Thermodynamic parameters such as ΔH , ΔS , and ΔG were obtained by applying the Van't Hoff equation to the collected data at temperatures of 303, 313, 323, 333, and 343 K. Accordingly, the thermodynamic parameters of change in enthalpy (ΔH), change in entropy (ΔS), and change in Gibbs free energy (ΔG) were determined using Eqs. 8–13.

$$\ln K_d = -\frac{\Delta G}{RT} = -\frac{\Delta H}{RT} + \frac{\Delta S}{R} \quad (8)$$

$$\Delta G = -RT \ln K_d \quad (9)$$

$$K_d = \frac{q_e}{C_e} \quad (10)$$

$$\Delta G = \Delta H - T \Delta S \quad (11)$$

K_d is the equilibrium constant, R is the universal gas constant (8.314 J/mol/K), T is the solution temperature (K), q_e is the equilibrium adsorption capacity (mg/g), C_e equilibrium concentration. The values of ΔH and ΔS can be determined respectively from the slope and intercept of the plot of $\ln K_d$ vs $1/T$, Slope = $-\Delta H/R$, and intercept = $\Delta S/R$.

Isosteric heat of adsorption (ΔH_x) The isosteric heat of adsorption is defined as the ratio of the infinitesimal change in the amount adsorbed at constant temperature to the infinitesimal change in the adsorbate enthalpy (Nuhnen & Janiak 2020; Tun & Chen 2021). This parameter, which is essential for characterising adsorption processes, is frequently calculated using the Clausius–Clapeyron equation, as shown in Eq. 12 (Park et al. 2014; Solovtsova et al. 2023).

$$\frac{d(\ln C_e)}{dT} = -\frac{\Delta H_x}{RT^2} \quad (12)$$

C_e equilibrium concentration, R is the universal gas constant (8.314 J/mol/K), T is the solution temperature

(K). Plot the natural logarithm of the equilibrium concentration ($\ln C_e$) against the inverse of the temperature ($1/T$). Slope = $-\Delta H_x/R$

Adsorption Potential (A) The movement of an adsorbate molecule from solution to the surface of an adsorbent during the adsorption process generates a chemical potential known as the adsorption potential. This parameter determines the ability of an adsorbent to adsorb molecules at a specific temperature. The adsorption potential (A) was calculated using Eq. 13 (Ebelegi et al. 2020).

$$A = -RT \ln \frac{C_o}{C_e} \quad (13)$$

C_o and C_e are the initial and equilibrium concentration of the adsorbate solution (mg/L), R is the universal gas constant (8.314 J/mol/K), T is the absolute temperature at which the sorption occurred (K).

2.10 Adsorption isotherms

Adsorption isotherms are used to analyse the surface properties of adsorbents and to describe the equilibrium between pollutants and adsorbent materials, taking into account the influence of temperature on adsorption systems. They are employed to determine the optimal performance of the system. The development of large-scale adsorption systems and the optimisation of adsorption processes for reliable performance evaluation depend heavily on isotherm models, as presented in Table 4. The interaction between the adsorbent and adsorbate at equilibrium is assessed using basic isothermal models, including the Freundlich, Temkin, Elovich, Dubinin–Radushkevich, and Langmuir models. In this study, isothermal experiments were conducted to determine how the concentration of the adsorbate influenced the amount of methylene blue adsorbed from aqueous solution.

2.11 Adsorption kinetics

Adsorption kinetics examines the line or curve that represents the rate at which a solute is released or retained at the solid–phase interface from an aqueous solution, while accounting for parameters such as flow rate, pH, adsorption temperature, and dose (William Kajjumba et al. 2019). The rate of sorption over time is used to evaluate the adsorption kinetics of adsorbates on the adsorbent. This assessment provides insight into the process's viability and yields valuable information about the adsorption mechanism (Baba et al. 2023). This method is employed to determine the efficacy of the adsorption process over time. Selecting the most appropriate kinetic model is essential to

Table 4 Isotherm model equations

Isothermal model	Equation		Plot	Reference
	Linear	Non-Linear		
LANGMUIR	$\frac{1}{q_e} = \frac{1}{Q_m K_L C_e} + \frac{1}{Q_m}$	$q_e = \frac{Q_m K_L C_e}{1 + K_L C_e}$	<i>Plot</i> $\frac{1}{q_e}$ vs. $\frac{1}{C_e}$ The slop and the intercept give the Langmuir constant Q_m and K_L	Kuang et al. (2020), Amode et al. (2016), Chang et al. (2020), Liu & Cao (2023), Abin-Bazaine et al. (2022), Sariana Sarana Miri & Narimo, (2022), Erdem et al. (2016)
DUBININ RADUSHKEVICH	$\ln q_e = \ln q_m - \beta \epsilon^2$ where : $\epsilon = RT \ln(1 + 1/C_e)$	$q_e = q_m \exp(-\beta \epsilon^2)$	<i>Plot</i> $\ln q_e$ vs. ϵ^2 The slop and the intercept give the D-R constant q_m and β	Asadu et al. (2021a, b), Abdelnaeim et al. (2016), Acosta et al. (2016),
FREUNDLICH	$\log q_e = \log K_f + \frac{1}{n} \log C_e$	$q_e = K_f C_e^{1/n}$	<i>Plot</i> $\log q_e$ vs. $\log C_e$ The slop and the intercept give the Freundlich constant K_f and n	Rashidi et al. (2016), Zaira Zaman Chowdhury (2011), King et al. (2024), Mabuza et al. (2022),
TEMKIN	$q_e = B \ln A + \frac{B}{b} \ln C_e$ where : $B = \frac{RT}{b}$	$q_e = \frac{RT}{b} \ln(ACe)$	<i>Plot</i> q_e vs. $\ln C_e$ The slop and the intercept give the Temkin constant A and B	Serafin & Dziejarski (2023a, b), Abbas & Ahmed (2016), Gautam et al. (2023), Rajabi et al. (2023)
ELOVICH	$\ln \frac{q_e}{C_e} = \ln K_e q_m - \frac{q_e}{q_m}$	$q_e = C_e K_e q_m e^{-\frac{q_e}{q_m}}$	<i>Plot</i> $\ln \frac{q_e}{C_e}$ vs. q_e The slop and the intercept give the Temkin constant K_e and q_m	Ogbodo et al. (2021), Debord et al. (2022), Riyanto & Prabalaras (2019)

Where q_e is the amount of adsorbate adsorbed per unit mass of adsorbent at equilibrium (mg/g), q_m is the maximum adsorption capacity (mg/g), K_L is the Langmuir adsorption constant (L/mg), C_e is the equilibrium concentration of adsorbate in solution (mg/L), β is Constant related to the mean free energy of adsorption per mole of the adsorbate (mol^2/J^2), ϵ is the Polanyi potential, calculated as $\epsilon = RT \ln(1 + 1/C_e)$ (J/mol), R is the Universal gas constant (8.314 J/mol.K), T is the temperature (K), K_f Freundlich adsorption constant $[(\text{mg/g})(\text{L/mg})^{(1/n)}]$, n is the Freundlich intensity parameter (dimensionless), A is the Temkin isotherm constant (L/g), b is the Temkin constant related to heat of adsorption (J/mol), α is the initial adsorption rate (mg/g. min.). K_e is the Elovich equilibrium constant

establish how time influences sorption. Adsorption kinetic models are generally classified into adsorption reaction models and adsorption diffusion models. Reaction models describe the rate at which adsorbents adsorb adsorbates without identifying the underlying cause of adsorption, whereas diffusion models reveal mass action, external diffusion, and internal (pore) diffusion mechanisms. The adsorption rate constant of methylene blue (MB) from aqueous solution using RSSS, SSS-KOHAC, and

SSS- $\text{H}_3\text{PO}_4\text{AC}$ at varying initial concentrations was calculated using several models, including intraparticle diffusion, first-order, pseudo-second-order, and Avrami models. The linearised equations of the kinetic models presented in Table 5 were used to fit the experimental data. Microsoft Excel was employed to calculate constants and perform linear regression analyses. Adsorption kinetic experiments were conducted with initial adsorbate concentrations of 200, 400, 600, 800, and 1000 mg/L to ensure consistency.

Table 5 Kinetic model equations

Kinetic models	Equation	Plot	References
First-order model	$\ln C_t = \ln C_0 - k_1 t$	$\ln\left(\frac{C_t}{C_0}\right)$ vs t	Asadu et al. (2021a, b), Ouaddari et al. (2024)
Pseudo-second-order model	$\frac{t}{q_t} = \frac{1}{K_2 q_e^2} + \frac{1}{q_e} t$	$\frac{t}{q_t}$ vs t	Eslami et al. (2020), Juzsakova et al. (2023), Majamo et al. (2024)
Avrami model	$\ln\left[-\ln\left(1 - \frac{q_t}{q_e}\right)\right] = \ln K + n \ln t$	$\ln\left[-\ln\left(1 - \frac{q_t}{q_e}\right)\right]$ vs $\ln t$	Grimm et al. (2023), Kouhi et al. (2023), Vega et al. (2024), Gonçalves Dos Santos et al. (2025)
Intra-particle diffusion model	$q_t = K_{\text{int}} t^{1/2} + C$	q_t vs $t^{1/2}$	Thithai et al. (2024), Wasilewska et al. (2024), Liya-naarachchi et al. (2023)

Where C_t is the concentration of adsorbate at time t , C_0 is the initial concentration, q_e and q_t are the amount of MB adsorbed (mg/g) at equilibrium and at time t (min), respectively, K_1 is the rate constant of adsorption (1/min), K_2 is the pseudo second-order rate constant of adsorption $[\text{g}/(\text{mg} \cdot \text{min})]$, k is Avrami rate constant, n is Avrami exponent, K_{int} is the rate constant of intraparticle diffusion $[\text{mg}/(\text{g} \cdot \text{min})^{1/2}]$, C is the intercept of linear plot of q_t against $t^{1/2}$ (mg/g)

The experiments were carried out at pH 7, with an agitation speed of 200 rpm and a temperature of 30 °C. Time intervals of 20, 30, 40, 50, and 60 min were used.

3 Results and discussion

3.1 Proximate analysis evaluation

Table 6 compares the proximate analysis of RSSS, SSS-KOHAC, and SSS-H₃PO₄AC. The RSSS samples contained 77% fixed carbon, 18% volatile matter, 4% ash, and 1% moisture. The high proportion of carbon supports the findings of Ogbodo et al. (2021), who reported that biomass with a high carbon content possesses adsorptive properties. This suggests that RSSS can be used as an adsorbent.

The fixed carbon percentage was highest in SSS-H₃PO₄AC, followed by RSSS, and lowest in SSS-KOHAC. The enhanced potential of acidic activators is consistent with the findings of Omar and Abdul Halim (2012), who observed that thermal treatment eliminates the volatile fraction of organic waste, thereby increasing fixed carbon and pore formation. The high fixed carbon content of SSS-H₃PO₄AC (78.8%) aligns with the findings of Ganjoo et al. (2023), who demonstrated that phosphoric acid dehydrates and cross-links lignocellulosic materials, leading to increased carbon yield and porosity.

The volatile matter content was reduced by the heat treatments of SSS-KOHAC and SSS-H₃PO₄AC. SSS-H₃PO₄AC exhibited the lowest volatile matter (15.4%) and was thermally more stable than SSS-KOHAC (15.8%). RSSS, with the highest volatile matter (18%), was less stable due to its greater thermal lability.

The ash content of RSSS increased with activation in SSS-KOHAC. The ash contents of SSS-H₃PO₄AC and SSS-KOHAC were 4.4% and 5.6%, respectively. The residual inorganic salts from KOH activation accounted for the higher ash content (5.6%) in SSS-KOHAC. Elevated ash content diminishes the pore structure's adsorption capacity, which is generally undesirable.

Moisture content increased in the activated compounds, particularly in SSS-KOHAC (4.0%). Since water can occupy

adsorption sites, low moisture levels are preferable to maintain adsorption efficiency.

Overall, the proximate analysis indicates that SSS-H₃PO₄AC is the most suitable compound for adsorption processes, owing to its highest fixed carbon, lowest volatile matter, relatively low ash content, and optimal moisture content.

3.2 Batch adsorption evaluation

Figures 2, 3, 4, 5, 6 summarise the batch adsorption performance of RSSS, SSS-KOHAC, and SSS-H₃PO₄AC as a function of contact time, adsorbent dosage, temperature, initial dye concentration, and solution pH, respectively. Table 3 and the figure captions indicate that all operational conditions were kept constant in each experiment, except for the parameter of interest, ensuring that the observed trends could be attributed solely to the selected parameter. All batch adsorption experiments were conducted at an agitation speed of 200 rpm and a temperature of 30 °C, with a contact time of 30 min to attain equilibrium.

Effect of contact time Figure 2 illustrates the influence of contact time on MB sorption at pH 6 and 30 °C, with an initial concentration of 1013 mg/L and a dosage of 0.6 g/L. SSS-H₃PO₄AC exhibited superior surface properties, achieving a peak adsorption capacity of 8.9 mg/g within 30 min, whereas SSS-KOHAC reached a peak adsorption capacity of 8.2 mg/g within 40 min. The high uptake rate observed during the initial 20–30 min indicates a large number of available, unrestricted external active sites and a strong mass-transfer driving force. Beyond this period, adsorption gradually approached equilibrium as intra-particle diffusion became the rate-limiting step. A high surface area and accessible pores are essential for the rapid kinetics that enable adsorption to occur within 30–40 min (Eldeeb et al. 2024).

Effect of adsorbent dosage Figure 3 illustrates the effect of adsorbent dosage on MB removal at pH 6, 30 °C, a contact time of 30 min, and an initial concentration of 1013 mg/L. As the dosage increased, the percentage removal of all adsorbents also rose due to the greater availability of active sites. Effective dosages of 0.4 and 0.6 g/L yielded maximum removal efficiencies of 98% and 99% for SSS-KOHAC and SSS-H₃PO₄AC at equilibrium, respectively, indicating efficient surface utilisation. At higher dosages, the incremental improvement in removal became marginal, which can be attributed to overlapping diffusion zones and partial saturation or shielding of micropores, particularly in the case of SSS-KOHAC. By contrast, SSS-H₃PO₄AC maintained high efficiency across a wider dosage range, consistent with its more favourable pore structure and surface chemistry reported for H₃PO₄-activated carbons (Liu et al. 2019).

Table 6 The proximate composition of Sandbox seeds shell (SSS)

Adsorbent properties	Adsorbents		
	RSSS	SSS-KOHAC	SSS-H ₃ PO ₄ AC
Fixed carbon (%)	77.0	74.6	78.8
Volatile matter (%)	18.0	15.8	15.4
Ash content (%)	4.0	5.6	4.4
Moisture content (%)	1.0	4.0	1.4

Fig. 2 Effect of time. Conditions: pH = 6, Temperature = 30 °C, Concentration = 1013 mg/L, Dosage = 0.6 g/L, Agitation speed of 200 rpm

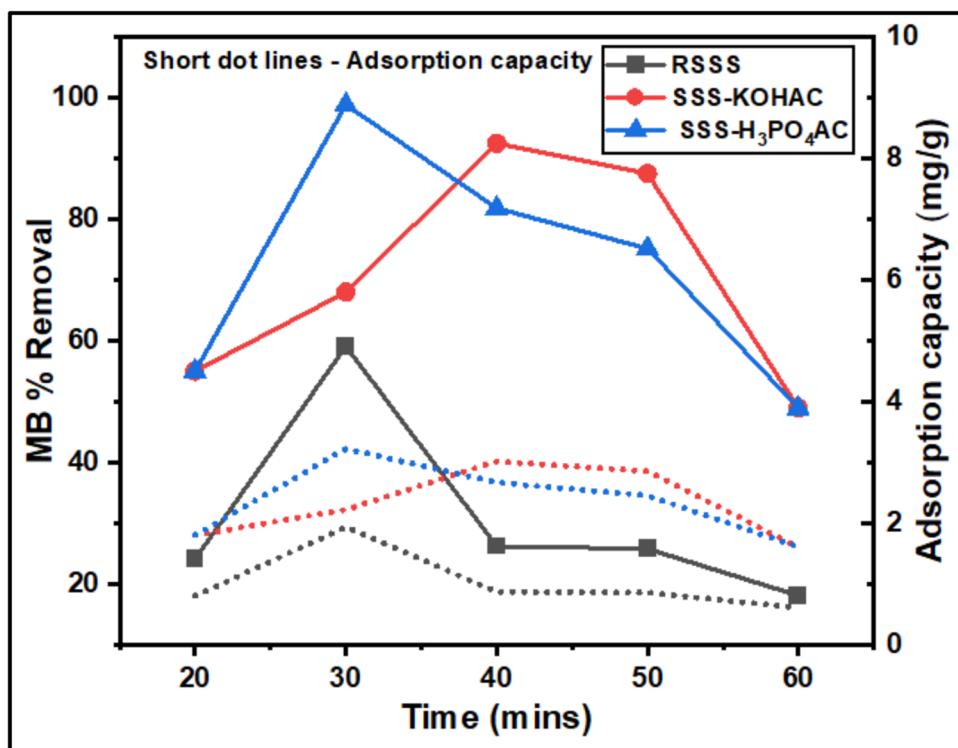
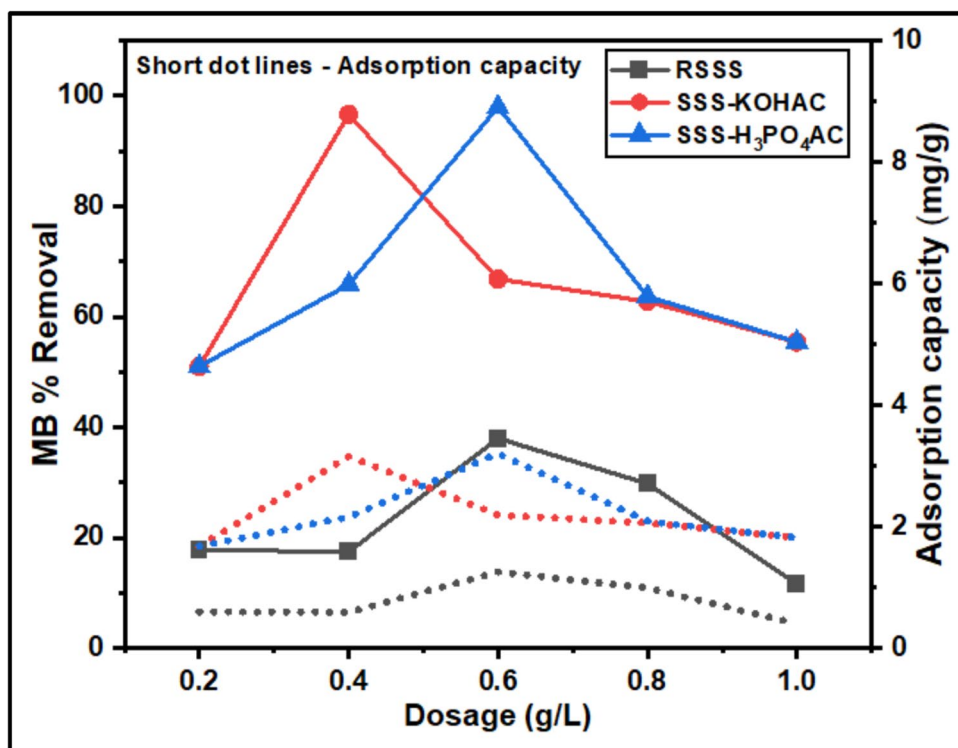


Fig. 3 Effect of adsorbent dosage. Conditions: pH = 6, Time = 30 min, Temperature = 30 °C, Concentration = 1013 mg/L, Agitation speed of 200 rpm



Effect of temperature Figure 4 illustrates the influence of temperature on MB removal at pH 6, 30–70 °C, a contact time of 30 min, a dosage of 0.6 g/L, and an initial concentration of 1013 mg/L. MB removal increased with temperature for all adsorbents, confirming the endothermic

nature of the adsorption process and the enhanced accessibility of pores at elevated temperatures. RSSS consistently exhibited the lowest removal efficiency, attributed to its limited porosity and weaker interactions between MB and the largely unmodified carbon surface. By

Fig. 4 Effect of temperature. Conditions: pH=6, Time=30 min., Dosage=0.6 g/L, Concentration=1013 mg/L, Agitation speed of 200 rpm

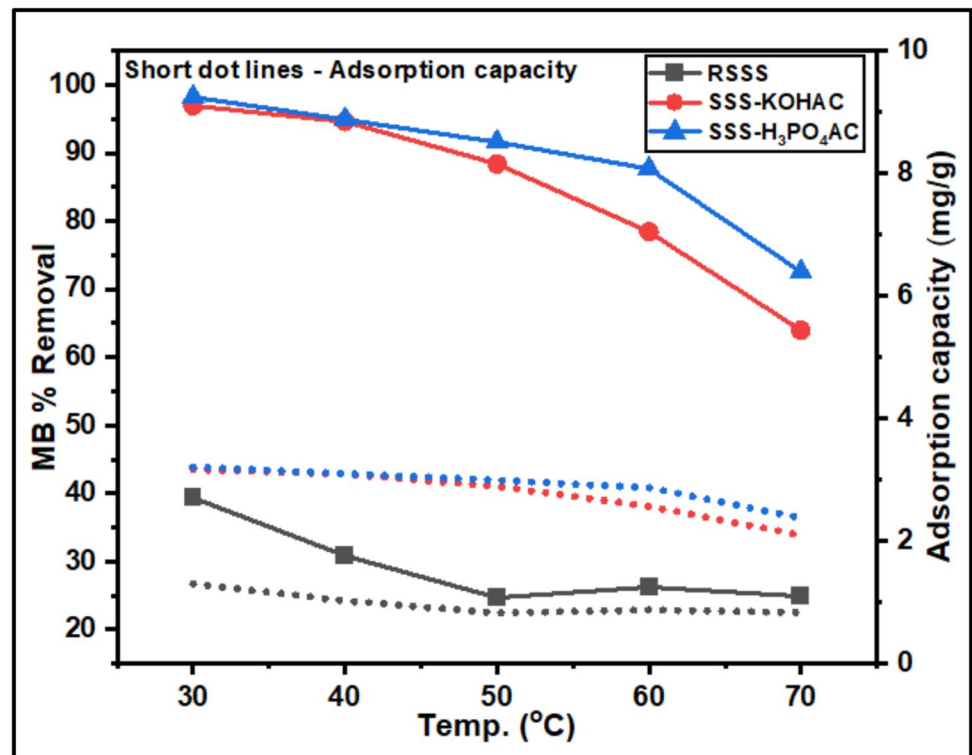
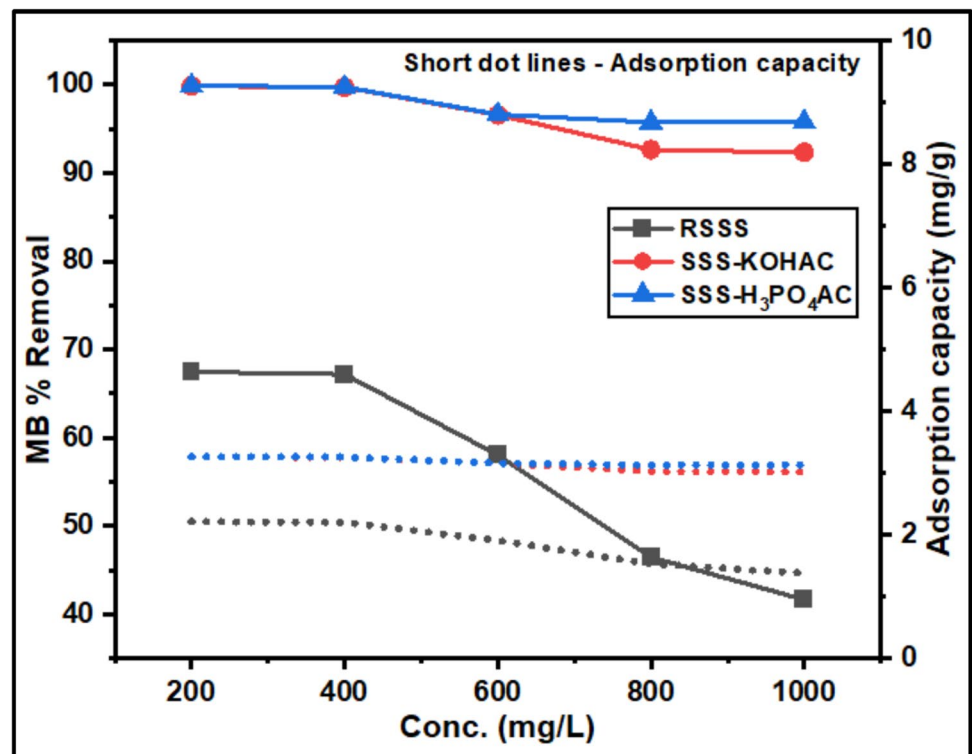


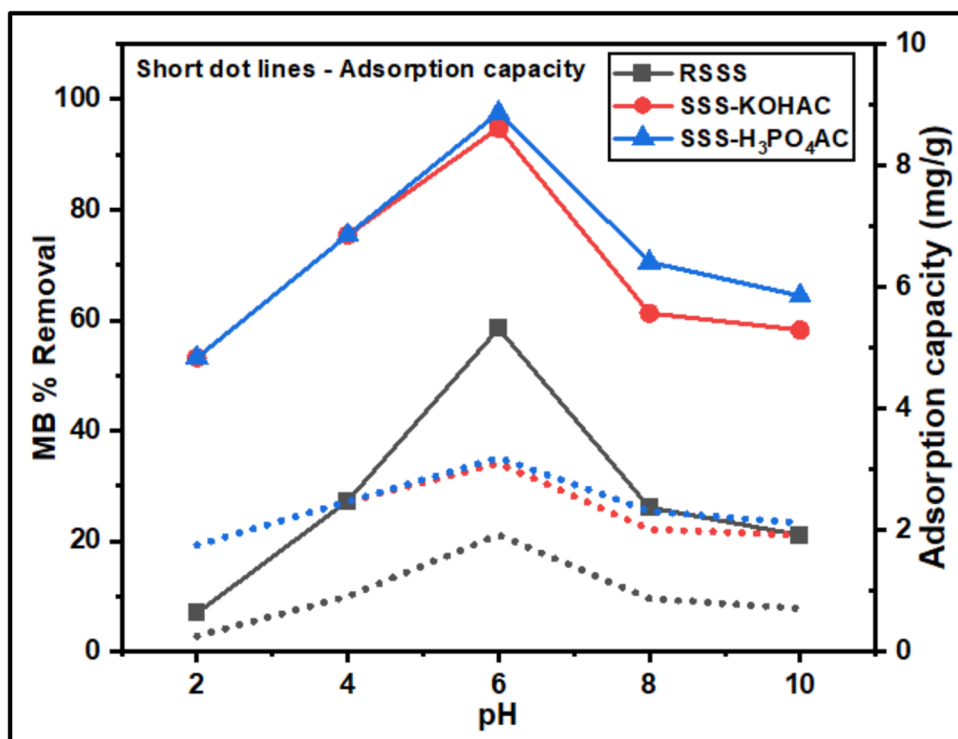
Fig. 5 Effect of concentration. Conditions: pH=6, Time=30 min., Dosage=0.6 g/L, Temperature=30 °C, Agitation speed of 200 rpm



contrast, SSS-KOHAC and SSS-H₃PO₄AC demonstrated higher and more stable removal, reflecting a greater density of energetically favourable adsorption sites and improved thermal stability. The superior performance of

SSS-H₃PO₄AC compared with SSS-KOHAC at higher temperatures further highlights the contribution of phosphate-derived functionalities to adsorption energy (Davoudi, 2022).

Fig. 6 Effect of pH Conditions: Concentration = 1013 mg/L, Dosage = 0.6 g/L, Temperature = 30 °C, Time = 30 min, Agitation speed of 200 rpm



Effect of initial concentration Figure 5 illustrates the effect of initial MB concentration at pH 6, 30 °C, a contact time of 30 min, and a dosage of 0.6 g/L. At lower concentrations, both the percentage removal and adsorption capacity increased due to the stronger driving force for mass transfer between the solution and solid. With further increases in concentration, the adsorbents became saturated at the surface, and the growth in adsorption capacity became less pronounced. The removal of MB with SSS-H₃PO₄AC was consistently the highest at the initial increase in concentration, followed by SSS-KOHAC, which exhibited a greater surface area and more developed pore networks compared with RSSS. This observation is consistent with previous studies, which reported that only adsorbents with sufficient surface area and pore capacity can maintain high removal efficiencies at elevated pollutant concentrations (Bansal et al. 2021; Liu et al. 2021).

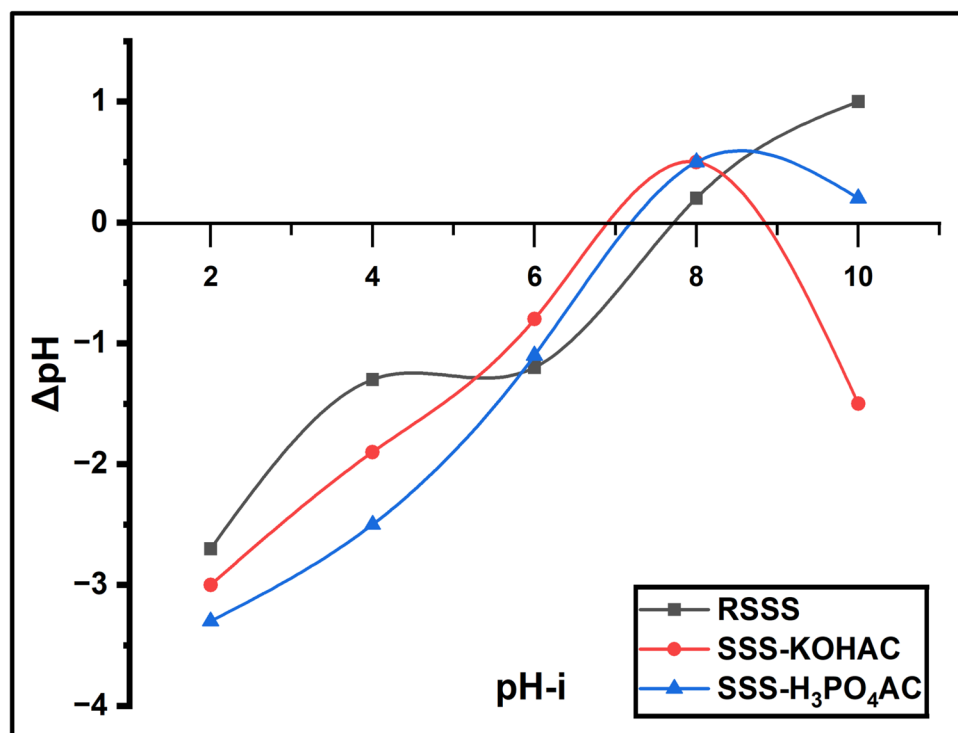
Effect of pH Figure 6 illustrates the influence of pH (2–10) on MB removal at 30 °C, a contact time of 30 min, a dosage of 0.6 g/L, and an initial concentration of 1013 mg/L. The percentage removal was highest near pH 6, with SSS-H₃PO₄AC achieving the greatest removal, followed by SSS-KOHAC and RSSS. The enhanced performance of the activated carbons resulted from modifications to

surface charge and functional groups during chemical activation. Treatment with H₃PO₄ introduced additional negatively charged oxygen-containing groups (–COOH, –OH, and phosphate species), which strengthened electrostatic attraction towards cationic MB molecules at near-neutral pH. Davoudi (2022) and Khomri et al. (2022) reported that methylene blue (MB) adsorption generally attains its maximum at pH 6–7 as a result of favourable electrostatic interactions and the enhanced ionic character of the surface sites. The combined PZC and pH results confirm that surface deprotonation and the formation of negatively charged sites at pH 6 are critical for maximising MB uptake by the activated carbons.

3.3 PZC evaluation

The PZC of the samples was determined in accordance with Asadu et al. (2021a, b). PZC values for RSSS, SSS-H₃PO₄AC, and SSS-KOHAC were 7.83, 7.19, and 6.90, respectively, as shown in Fig. 7. The ordering RSSS > H₃PO₄AC > KOHAC indicates progressively higher surface acidity after activation. The high PZC value of RSSS reflects a residual basic mineral that is neutral to positive and contains fewer acidic oxygenated groups on its surface. H₃PO₄ activation introduced phosphate-containing groups (C–O–P, –PO₃H/–PO₂H) and

Fig. 7 Point zero charge (PZC). **Conditions:** pH adjusted with $0.1 \text{ mol}\cdot\text{dm}^{-3}$ HCl and $0.1 \text{ mol}\cdot\text{dm}^{-3}$ NaOH to cover pH 2–10, 0.1 g/L of the carbon sample added to each 50 mL of $0.1 \text{ mol}\cdot\text{dm}^{-3}$ NaCl solution, shaken for 24 h at 500 rpm on a BA 200N to achieve equilibrium



other oxygenated functionalities on the carbon surface of SSS-H₃PO₄AC, thereby increasing surface acidity and lowering the PZC relative to RSSS. The aggressive etching and oxidation caused by KOH treatment resulted in the lowest PZC and the highest concentration of acidic groups (carboxyls, lactones, and phenolics) on the surface. The adsorbent surfaces were positively charged ($\text{pH} < \text{PZC}$), which caused electrostatic repulsion with the cationic dye, resulting in minimal removal across all samples at pH 2–4. SSS-KOHAC, which had the lowest PZC, attracted MB at the lowest pH, followed by SSS-H₃PO₄AC. RSSS exhibited the weakest uptake below neutral pH and required more alkaline conditions, despite having a PZC in the near-alkaline range. The pH 6 range of the activated carbons showed the highest removal and adsorption capacity, which is close to the PZC range. Partial deprotonation was more desirable at pH 6, where oxygenated sites promoted π - π interactions and specific binding. Dye aggregation was low, and pore filling was effective. The reduction in percentage removal at pH 8–10 was attributed to dye aggregation, hydroxide/solvation layers partially blocking the active sites, and the disruption of π - π and hydrogen bonding. SSS-KOHAC had the lowest PZC, followed by SSS-H₃PO₄AC and RSSS, indicating that chemical activation increased the adsorbents' surface acidity. The surface groups are more acidic when the PZC is low. Activation reduced the PZC of the raw sample (RSSS) by introducing additional acidic functionalities, particularly with KOH and H₃PO₄ treatments.

3.4 Fourier transform infrared (FTIR) evaluation

The FTIR analyses of RSSS, SSS-H₃PO₄AC, and SSS-KOHAC are shown in Figs. 8, 9, and 10, respectively. The band assignments were consistent with the findings of Asadu et al. (2021a, b), and functional groups were identified based on their characteristic frequencies. Distinct functional groups were indicated by several prominent adsorption peaks highlighted in the red spectral trace. The primary groups present included hydroxyl (–OH), aliphatic (C–H), carbonyl (C=O), and aromatic or nitro (N–O) functionalities. Figure 8 shows the RSSS FTIR spectrum. The lignin or cellulose content produced a broad peak at approximately 3283.8 cm^{-1} , corresponding to O–H stretching vibrations from alcohols or phenols. The peak at 2922.2 cm^{-1} represented typical aliphatic C–H stretching. Conjugated carbonyl contributions or combination bands appeared at 2124.6 cm^{-1} and 1968.0 cm^{-1} . A distinct peak at 1632.6 cm^{-1} reflected C=O stretching vibrations, indicating oxygenated functional groups naturally present in raw organic materials. The C–O–C stretching linked to cellulose-derived polysaccharides caused a sharp transmittance drop to 80.5% at 1028.7 cm^{-1} . Overall, the presence of O–H, C–H, and C–O groups highlighted the complex organic structure of RSSS and its suitability as a carbon precursor. Figures 9 and 10 display the FTIR spectra of SSS-KOHAC and SSS-H₃PO₄AC. Multiple distinct adsorption bands confirmed the presence of functional groups on the activated carbon surfaces. Strong signals at 3664.0 cm^{-1} (SSS-H₃PO₄AC) and

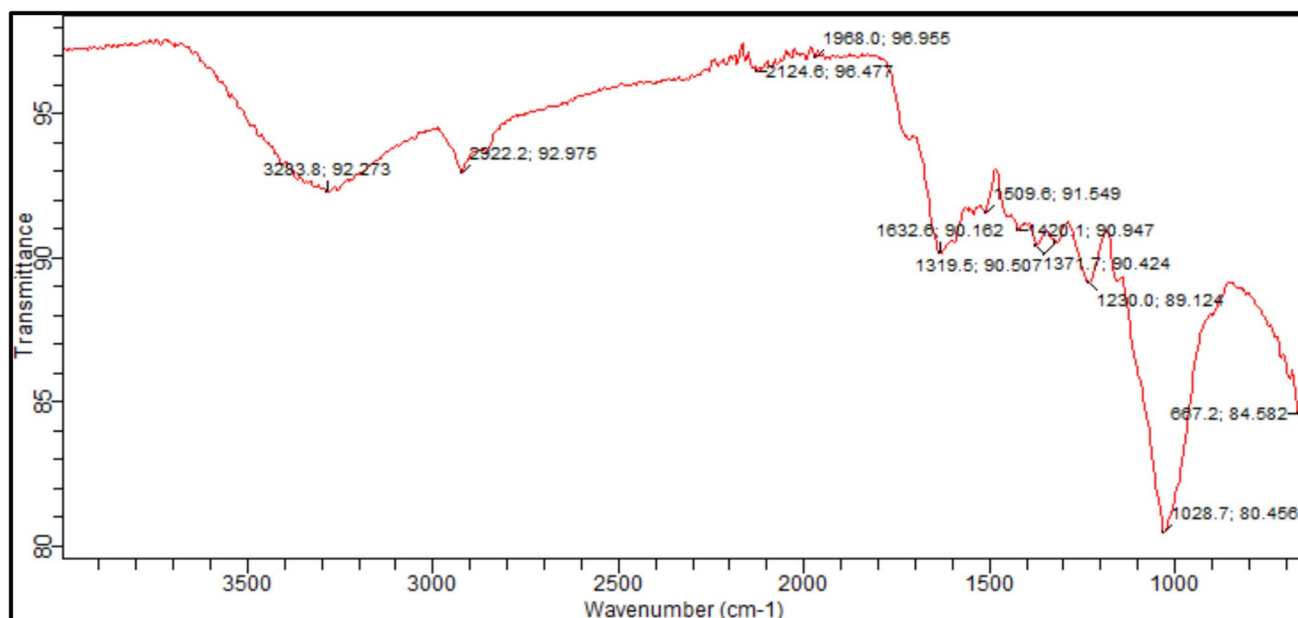


Fig. 8 FTIR image of RSSS before adsorption of methylene blue from aqueous solution

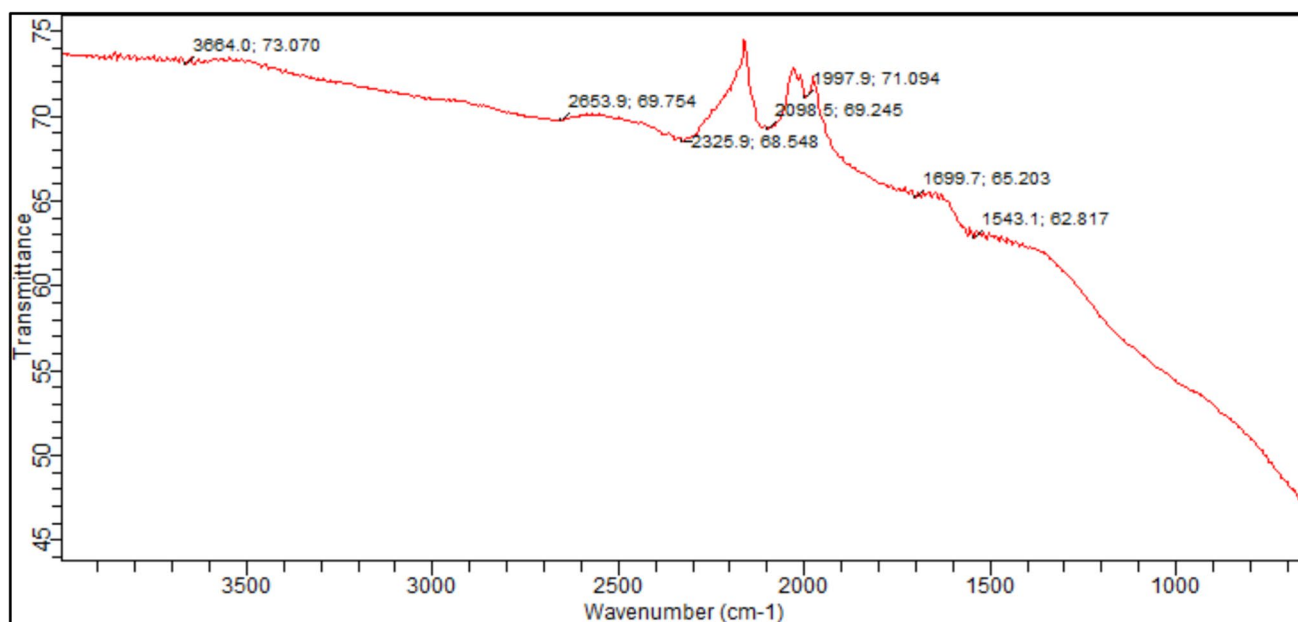


Fig. 9 FTIR image of SSS-H₃PO₄AC before adsorption of methylene blue from aqueous solution

3649.1 cm⁻¹ (SSS-KOHAC) corresponded to O–H stretching vibrations, indicative of alcohol or phenolic functionalities. Aliphatic C–H stretching was observed at 2851.4 cm⁻¹ for SSS-KOHAC and 2653.9 cm⁻¹ for SSS-H₃PO₄AC. Peaks at 2325.9 and 1997.9 cm⁻¹ (SSS-H₃PO₄AC) and 2333.3 and 1997.9 cm⁻¹ (SSS-KOHAC) suggested asymmetric C≡C and C≡N stretching vibrations or overtone bands, reflecting structural rearrangements induced by activation. The FTIR spectra demonstrate that H₃PO₄ activation successfully introduced and

stabilised a variety of oxygen-containing functional groups, significantly enhancing surface reactivity, adsorptive interactions, and the functional role of the activating agent within the carbon matrix.

3.5 XRF evaluation

Table 7 presents a comprehensive quantitative XRF analysis comparing RSSS, SSS-H₃PO₄AC, and SSS-KOHAC. The major

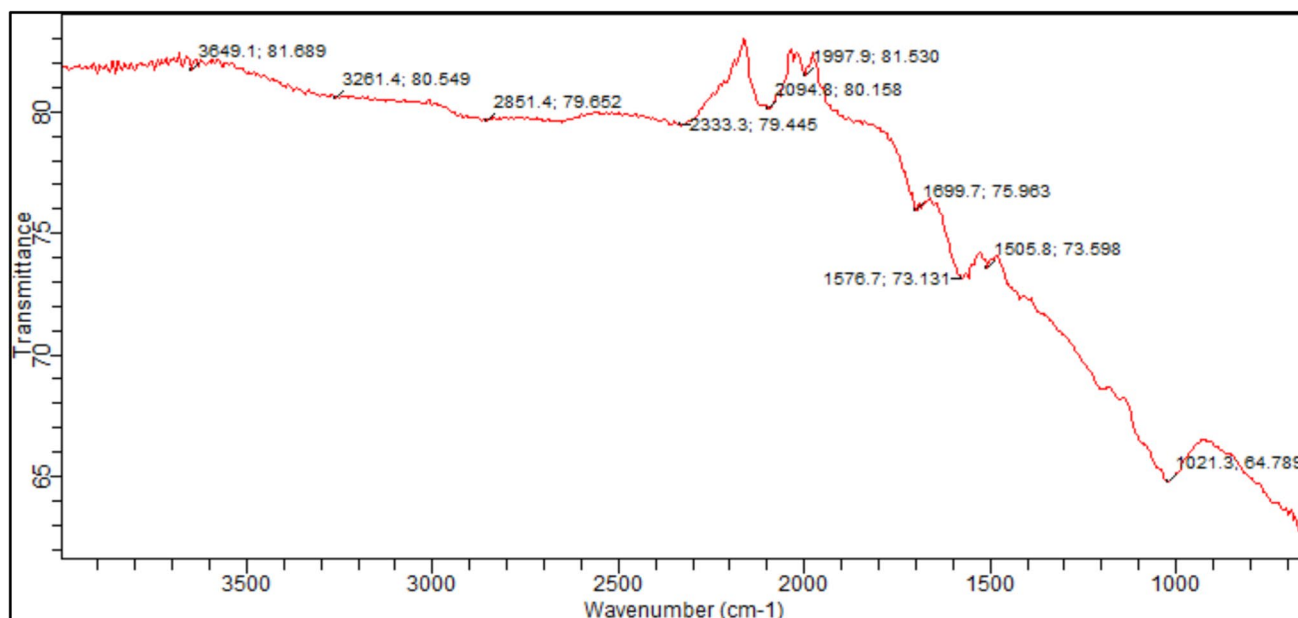


Fig. 10 FTIR image of SSS-KOHAC before adsorption of methylene blue from aqueous solution

constituents identified in the samples were SiO_2 , Fe_2O_3 , P_2O_5 , CaO , K_2O , and Al_2O_3 . RSSS contained the highest percentages of SiO_2 (17.481%) and CaO (21.506%), followed by SSS-KOHAC (10.874% SiO_2 , 10.953% CaO) and SSS- $\text{H}_3\text{PO}_4\text{AC}$ (6.657% SiO_2 , 2.586% CaO). The elevated SiO_2 and CaO contents in RSSS reflect the presence of silicate and carbonate precursors, indicating a mineral-rich matrix. Activated samples must be depleted of these compounds, as they reduce surface area and obstruct pores. The observed decrease in SiO_2 and CaO following activation suggests structural opening through leaching or transformation during acid/base treatment. This trend is consistent with Bedia et al. (2020), who reported that mineral matter removal during activation enhances pore accessibility and adsorption efficiency. SSS-KOHAC exhibited the highest K_2O content (29.049%), while SSS- $\text{H}_3\text{PO}_4\text{AC}$ showed the highest P_2O_5 content (70.164%). KOH activation promotes potassium intercalation into the carbon matrix, thereby increasing surface area and pore development. The elevated K_2O content in SSS-KOHAC supports the findings of Singh (2024), who demonstrated that KOH activation enhances micropore formation and adsorption of volatile organic molecules. RSSS contained the highest Al_2O_3 concentration (9.966%), followed by SSS-KOHAC (9.354%) and SSS- $\text{H}_3\text{PO}_4\text{AC}$ (4.443%). Quantitative XRF analysis of elemental composition (wt%) also revealed oxygen contents of 33.678% in RSSS, 50.414% in SSS- $\text{H}_3\text{PO}_4\text{AC}$, and 31.89% in SSS-KOHAC.

The addition of oxygenated functional groups ($-\text{OH}$, $-\text{COOH}$, and $-\text{PO}_4$) to SSS- $\text{H}_3\text{PO}_4\text{AC}$ (50.414%) increased its oxygen content and enhanced its hydrophilicity and chemical reactivity—two crucial properties for activated carbon in

aqueous-phase adsorption. Heidarinejad et al. (2020) reported that H_3PO_4 activation is particularly effective for aqueous-phase adsorption of dyes and heavy metals, as it introduces oxygenated functional groups ($-\text{OH}$, $-\text{COOH}$, $-\text{PO}_4$) and promotes mesoporosity. All three samples contained varying amounts of Ca, Si, P, Al, and K. The potassium content of SSS- $\text{H}_3\text{PO}_4\text{AC}$ was the most significant, at 30.622%. Phosphorus (P), especially when introduced chemically through activation, enhances surface functionality, while traces of aluminium can alter the Lewis acidity of the carbon surface and interact with diverse organic pollutants. SSS- $\text{H}_3\text{PO}_4\text{AC}$ exhibited the highest phosphorus levels (30.622%), whereas RSSS contained the highest calcium content. The XRF results are consistent with the FTIR and XRF investigations of Shahcheragh et al. (2023), which demonstrated that activation techniques influence adsorption efficiency and surface chemistry by introducing oxygenated functional groups and increasing porosity. SSS- $\text{H}_3\text{PO}_4\text{AC}$ is particularly effective for heavy metal and dye adsorption due to its mesoporosity and acidic functional groups. In contrast, KOH activation (SSS-KOHAC) promotes micropore formation and a large surface area, making it more suitable for gas-phase adsorption and the removal of organic pollutants (Cheng et al. 2015; Jawad et al. 2021; Wang & Kaskel 2012). RSSS, although mineral-rich, lacks the specific surface chemistry and porosity required for highly efficient adsorption.

3.6 XRD evaluation

The diffracting peaks of varying intensities and angles observed in the XRD patterns were attributed to crystalline

Table 7 XRF quantitative analysis of Samples

Comp	Concn. (wt. %)			Mole %			Elemnt	Concn. (wt. %)			Intensity (c/s)		
	RSSS	SSS-H ₃ PO ₄ AC	SSS-KOHAC	RSSS	SSS-H ₃ PO ₄ AC	SSS-KOHAC		RSSS	SSS-H ₃ PO ₄ AC	SSS-KOHAC	RSSS	SSS-H ₃ PO ₄ AC	SSS-KOHAC
SiO₂	17.481	6.657	10.874	21.829	12.915	14.398	O	33.678	50.414	31.889	0.000	0.000	0.000
V₂O₅	0.048	0.009	0.016	0.020	0.006	0.007	Mg	0.000	0.000	5.535	0.000	0.000	1.136
Cr₂O₃	0.020	0.653	2.618	0.010	0.501	1.370	Al	5.275	2.352	4.951	10.039	6.498	10.968
MnO	0.555	0.140	0.241	0.587	0.229	0.270	Si	8.171	3.112	5.083	82.683	48.204	62.410
Fe₂O₃	3.261	9.826	16.456	1.532	7.173	8.198	P	3.033	30.622	2.223	92.124	1453.734	89.820
CoO	0.030	0.057	0.104	0.030	0.088	0.111	S	1.795	0.599	0.311	98.274	30.981	23.315
NiO	0.041	0.087	1.505	0.042	0.135	1.603	Cl	3.204	1.885	1.560	216.653	129.292	152.729
CuO	0.545	0.266	0.306	0.514	0.390	0.306	K	22.193	1.185	24.116	1825.887	124.050	2992.271
Nb₂O₅	0.078	0.021	0.053	0.022	0.009	0.016	Ca	15.371	1.848	7.828	1141.601	291.058	846.249
WO₃	0.015	0.000	0.012	0.005	0.000	0.004	Ti	1.175	0.076	0.126	152.082	26.125	26.348
P₂O₅	6.949	70.164	5.094	3.673	57.624	2.855	V	0.027	0.005	0.009	4.721	2.430	2.590
SO₃	4.482	1.494	0.775	4.200	2.176	0.770	Cr	0.014	0.447	1.791	3.039	273.592	656.365
CaO	21.506	2.586	10.953	28.774	5.375	15.538	Mn	0.430	0.108	0.187	117.032	79.591	81.995
MgO	0.000	0.000	9.178	0.000	0.000	18.115	Fe	2.281	6.873	11.510	746.417	5862.485	5698.689
K₂O	26.734	1.428	29.049	21.294	1.767	24.533	Co	0.024	0.045	0.082	9.172	45.277	47.731
BaO	2.325	0.000	0.162	1.138	0.000	0.084	Ni	0.032	0.068	1.183	13.750	61.156	609.689
Al₂O₃	9.966	4.443	9.354	7.334	5.080	7.298	Cu	0.436	0.213	0.244	212.068	220.724	144.857
Ta₂O₅	0.020	0.006	0.000	0.003	0.002	0.000	Zn	0.113	0.044	0.075	60.868	51.403	47.926
TiO₂	1.960	0.127	0.210	1.841	0.185	0.210	Rb	0.047	0.004	0.013	27.645	4.925	9.193
ZnO	0.140	0.055	0.093	0.129	0.079	0.091	Sr	0.035	0.012	0.021	18.291	13.694	13.185
Ag₂O	0.109	0.025	0.051	0.035	0.013	0.018	Zr	0.019	0.011	0.017	8.597	11.639	9.123
Cl	3.204	1.885	1.560	6.782	6.199	3.499	Nb	0.055	0.014	0.037	21.291	13.003	18.058
ZrO₂	0.026	0.016	0.023	0.016	0.015	0.015	Ag	0.102	0.023	0.048	3.747	2.019	2.204
SnO₂	0.000	0.000	1.235	0.000	0.000	0.652	Sn	0.000	0.000	0.973	0.000	0.000	38.187
PbO	0.409	0.037	0.036	0.137	0.019	0.013	Cs	0.000	0.000	0.000	0.000	0.000	0.000
Rb₂O	0.051	0.004	0.014	0.020	0.003	0.006	Ba	2.082	0.000	0.145	66.442	0.000	7.468
Cs₂O	0.000	0.000	0.000	0.000	0.000	0.000	Ta	0.017	0.005	0.000	2.243	1.395	0.000
SrO	0.041	0.014	0.024	0.030	0.015	0.019	W	0.012	0.000	0.010	1.647	0.000	1.654
							Pb	0.379	0.034	0.034	81.388	16.132	8.723

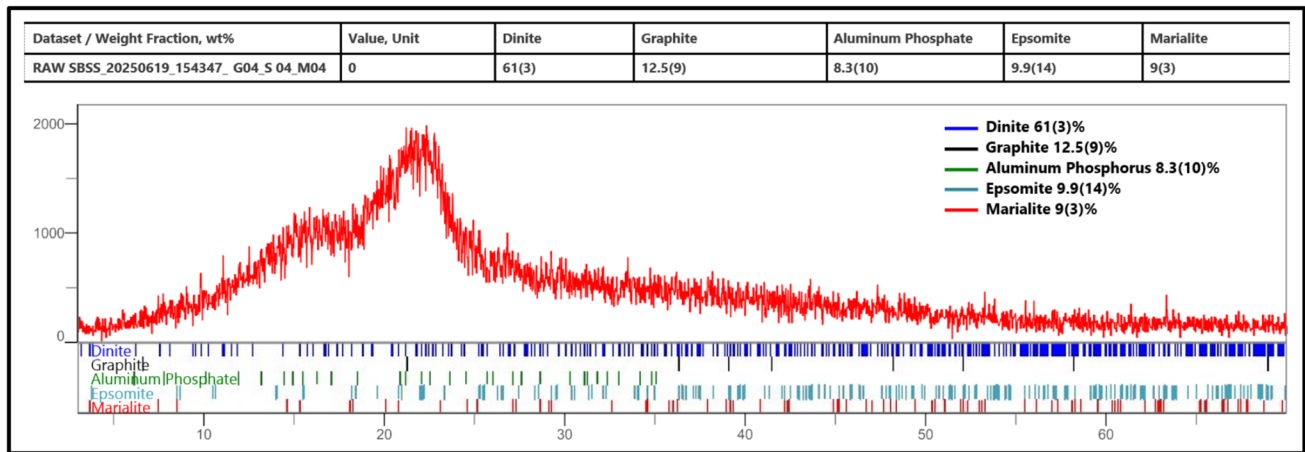


Fig. 11 XRD image of RSSS

phases. The RSSS XRD pattern (Fig. 11) exhibited a prominent peak at approximately $20^\circ 2\theta$, signifying a dominant crystalline phase. This feature was linked to the mineral dinite, which comprised the majority of the sample. Smaller peaks indicated the presence of other crystalline materials such as marialite, epsomite, graphite, and aluminium

phosphate. The detection of epsomite confirmed the presence of magnesium sulphate hydrate, which may arise either synthetically or through environmental exposure. Overall, the RSSS XRD pattern revealed a predominantly amorphous structure with distinct peaks near $20^\circ 2\theta$, likely due to residual crystalline minerals, including silicates (SiO_2) and

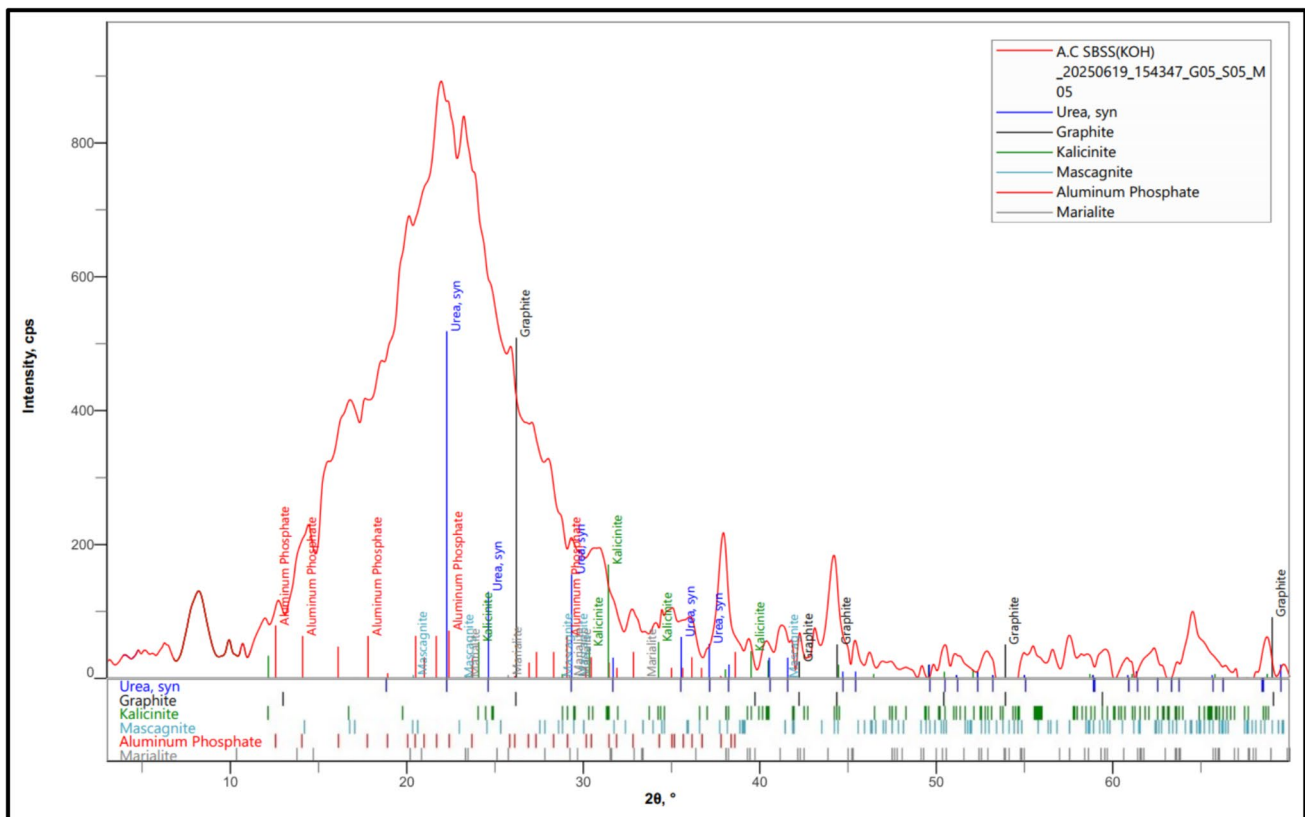


Fig. 12 XRD image of SSS-KOHAC

aluminium phosphate, incorporated into the carbonaceous matrix. These diffraction peaks were caused by inorganic impurities rather than carbon crystallinity, confirming that the carbon framework itself was amorphous, with minor mineral residues producing crystalline effects.

The XRD pattern of SSS-KOHAC (Fig. 12) displayed peaks between $20\text{--}25^\circ$, indicating the presence of distinct crystalline components. This result was consistent with the XRD analysis of Asadu et al. (2021a, b), which reported minimal variation in biomass crystallinity. Aluminium phosphate was identified as a dominant phase, evidenced by a strong peak near 20° , in agreement with Ali et al. (2022), who noted that cubic aluminium phosphate and related phases produce strong peaks in this region depending on precursor and annealing conditions. Additional peaks revealed the presence of residual urea and graphite. The large graphite peak at $\sim 26^\circ 2\theta$ was attributed to carbonaceous materials formed during pyrolysis, either from support materials or heat treatment. The presence of urea suggested unreacted or residual precursors from synthesis. KOH activation also produced potassium aluminium carbonate (kalcanite), while sulphur and alkali precursors contributed to the formation of mascagnite and marialite.

The XRD pattern of SSS- $\text{H}_3\text{PO}_4\text{AC}$ (Fig. 13) showed a strong peak at $\sim 20^\circ 2\theta$, corresponding to the crystalline

phase of flagstaffite and a phosphate-based structural order (Mezned 2023). Smaller peaks indicated additional crystalline phases, including graphite, mascagnite, brushite, marialite, and silicon oxide. Graphite peaks confirmed the presence of carbon, introduced either during synthesis or activation. Calcium and ammonium sulphate components were associated with brushite and mascagnite, arising from phosphorus-related treatments, while silicon oxide and marialite were linked to residual precursors or contaminants.

3.7 Thermal stability evaluation

Thermogravimetric analysis (TGA) is an analytical technique that measures a material's mass change or weight loss as a function of temperature over time in a controlled environment (Rajisha et al. 2011). Weight loss may result from physical processes such as evaporation, desorption, and drying, as well as chemical reactions including combustion and decomposition. TGA is essential for evaluating pore accessibility and surface hydration in thermally treated materials (Akash & Rehman 2025). Figure 14 presents the TGA curves for RSSS, SSS-KOHAC, and SSS- $\text{H}_3\text{PO}_4\text{AC}$. Evaporation of physically adsorbed water produced a slight weight decrease in all samples between $\sim 25\text{--}120^\circ\text{C}$. Hemicellulose pores absorbed more moisture in RSSS, resulting

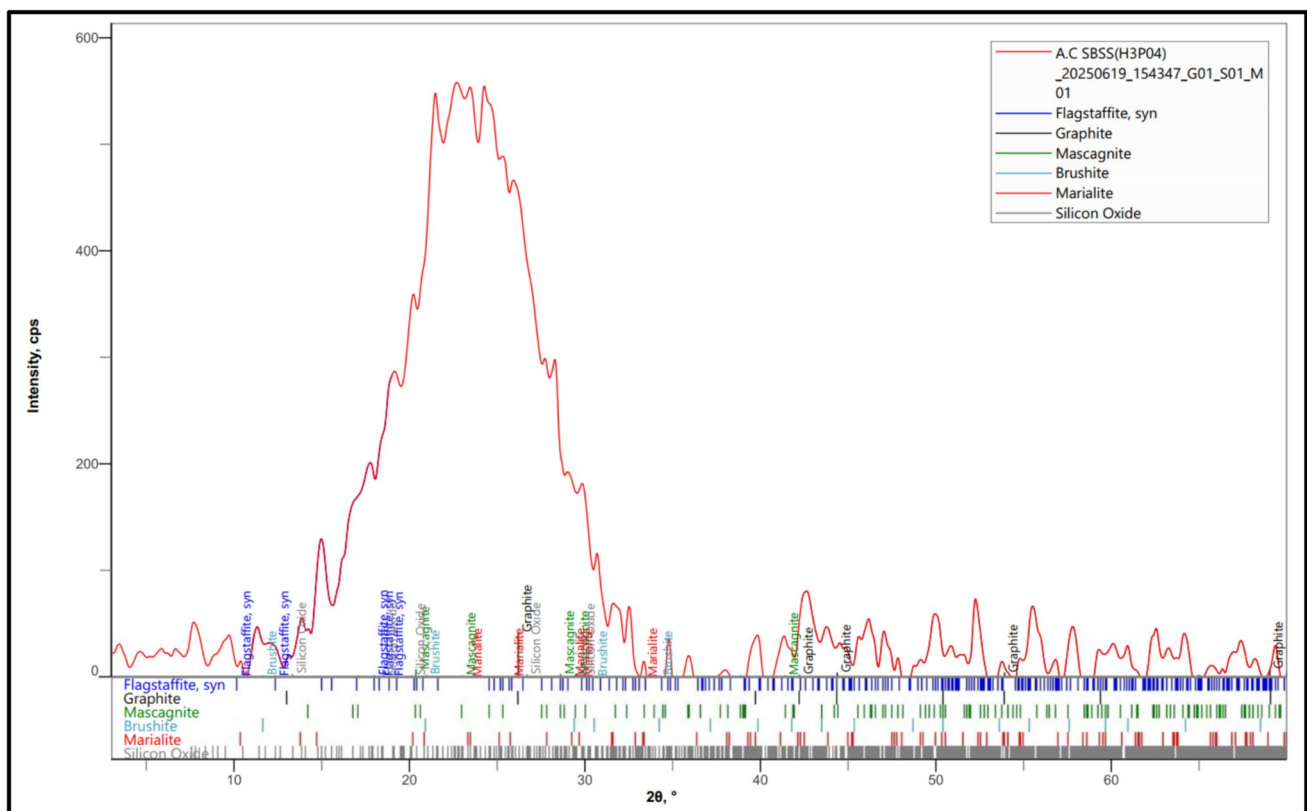


Fig. 13 XRD image of SSS- $\text{H}_3\text{PO}_4\text{AC}$

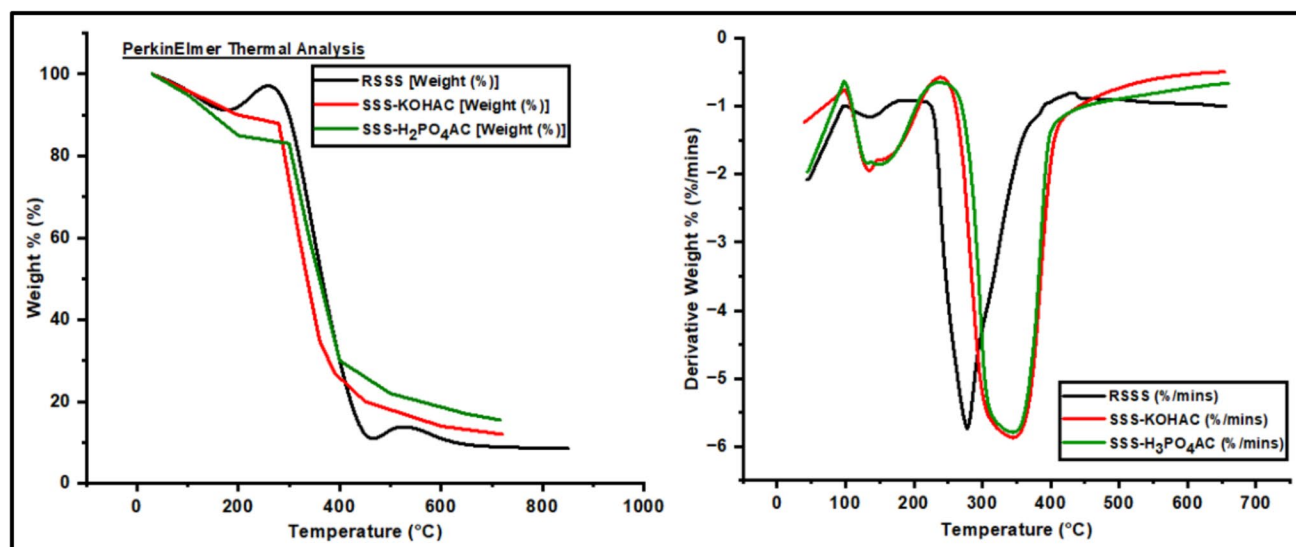


Fig. 14 TGA and DTG curves for all samples

in a marginally lower mass loss in the untreated biomass. Decomposition of SSS-KOHAC and SSS-H₃PO₄AC occurred earlier than that of RSSS, at ~200–320 °C, suggesting the breakdown of oxygenated surface groups introduced during chemical activation. SSS-H₃PO₄AC degraded more slowly, as phosphoric acid promotes phosphate-ester crosslinking and charring, thereby inhibiting rapid volatilisation. Amin et al. (2022) demonstrated that H₃PO₄ enhances carbon stability through esterification and generates thermally stable phosphate species. By contrast, alkali salts in SSS-KOHAC catalysed bond cleavage and dehydration, producing more volatile compounds and resulting in earlier degradation.

The greatest mass loss in all samples occurred in the 320–420 °C range, which is typical for biomass devolatilisation. This stage was associated with the degradation of weaker lignin fractions, cellulose, and hemicellulose. RSSS exhibited the steepest loss and the lowest intermediate weight, reflecting the presence of native biopolymers. Activation removed most of the labile components from the carbons, thereby facilitating breakdown. Slower reactions such as aromatisation, the elimination of surface oxygen groups (CO and CO₂ evolution), and partial gasification in SSS-KOHAC were reflected in the curves that levelled off above 450 °C, indicating pore formation.

The highest residue was observed for SSS-H₃PO₄AC at 700–800 °C, followed by SSS-KOHAC and RSSS. SSS-H₃PO₄AC yielded the greatest char due to enhanced carbonisation and the presence of thermally stable phosphate and pyrophosphate species, resulting in higher ash content and apparent thermal stability. Amin et al. (2022) reported that increased thermal stability and residue are attributable to stable phosphorus products formed during H₃PO₄ activation. By contrast, SSS-KOHAC left less residue because alkali

activation promoted deeper devolatilisation and pore formation, producing a more carbon-lean, porous structure. RSSS exhibited the lowest mass retention, consistent with its low mineral content and complete burnout of native organics. Significant devolatilisation occurred between 320–420 °C, while moisture loss was observed below 120 °C. Activated carbons decomposed earlier but more gradually. SSS-KOHAC displayed intermediate behaviour, with lower final residue and stronger early catalysis. RSSS showed the least thermal stability due to its unmodified biopolymer content.

3.8 SEM and EDS evaluation

Scanning electron microscopy (SEM) was employed to compare the surface morphology of RSSS, SSS-KOHAC, and SSS-H₃PO₄AC, as shown in Figs. 15, 16, 17. SEM was combined with energy-dispersive spectroscopy (EDS) to determine the elemental distribution of the adsorbents (Nazari et al. 2022). The SEM image of RSSS revealed small pore structures with a rough, coarse surface prior to activation. EDS analysis of RSSS identified the elements C, O, Ne, Mg, Al, Si, P, S, K, and Ca. The surfaces of SSS-KOHAC and SSS-H₃PO₄AC appeared rougher and more fractured following chemical activation. EDS of SSS-KOHAC showed a more pronounced porous surface with a high carbon domain, reflected in increased K and C intensities and a reduction in oxygen-containing functional groups. The SEM image of SSS-KOHAC revealed a fractured, etched surface with visible micro-mesopore mouths, resulting from the chemical etching of lignin and cellulose. Chen et al. (2020) and Ma et al. (2020) reported that KOH activation promotes decarboxylation and dehydration, leading to a more graphitised crystal structure. The clear K peaks indicated residual

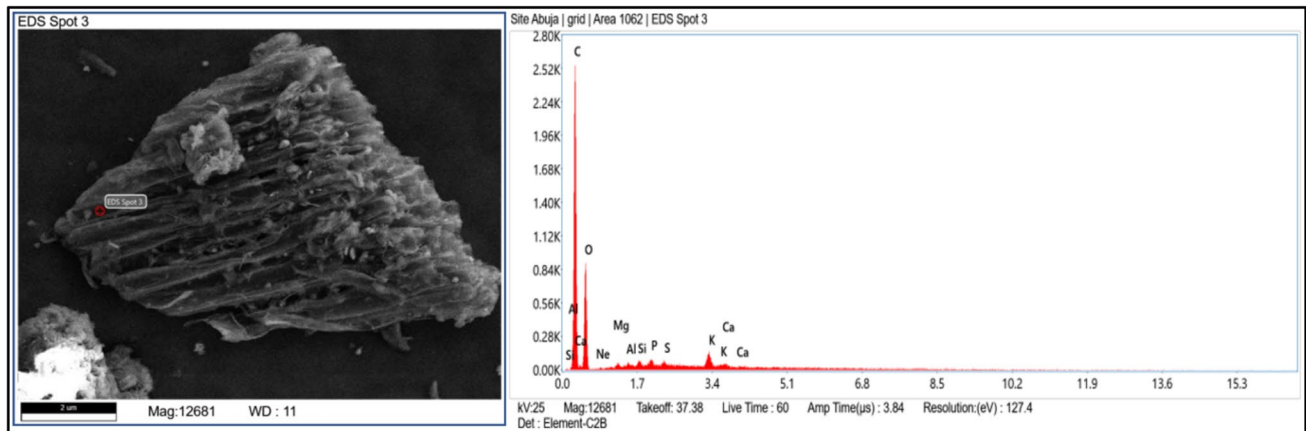


Fig. 15 SEM & EDS of RSSS

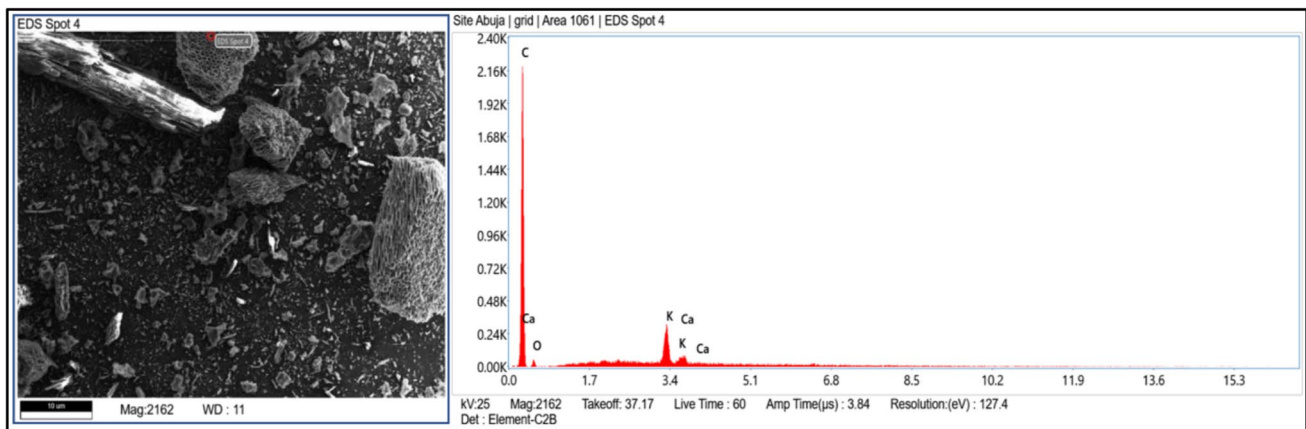
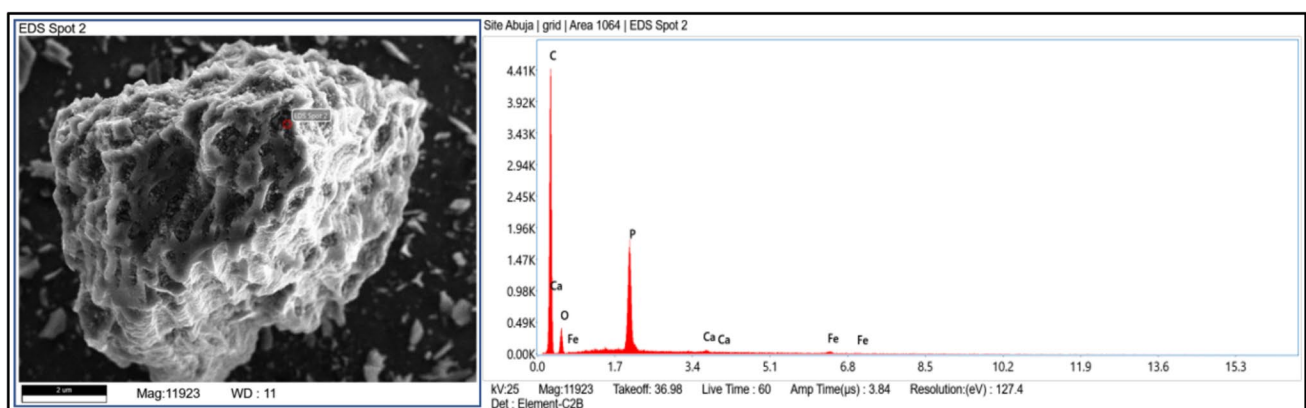


Fig. 16 SEM & EDS of SSS-KOHAC

Fig. 17 SEM & EDS of SSS-H₃PO₄AC

potassium species and ash, confirming effective KOH treatment and a more basic surface chemistry, which favours the adsorption of cationic dyes. SSS-H₃PO₄AC particles

exhibited sponge-like, highly porous, rounded pore walls, indicative of phosphoric-acid-induced dehydration and cross-linking. EDS revealed a distinct P peak, confirming

phosphate incorporation or retention within the carbon matrix. The stronger phosphate groups, higher carbon content, enhanced porosity, and improved thermal stability of SSS-H₃PO₄AC produced more pronounced outcomes compared with RSSS and SSS-KOHAC. The operational resilience of SSS-H₃PO₄AC was attributed to the thermal stability of phosphate groups. Furthermore, SSS-H₃PO₄AC displayed a cleaner elemental profile, reduced inorganic residue, and greater porosity, resulting in lower oxygen content and higher carbon content (Nazari et al. 2022; Zeng et al., 2022).

3.9 Textural properties evaluation

Table 8 summarises the textural properties of the samples, while Fig. 18 displays the BET graph. The specific surface area of the prepared adsorbents was determined using the BET method, which provides a precise and comprehensive evaluation of adsorption potential (Tian & Wu 2018). The textural properties of the chemically activated carbons were significantly improved compared with the raw material (RSSS). SSS-KOHAC exhibited the highest BET surface area and micropore volume (0.194 cc/g), consistent with the strong etching and redox characteristics of KOH, which promote ultramicro-pore formation. The removal of methylene blue (MB) at low dosages was enhanced by SSS-KOHAC due to the increased initial adsorption rate arising from greater microporosity. SSS-KOHAC recorded the highest BET surface area, micropore surface area, pore volume, and micropore volume, with values of 543.378 m²/g, 546.532 m²/g, 0.280 cc/g, and 0.194 cc/g, respectively. Since surface area directly influences adsorption performance, biochars with higher BET values (> 236 m²/g) generally exhibit superior nutrient adsorption (Irewale et al. 2025). Maneerung et al. (2016) similarly reported that activated carbons with BET values exceeding 500 m²/g perform better in adsorbing dyes and heavy metals. SSS-H₃PO₄AC displayed a slightly larger pore diameter (2.126 nm) and the widest pore width (6.65 nm), indicating easier pore accessibility and a more uniform distribution of micro- and mesopores. Despite having a smaller BET surface area than SSS-KOHAC, SSS-H₃PO₄AC achieved the highest iodine number (1142 mg/g), attributed to phosphorus–oxygen surface groups that enhance iodine adsorption capacity. Adsorption energies ranged between 4–4.45 kJ/mol across all samples, characteristic of physisorption. RSSS exhibited a lower iodine capacity (482 mg/g), though its slightly higher adsorption energy was likely due to strong native adsorption sites, such as oxygen groups or mineral inclusions, rather than extensive porosity. The iodine number of RSSS reflected medium adsorption capacity and microporosity, consistent with its raw,

Table 8 Textural properties

Parameters	BET surface area (m ² /g)	Micropore surface area (m ² /g)	Pore volume (cc/g)	Micropore volume (cc/g)	Pore diameter (nm)	Pore width (nm)	Adsorption energy (KJ/mol)	Iodine number (mg/g)
RSSS	209.454	253.759	0.126	0.189	2.118	5.842	4.451	482.334
SSS-KOHAC	543.378	546.532	0.280	0.194	2.112	6.323	4.112	977.361
SSS-H ₃ PO ₄ AC	433.493	480.844	0.240	0.171	2.126	6.647	4.130	1142.37

unactivated state. Begum et al. (2023) noted that raw precursors lacking chemical activation show limited pore development and iodine uptake due to the absence of etching or pore-opening processes. The iodine number of SSS-KOHAC significantly increased its adsorption capacity, as KOH activation promotes micropore formation through redox reactions and carbon rearrangement. This agrees with Chen et al. (2014), who demonstrated that KOH activation enhances small-molecule adsorption alongside increasing BET surface area and iodine number. The high iodine number of SSS-H₃PO₄AC, restored through pore formation and surface chemistry, makes it particularly effective for gas adsorption, dye removal, and water purification (Vallejo et al. 2025).

The BET plot illustrates the relationship between $1/[W(P/P_0) - 1]$ and relative pressure, as shown in Fig. 18. Reduced y-values at a given P/P_0 point signify increased monolayer capacity (W_m) and the corresponding contribution to surface area. RSSS displayed the largest line due to its low BET value, which was associated with the lowest W_m and weakest uptake. Chemical activation enhanced monolayer adsorption, as evidenced by the downward shift of SSS-KOHAC and SSS-H₃PO₄AC. SSS-KOHAC exhibited the highest BET surface area and the largest W_m , making it the most effective in generating ultramicropores. SSS-H₃PO₄AC occupied an intermediate position, with a moderately higher BET value, balanced micro–mesoporous structure, and greater uptake compared with RSSS.

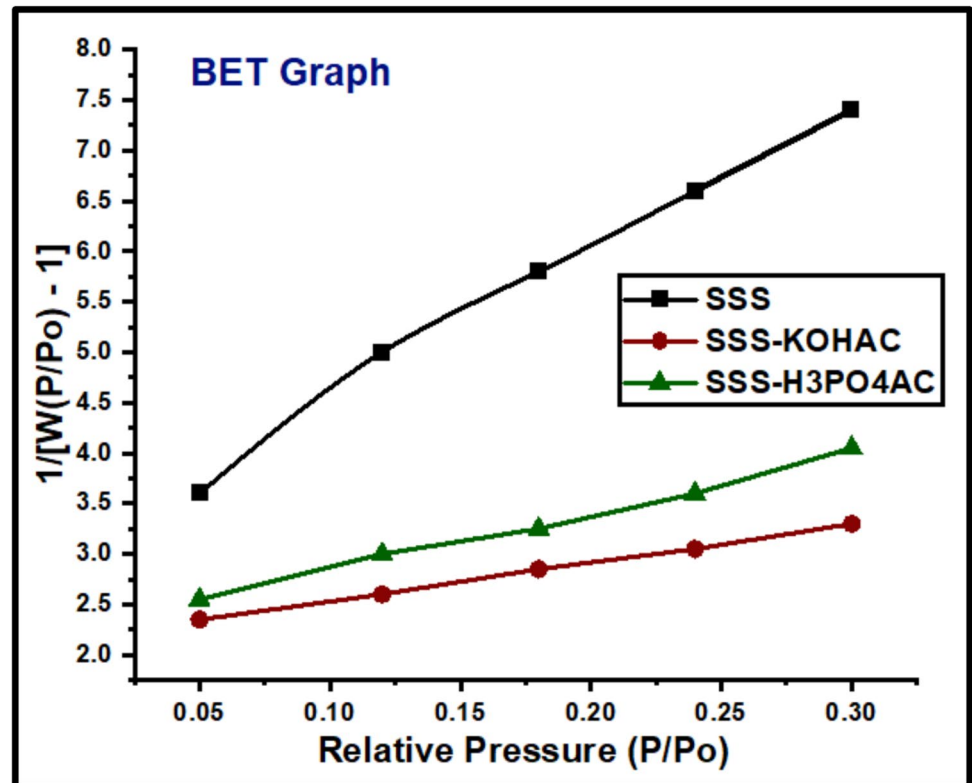
3.10 Thermodynamics model

The standard Gibbs free energy change (ΔG°), standard enthalpy change (ΔH°), and standard entropy change (ΔS°) are the parameters used to characterise the system in order to establish the thermodynamic model. Table 9 shows that the negative Gibbs free energy (ΔG°) values of SSS-KOHAC and SSS-H₃PO₄AC across the temperature range indicate spontaneous adsorption, consistent with the findings of Ebelegi et al. (2020), who reported that negative ΔG° values validate the feasibility of adsorption processes. By contrast, RSSS exhibited positive ΔG° values, indicating non-spontaneous adsorption due to its low porosity and absence of activating functional groups. Activated samples ($\Delta G^\circ < 0$) adsorbed MB spontaneously as a result of their high surface energy, in agreement with Amin et al. (2017). Tran et al. (2021) noted that negative enthalpy changes (ΔH°), which signify energy release during adsorbate binding, are characteristic of exothermic adsorption. SSS-KOHAC, SSS-H₃PO₄AC, and RSSS all exhibited negative ΔH° , confirming exothermic behaviour. Negative entropy changes (ΔS°) reflected reduced disorder when molecules adsorbed onto the activated carbon surfaces. The larger negative ΔH° values of SSS-KOHAC and SSS-H₃PO₄AC suggested stronger adsorbent–adsorbate interactions. The Van't Hoff model demonstrated strong linearity and reliability, with R^2 values > 0.97 for SSS-KOHAC and SSS-H₃PO₄AC (Girish 2025). RSSS showed moderate linearity ($R^2 = 0.77$), reflecting its considerable but variable raw adsorption capacity. According to Girish (2025) and Tran et al. (2021), raw

Table 9 Thermodynamics model

	Temp. K	Slope	Intercept	R^2	ΔH° (KJ/mol)	ΔS° (KJ/mol.k)	ΔG° (KJ/mol)	Equation	ΔH_x (J/mol)	Adsorption potentials		
										A (kJ/mol)	R^2	Equation
RSSS	303	1664.443	−6.24422	0.76833	−13.8382	−0.05191	1.891902		1.197E-05	−1.260056199	0.6356	
	313						2.411046			−0.959784837		
	323						2.930191	$y = 1664.4x - 6.2442$		−0.760980558		$y = 0.0101x - 4.1756$
	333						3.449335			−0.841815443		
	343						3.96848			−0.816286301		
SSS-KOHAC	303	7599.535	−21.7145	0.99486	−63.1825	−0.18053	−8.48052		0.000152	−8.763920174	0.9962	
	313						−6.67517			−7.596701907		
	323						−4.86983	$y = 7599.5x - 21.715$		−5.785457522		$y = 0.1506x - 54.513$
	333						−3.06448			−4.243882426		
	343						−1.25913			−2.90888003		
SSS-H ₃ PO ₄ AC	303	7314.171	−20.4191	0.97586	−60.81	−0.16976	−9.37131		1.44E-04	−10.13645722	0.9693	
	313						−7.67366			−7.749835902		
	323						−5.97601	$y = 7314.2x - 20.419$		−6.655006157		$y = 0.1486x - 54.79$
	333						−4.27836			−5.800191242		
	343						−2.58072			−3.683299074		

Fig. 18 BET graph



adsorbents without chemical activation often exhibit heterogeneous binding sites and non-uniform surface characteristics. The low R^2 of RSSS confirmed heterogeneous adsorption and non-uniform mechanisms due to the absence of an activator. Figure 19 presents the MB fit statistics for RSSS, RSSS-KOHAC, and RSSS-H₃PO₄AC, while Fig. 20 shows the adsorption potential. RSSS displayed an R^2 of 0.6356 and a low adsorption potential range (−0.76 to 1.26). By contrast,

RSSS-KOHAC and RSSS-H₃PO₄AC exhibited higher adsorption potentials, with ranges of −2.91 to 8.76 and −3.68 to 10.14, respectively, and R^2 values of 0.9962 and 0.9693. Adsorption potentials with small negative values indicated weak physical adsorption due to ineffective adsorbate interaction. The more negative adsorption potential values of RSSS-H₃PO₄AC reflected higher adsorption energy and stronger affinity. Negative physisorption is characterised by exothermic and spontaneous adsorption. Negative Gibbs

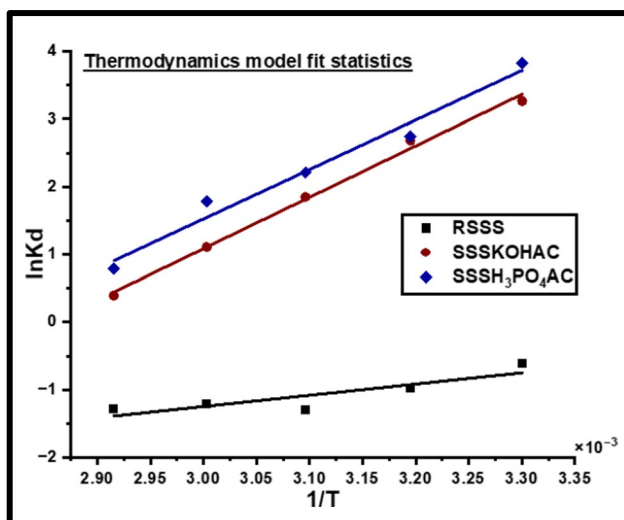


Fig. 19 Thermodynamics fit statistics

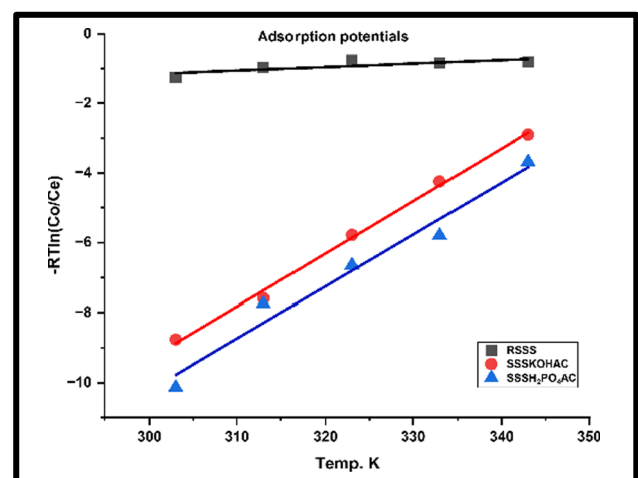


Fig. 20 Adsorption potentials

Table 10 MB adsorption Isotherm model

I-M	Parameters	Temperature (K)			313 K			323 K		
		303 K			SSS-KOHAC	SSS-H ₃ PO ₄ AC	RSS	SSS-KOHAC	SSS-H ₃ PO ₄ AC	RSS
LI	q_{max} (mg/g)	7.35835173	1.155508308	1.0985752	1.1532456	1.3883875	12.41773252	1.3216849	12.41773252	
	K_L (L/mg)	-0.0266451	-0.00874	-0.00858	-0.031554312	-0.00891	-0.037146376	-0.00885	-0.037146376	
	R^2	0.85025	0.52995	0.67013	0.53159	0.3601	0.57285	0.66821	0.57285	
	K_f (L/mg)	1426165742	1.23435E+50	1.47867E+50	22322354.46	1.091E+48	2723892.8	1.13731E+42	2723892.8	
	$1/n$	-23.32911	-23.3291	-23.32911	-22.396	-22.3955	-13.2913	-22.3955	-13.2913	
TI	R^2	0.97509	0.70838	0.88101	0.79696	0.61034	0.8367	0.87972	0.8367	
	n	-0.2241203	-0.04286	-0.0429505	-0.29177	-0.04465	-0.33743	-0.0513322	-0.33743	
	B_T (J/mol)	-233.37791	-1038.72195	-986.8003	-204.22603	-1116.833	-176.75392	-818.40619	-176.75392	
	K_T (L/mg)	0.016350295	0.007918	0.00775653	0.016672237	0.008047	0.0178904	0.00783614	0.0178904	
	R^2	0.93016	0.83259	0.97082	0.95024	0.87306	0.97359	0.93183	0.97359	
EI	K_e	-0.006046339	-0.00216	-0.0020844	-0.006402465	-0.00221	-0.007143	-0.002139	-0.007143	
	q_m (mg/g)	-45.49590537	-42.0875	-41.1522634	-44.80286738	-42.6621	-43.535046	-41.25413	-43.535046	
	R^2	0.93235	0.89682	0.89379	0.96829	0.90468	0.9713	0.89603	0.9713	
	q_m (mg/g)	4.27886E-13	1.74163E-44	2.18985E-44	4.76E-11	5.80E-43	3.02778E-10	4.94E-38	3.02778E-10	
	β (mol ² /K ² J ²)	-3.98E-08	-2.32E-07	-2.33E-07	-2.81E-08	-2.09E-07	-2.25E-08	-1.82E-07	-2.25E-08	
DRI	E (KJ/mol)	-6.27E+06	-1.08E+06	-1.07E+06	-8.90E+06	-1.20E+06	-4.44E+07	-1.37E+06	-4.44E+07	
	R^2	0.97418	0.7066	0.87954	0.79055	0.60956	0.83072	0.87923	0.83072	
I-M	Parameters	333 K			343 K			SSS-H ₃ PO ₄ AC		
		SSS-KOHAC	SSS-H ₃ PO ₄ AC	RSS	SSS-KOHAC	SSS-H ₃ PO ₄ AC	RSS	SSS-KOHAC	SSS-H ₃ PO ₄ AC	RSS
LI	q_{max} (mg/g)	2.106416	1.77481187	21.59361	1.6220074	1.8895019	21.54708037	1.93431081	3.811701925	
	K_L (L/mg)	-0.00979	-0.00959	-0.056242	-0.00986	-0.01	-0.065198151	-0.01201	-0.01284	
	R^2	0.59769	0.50094	0.34159	0.55274	0.51831	0.40951	0.54406	0.89834	
	K_f (L/mg)	6.1877E+28	5.29578E+36	16702.4431	1.9306E+40	1.7447E+35	12406.24	2.2704E+37	1.91232E+13	
	$1/n$	-13.2913	-13.29125	-19.03173	-19.0317	-19.03173	-18.3546	-18.3546	-18.3546	
FI	R^2	0.83493	0.7654	0.63013	0.80248	0.7815	0.72752	0.76992	0.98399	
	n	-0.07524	-0.058307032	-0.630983	-0.05254	-0.06038	-0.638211	-0.05448	-0.168220463	
	B_T (J/mol)	-601.12282	-824.11123	-101.28971	-907.77089	-804.70596	-98.03066	-951.0081	-244.42023	
	K_T (L/mg)	0.008222	0.00839106	0.0141119	0.008746	0.00870933	0.016029462	0.010629	0.00863572	
	R^2	0.9483	0.96518	0.83204	0.96198	0.96896	0.90102	0.92673	0.87553	

Table 10 (continued)

I-M	333 K				343 K			
	SSS-KOHAC	SSS-H ₃ PO ₄ AC	RSSS	SSS-KOHAC	SSS-H ₃ PO ₄ AC	RSSS	SSS-KOHAC	SSS-H ₃ PO ₄ AC
EI	-0.00234	-0.002363027	-0.0070154	-0.00248	-0.0024854	-0.007967231	-0.00316	-0.00275
	-41.8936	-42.91845494	-39.123631	-43.7828	-43.59198	-37.92188093	-47.2144	-39.556962
DRI	0.90746	0.91036	0.98781	0.91088	0.91363	0.98772	0.9233	0.89343
	1.4301E-27	3.29858E-34	1.6243E-07	1.2864E-37	2.4701E-33	1.53991E-07	3.8682E-37	3.3691E-15
	-1.15E-07	-1.49E-07	-1.10E-08	-1.55E-07	-1.35E-07	-1.01E-08	-1.37E-07	-4.50E-08
	-8.66E+06	-6.71E+06	-9.06E+07	-6.45E+06	-7.42E+06	-9.86E+07	-7.28E+06	-2.22E+07
	0.8334	0.76431	0.61916	0.80161	0.78056	0.71452	0.76908	0.98395

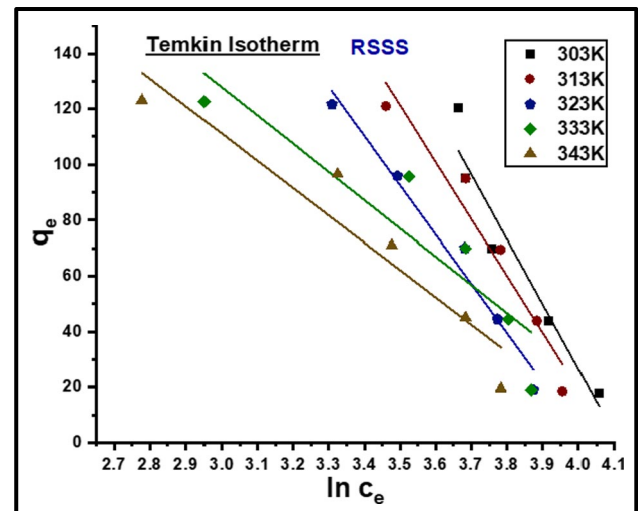


Fig. 21 RSSS Temkin model fit statistics

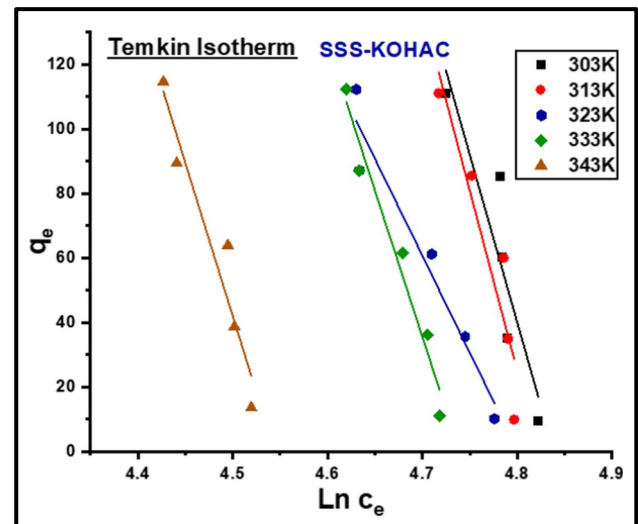
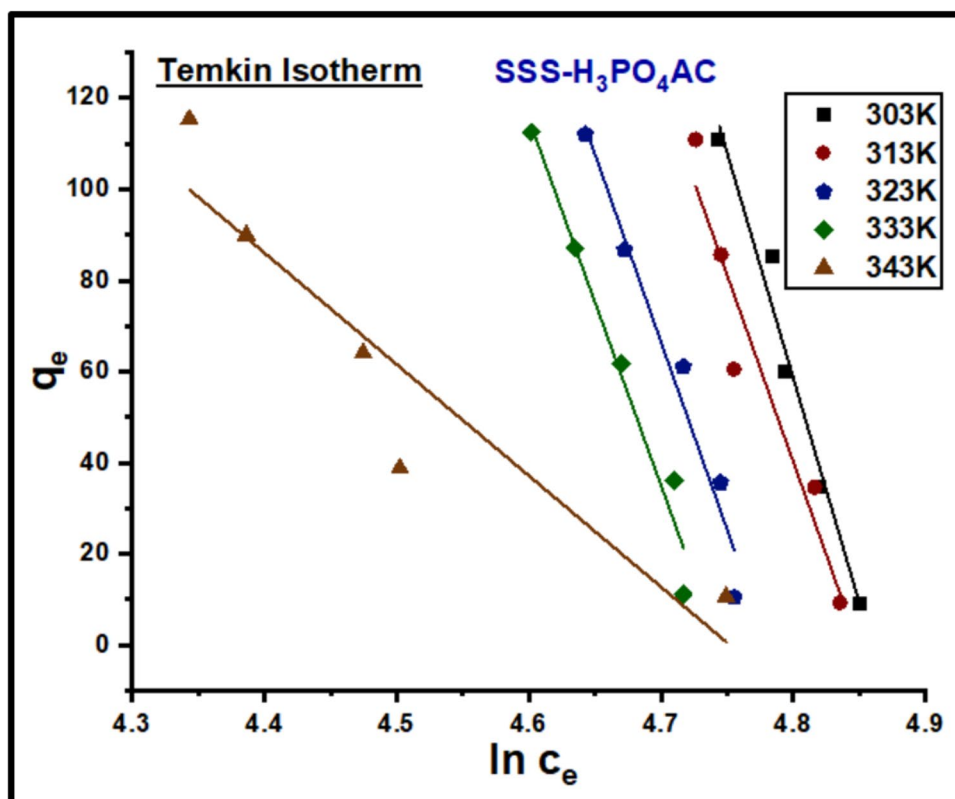


Fig. 22 SSS-KOHAC Temkin model fit statistics

free energy (ΔG°) confirmed spontaneity, while negative enthalpy (ΔH°) supported exothermic behaviour (Ebelegi et al. 2020). The stronger the adsorbent–adsorbate bond, the greater the negative adsorption potential. The higher adsorption energy values of SSS-H₃PO₄AC compared with SSS-KOHAC resulted in more effective uptake and potentially selective adsorption. H₃PO₄ activation introduced surface functional groups that enhanced selectivity and affinity, whereas KOH activation primarily generated micropores (Karapınar 2022; Sahoo & Mishra 2023).

Fig. 23 SSS-H₃PO₄AC PSO model fit statistics



3.11 Isotherm model evaluation

Adsorption isotherms are a fundamental prerequisite for designing adsorption systems (Rajeshkannan et al. 2011). The amount of material extracted (adsorbed) from the liquid phase by a unit mass of activated material (adsorbent) depends on the solute concentration at a fixed temperature. Table 10 summarises the constants and fitness for the TI, EI, DRI, FI, and LI models, while Figs. 21, 22, 23 present the fit statistics. The regression coefficient (R^2) was used to identify the model that best represents the adsorption process. For RSSS, the LI ranged from 7.36 to 21.55 mg/g and increased with temperature, although the model was poorly fitted. Ahmed et al. (2025) noted that LI typically ignores heterogeneity in modified surfaces. RSSS adsorbed more material at higher temperatures, but modest to low R^2 values indicated poor LI applicability, particularly for chemically modified adsorbents. The FI model also failed to fit the adsorption phase, as $1/n$ and n values were negative, despite adsorption being favourable when $0 < 1/n < 1$. In the TI model, negative BT values indicated exothermic adsorption, while KT values were consistent and positive, with RSSS showing slightly higher affinity. H₃PO₄ activation proved crucial for adsorption, as reflected by the high R^2 values, particularly for SSS-H₃PO₄AC. The EI model

fitted well, with $R^2 > 0.9$, although its negative K_e and q_m values suggested unfavourable adsorption despite the strong statistical fit. The D-R isotherm yielded extremely low q_m values, indicating very limited adsorption capacity. Overall, the TI and EI models were the most reliable. The TI model consistently produced high R^2 values (up to 0.97), demonstrating strong linearity and appropriate adsorption energies with moderate BT and KT values. Rahman et al. (2021) emphasised that TI is robust in systems with moderate energy heterogeneity, particularly when R^2 exceeds 0.9. While EI showed promise as an empirical model with good data fit, its negative constants limited mechanistic interpretation. Thus, the TI model was identified as the most suitable for mechanistic insight into the adsorption process.

3.12 Kinetic modeling evaluation

The kinetic model of MB adsorption provides insight into the solute uptake rate during the reaction. Kinetic modeling demonstrated that pseudo-second-order (PSO) adsorption of methylene blue on the adsorbents was satisfied at concentrations ranging from 200 to 1000 mg/L. Table 11 shows that the PSO model provided an excellent fit, as indicated by consistently high correlation coefficients (R^2).

Table 11 MB adsorption kinetic model

K-M	Parameters	Initial concentration(C_0)									
		200(mg/L)		400(mg/L)		600(mg/L)		800(mg/L)		1000(mg/L)	
		SSS	S-KOHAC	S-H ₃ PO ₄ AC	SSS	S-KOHAC	S-H ₃ PO ₄ AC	SSS	S-KOHAC	S-H ₃ PO ₄ AC	SSS
FO	K_1 (1/min)	-0.0362	-0.000609	-0.01116	-0.0372	-0.0007177	-0.0118	-0.0409	-0.00499	-0.0135	-0.0004862
	$T^{1/2}$	-19.174	-1138.45	-62.10996	-18.653	-965.82	-58.74129	-16.968	-138.91	-51.34424	-1425.65
	R^2	0.62798	0.38567	0.8491	0.65448	0.2873	0.78457	0.73893	0.12952	0.72067	0.11757
PSO	q_{ecal} (mg/g)	18.822	17.9759	14.03903	43.3276	42.95533	38.61004	66.9792	66.4011	62.5782	93.02326
	K_2 (1/min)	-0.0269	-0.690781	-0.01947	-0.0201	-0.857905	-0.0158	-0.0136	-0.76458	-0.01288	4.040138
	R^2	0.98666	0.99995	0.99083	0.99739	0.99998	0.9973	0.9993	0.99619	0.99774	0.99999
AM	N	-0.4588	-0.01397	-0.23639	-0.2377	-0.0095	-0.13575	-0.4314	-0.07912	-0.15739	-0.03826
	K	8.78082	1.35531	2.416427	4.58344	1.77354	2.45852	13.3423	3.00937	3.75257	5.58179
	R^2	0.65415	0.52829	0.71407	0.6669	0.43572	0.66645	0.76516	0.21831	0.61754	0.29643
IPD	K_{int} [mg/(g.min ^{1/2})]	-1.1444	-0.04739	-0.92904	-1.2263	-0.05756	-1.00328	-1.4746	-0.5085	-1.21153	-0.04246
	C (mg/g)	27.1121	18.30547	21.65051	52.3782	43.33612	46.89492	78.3403	69.46681	72.56232	93.23539
	R^2	0.57973	0.45832	0.78678	0.62029	0.35886	0.72054	0.75574	0.17517	0.647	0.171

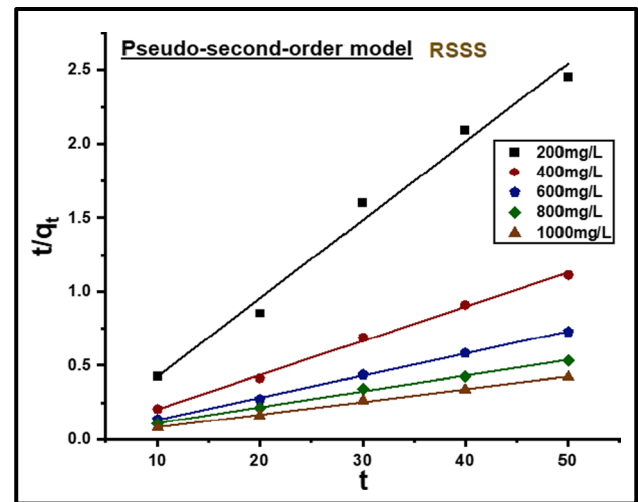
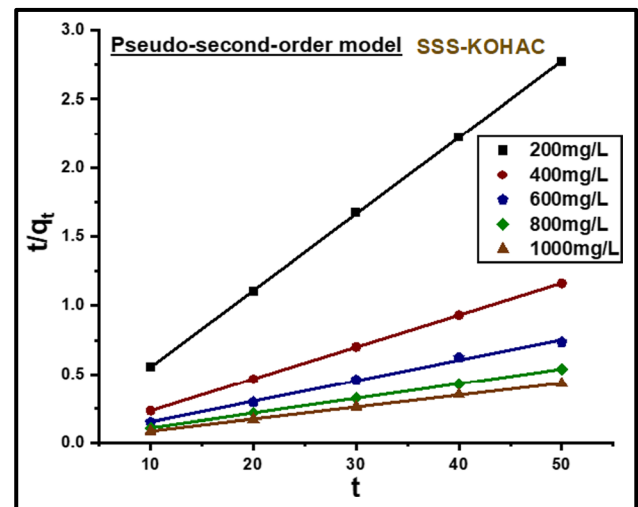
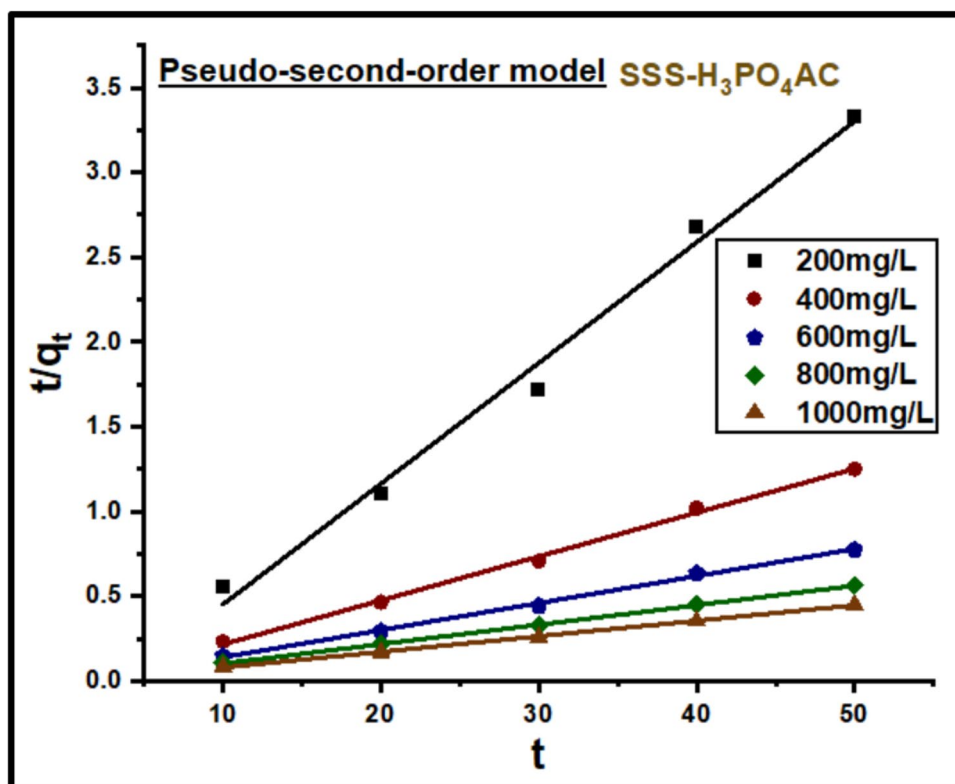
**Fig. 24** RSSS PSO model fit statistics**Fig. 25** SSS-KOHAC PSO model fit statistics

Figure 24 presents the PSO fit statistics for RSSS, while Figs. 25 and 26 show the statistics for SSS-KOHAC and SSS-H₃PO₄AC, respectively. The adsorption process of SSS-KOHAC yielded the best model fitness, with every point along the line well aligned. The PSO model fitted the data most effectively, with R^2 values ranging from 0.986 to 0.9999. The PSO model also revealed that as solute concentration increased, the calculated equilibrium adsorption capacity ($q_{e(cal)}$) rose correspondingly. Table 11 displays the constants and R^2 values for each adsorbent system, along with the results of the IPD, AM, PSO, and FO kinetic models. SSS-H₃PO₄AC consistently exhibited high R^2 values in both the PSO and IPD models. Phosphoric acid activation enhanced adsorption kinetics and capacity by increasing surface area and introducing

Fig. 26 SSS-H₃PO₄AC PSO model fit statistics**Table 12** Comparative efficiency of biomass-based adsorbents for methylene blue dye removal

Agro-waste adsorbent	Target pollution	% Removal	References
Sandbox seed shell (KOH)	Methylene blue	98	This study
Sandbox seed shell (H ₃ PO ₄)	Methylene blue	99	This study
Banana peels	Methylene blue	99.6	Ali (2018)
Waste peach wood branches	Methylene blue	94.91	Zhang et al. (2024)
Seaweed	Methylene blue	100	Kumar et al. (2022)
Tea-waste adsorbent	Methylene blue	99	Salameh et al. (2025)
Rice husk (torrefied)	Methylene blue	84	Hummadi et al. (2022)
Orange & lemon peel	Methylene blue	98	Ramutshatsha-Makh-wedzha et al. (2022)
Bamboo-derived biochar (KOH)	Methylene blue	99.5	Ge et al. (2023)
Tamarind seed powder	Methylene blue	97.5	Kotla et al. (2025)
Rice husk	Methylene blue	95.2	Kotla et al. (2025)
Wheat hull (Siyez variety)	Methylene blue	96.8	Yetgin & Amlani (2025)
Rice hull (Sarı Kılçık variety)	Methylene blue	94.3	Yetgin & Amlani (2025)
Garlic stalk (Taşköprü variety)	Methylene blue	98.1	Yetgin & Amlani (2025)
Lignocellulosic biomass (generic)	Methylene blue	99.2	G.P. Avinash et al. (2025)

functional groups. By contrast, the comparatively lower R^2 values of RSSS indicated that heterogeneous active sites and mixed surface energies caused deviations from ideal monolayer dynamics. As the surface chemistry became more homogeneous, the R^2 values of the activated carbons (SSS-KOHAC and SSS-H₃PO₄AC) increased, confirming their superior adsorption performance.

3.13 Comparison with other adsorbents

Table 12 compares the findings of this study with those of other investigations on methylene blue uptake using readily available and low-cost agricultural waste adsorbents. The comparison was based on the percentage of methylene blue removed by the adsorbents. The results of the present study

indicate that the performance of the other adsorbents was broadly similar. The relatively high percentage removals of 98% and 99% within a short contact time of 30 min demonstrated that SSS-KOHAC and SSS-H₃PO₄AC are efficient, low-cost adsorbents for methylene blue removal in aqueous solution.

4 Conclusion

The study successfully demonstrated that methylene blue (MB) in aqueous solutions can be effectively removed using activated carbon derived from sandbox seed shell (SSS), an underutilised agro-waste. SSS-KOHAC and SSS-H₃PO₄AC achieved high removal efficiencies of 98% and 99%, respectively. Activation with KOH and H₃PO₄ significantly enhanced the textural and chemical properties of the raw biomass, improving adsorption performance by increasing surface area, porosity, and the incorporation of functional groups. The mesoporous structure and phosphate-derived surface chemistry of SSS-H₃PO₄AC supported strong interactions with the cationic dye, resulting in improved adsorption capacity and energy. SSS-KOHAC exhibited a high degree of surface area and microporosity, making it particularly effective at low adsorbent dosages. The adsorption process for both activated samples was exothermic and spontaneous. Equilibrium analysis revealed that the Temkin isotherm model best described the adsorption mechanism. Kinetic measurements confirmed that the process followed the pseudo-second-order (PSO) model, suggesting chemisorption. Thermodynamic modelling further validated the feasibility and spontaneity of MB adsorption on the activated carbons.

Future research should focus on bridging existing knowledge gaps to determine the lifespan and economic feasibility of activated carbons in real-world applications. This includes analysing the regeneration and reusability of SSS-derived activated carbons through repeated adsorption cycles. The effectiveness of these adsorbents against a broader range of contaminants, such as heavy metals, other dyes, and newly identified organic pollutants in industrial wastewater, should also be investigated to provide a more comprehensive assessment. In addition, advanced modelling techniques could be employed to optimise activation process parameters, maximising yield and adsorption performance while minimising chemical and energy consumption. Scaling up production and conducting pilot-scale studies will be essential to translate laboratory success into practical technologies for large-scale water treatment.

This work is significant because it advances the principles of sustainable environmental management and the circular economy. It provides a dual solution: addressing

solid waste management by converting agro-waste into a valuable product, while simultaneously offering a method to treat dye-contaminated wastewater through the valorisation of sandbox seed shell, a readily available and low-cost agricultural waste. The development of such eco-friendly, high-performing, and inexpensive adsorbents presents a viable alternative to costly commercial activated carbons, making clean water treatment more accessible, particularly in developing nations. In summary, this study demonstrates that locally sourced agro-waste biomass holds considerable promise for creating sustainable solutions to contemporary environmental pollution challenges, paving the way for more affordable and environmentally responsible water purification systems.

Acknowledgements The authors would like to express their gratitude to the Ahmadu Bello University Zaria Chemical Engineering Department for providing the necessary materials, facilities and equipment. Additionally, Federal Polytechnic Nekede is sincerely appreciated for granting access to the campus environment for the gathering of sandbox seed shells.

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Data curation; Formal analysis; Visualization; Writing – original draft.

Macdonald Chinyere Sunday: Investigation; Methodology; Resources; Data curation; Validation.

Uzochukwu Franklin Onwuka: Formal analysis; Writing – review & editing.

Larry Jacob King: Investigation; Visualization; Writing – original draft.

Maxwell Chukwuma Anukwonke: Methodology; Validation; Writing – review & editing.

Onyeyirichi Gold Uzokwe: Methodology; Validation; Writing – review & editing.

Funding This research received no external funding.

Data availability Data sets generated during the current study are available from the corresponding author on reasonable request.

Declarations

Competing interests 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.

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