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ADSORPTION STUDIES OF ACTIVATED CARBON DERIVED FROM AGRICULTURAL WASTE FOR GLYPHOSATE REMOVAL FROM WATER

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ABSTRACT

This study investigated the adsorption efficiency of activated carbons derived from corn cob (CCAC), millet husk (MHAC), and rice straw (RSAC) for the removal of glyphosate from aqueous solutions. The adsorbents were prepared via hydrothermal treatment followed by chemical activation and carbonization. FTIR analysis confirmed the presence of functional groups such as hydroxyl, carboxyl, and carbonyl that facilitated hydrogen bonding and electrostatic interactions with glyphosate. Batch adsorption experiments were performed to evaluate the effects of contact time, pH, adsorbent dosage, initial glyphosate concentration, and temperature on adsorption performance. The maximum removal efficiencies recorded were 94.2% (CCAC), 92.5% (MHAC), and 91.0% (RSAC) at optimized conditions. The equilibrium data fitted well to both Langmuir (R^2 = 0.936-0.965) and Freundlich (R^2 = 0.972-0.990) isotherm models, indicating favorable monolayer and multilayer adsorption processes. The kinetics of glyphosate uptake were best described by the pseudo second order model (R^2 = 0.950-0.962), suggesting chemisorption involving valence interactions between adsorbent surfaces and glyphosate molecules. Overall, CCAC demonstrated the highest adsorption capacity (35.56 mg/g), indicating its potential as a low cost and sustainable adsorbent for glyphosate removal from contaminated water remediation.

Key words: Activated Carbon, Glyphosate, Adsorption, Isotherm, Kinetics

1.0 INTRODUCTION

Glyphosate N-(phosphono-methyl) glycine remains the most extensively applied herbicide globally, valued for its broad-spectrum and systemic weed control but increasingly scrutinized for its environmental and health implications (Duke and Powles 2023; Alimentarius 2013). Its frequent detection in surface and groundwater, along with degradation products such as amino methyl phosphonic acid (AMPA) and surfactant residues like octyl phenol ethoxylate amine (OPEA) underscores its persistence and mobility within the aquatic environment (Mijangos-Novelo *et al.,* 2021; Salas-Perez *et al.,* 2021). Owing to its classification as a probable human carcinogen (International Agency for Research on Cancer (IARC) 2015) and documented acute and chronic toxicological effects including irritation, gastrointestinal disturbances and potential hepatic, renal, reproductive and genotoxic impacts, regulatory bodies such as World Health Organization (WHO) have established maximum contaminant limits to mitigate exposure risks (Heap 2022; Gali 2024; WHO 2022).

These concerns have stimulated the development of effective and sustainable water treatment technologies for glyphosate and other contaminants removal such as membrane filtration, electrochemical treatments, photocatalysis, ozonolysis and other advanced techniques have been explored with adsorption remains particularly attractive due to its operational simplicity, cost-effectiveness, and high removal efficiency (Tahir *et al.,* 2020; Wang *et al.,* 2022; Rizwan *et al.,* 2021; Li *et al.,* 2024). In particular, activated carbons derived from agricultural residues offer a low-cost,

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renewable, and environmentally sound alternative, benefiting from their carbon-rich composition, high volatile content, and ability to form well-developed porous structures while also alleviating waste-disposal challenges (Opia *et al.,* 2020).

This study therefore explored the potentials of these wastes into useful activated carbon which was applied for the glyphosate removal from water using batch adsorption method while focusing on adsorption efficiency, adsorption isotherm and kinetics.

2.0 MATERIALS AND METHODS

2.1 Sample and Chemicals

The samples of corn cob, millet husk and rice straw were collected in Bakolori Irrigation Project, Talata Mafara, Zamfara State, Nigeria. The samples were collected in polyethene bag, labelled and brought to laboratory for further analysis. All chemicals used for this study were analytical reagent grade, the glyphosate standard were purchased from Sigma Aldrich while H_2SO_4, HNO_3 and $Zncl_2$ were obtained from Central drug house (CDH). A 1000 ppm glyphosate stock solution were prepared by dissolving 250mg of glyphosate standard in distilled water and stored at 4 °C, while calibration standards (5-35ppm) and adsorption working solutions (5-200ppm) were subsequently prepared from the stock solution using serial dilutions based on the formula $C_1V_1 = C_2V_2$.

2.2 Preparation and Characterization of Activated Carbon

Synthesis was carried out according to the method reported by Adekola *et al.,* (2017) with some modifications. The collected samples were washed with clean water to remove any impurities, dust, filth particles or other fine dirt particles that may be stocked in the surface of the samples. The washed samples were sun dried for three days before subjecting to crushing using local pestle and mortar. In order to increase surface area of the samples, the samples were further grinded to powder form using blender (KitechenAid: K1503-). The samples were later subjected to hydrothermal pretreatment under hot plate (model: EW-04805 Series) using water at 200°C for 3 hours. Further, chemical activation using H_2SO_4, HNO_3 and $Zncl_2$ was carried out prior to carbonization using muffle furnace (model: PAP-5054). The yield of the activated carbon was calculated using the equation (1). Brunauer-Emmett-Teller (BET) analyzer (Horiba SA-9600) and Fourier Transform infrared (FTIR) analyzer (Shidmazu: IRTracer-100) were further employed to study the porosity and functional groups of the activated carbon synthesized.

$$\text{Yield (\%)} = \frac{final\ weight\ of\ actvated\ carbon}{initial\ weight\ of\ he\ sample}\ X\ 100 \ldots\ldots\ldots\ eqn.(1)$$

2.3 Batch Adsorption Experiments

Batch adsorption experiments for glyphosate removal were conducted following a modified method of Nyijime and Ayuba (2021). Precisely 0.05g of each activated carbon (CCAC, MHAC, and RSAC) was added to 25mL of glyphosate solution in 250mL conical flasks, sealed and agitated at 150rpm using a mini-incubator shaker (Model: MIDSCI IN20-H). After equilibrium, the mixtures were filtered through Whatman filter paper and the residual glyphosate concentrations were determined using a UV-Visible spectrophotometer (Model: Shimadzu UV-mini1240) at a wavelength of 204nm. The effects of contact time (5-240 min), pH (2-10), adsorbent dosage (0.05-4.0 g), glyphosate concentration (5-200ppm) and temperature (20-60 °C) on adsorption efficiency were systematically evaluated by varying one parameter at a time while keeping others constant. Solution pH was adjusted with 0.1 M HCl or NaOH and monitored using a calibrated pH meter. The amount of glyphosate adsorbed at equilibrium (q_e) and percentage removal (%R) were calculated using equations (2) and (3). Adsorption isotherm (Langmuir and Fredrich) and

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adsorption kinetics (Pseudo first and Pseudo second order) were investigated.

$$(qe) = \frac{(Co - Ce)V}{m} \ldots\ldots\ldots eqn.(2)$$

Where: (qe) is amount adsorbed, Co: initial concentration (mg/L), Ce: final concentration (mg/L)

V is the volume of glyphosate solution (L), m: mass of activated carbon (g)

the percentage removal (%R) were calculated using the formular:

$$(\%R) = \frac{(Co - Ce)}{Co} X\ 100 \ldots\ldots\ldots eqn.(3)$$

3.0 RESULTS AND DISCUSSION

3.1 Characterization of Activated Carbon

The porosity of activated carbon synthesized using H_2SO_4, HNO3, and $ZnCl_2$ as activation agents presented in (table 3.1) demonstrates variations in surface area, pore volume, pore size distribution, Langmuir surface area, and iodine number. CCAC exhibited the highest specific surface area (556.47 m²/g) and total pore volume (0.476 cm³/g), reflecting a well-developed porous structure dominated by mesopores (0.266 cm³/g) and micropores (0.175 cm³/g), which favors both surface adsorption and intraparticle diffusion (Zhang et *al.,* 2023). The high lignin content of corn cob also supports the formation of a stable and well-developed porous matrix (Nyamful *et al.,* 2021). MHAC showed a comparatively lower surface area (387.05 m²/g) and pore volume (0.353 cm³/g) but maintained a balanced micro-mesoporous structure, suggesting moderate accessibility of active sites (Adeola and Afolabi 2022). RSAC recorded an intermediate surface area (434.70 m²/g) and pore volume (0.388 cm³/g) with dominant mesopores (0.224 cm³/g), which enhance adsorbate mobility (Mansor *et al.,* 2024). The high Langmuir surface area and iodine number of CCAC (4,026.22 m²/g and 932.43 mg/g, respectively) further confirm its superior adsorption capacity due to abundant microporous and high energy adsorption sites (Ismail and Babarinde 2021). Overall, the trend CCAC > RSAC > MHAC in surface area and iodine number demonstrates that CCAC possesses the most developed porosity and hence the greatest potential for glyphosate adsorption (Saka 2012; Azeez *et al.,* 2024).

FTIR analysis (Figure 3.1) revealed functional groups associated with adsorption performance. All samples displayed C-H bending at 670-870 cm^{-1}, confirming carbon network formation, and C-O stretching at 1050-1150 cm^{-1} associated with alcohols, phenols, and ethers, which improve hydrophilicity and hydrogen bonding (Gonzalez-Garcia, 2021). CCAC and RSAC exhibited N-H bending at 1550-1650 cm^{-1}, enabling strong hydrogen bonding with glyphosate's carboxyl and phosphonic groups (Gao & Wang, 2022). Carbonyl (C=O) stretching at 1700-1750 cm^{-1} indicated carboxyl functionalities that enhance electron donor–acceptor interactions with herbicides (Hupian et *al.,* 2024). Peaks at 2100-2250 cm^{-1} (C≡C/C≡N) suggested nitrile groups that facilitate adsorption of ionic pesticides (Hassan *et al.,* 2021). The O=C=O band at 2340 cm^{-1} indicated carbonate species providing basic sites suitable for acidic pesticides such as glyphosate and paraquat (Gonzalez-Garcia, 2021). Aliphatic C-H stretching at 2850-2950 cm^{-1} in CCAC suggested hydrophobic domains capable of weak van der Waals interactions with non-polar contaminants (Gao *et al.,* 2020).

Table 3.1 Result of Porosity of Activated Carbon

Sample	Surface Area (m^2/g)	Pore Volume (CM^3/g)	Pore-size distribution (CM^3/g)			Langmuir Surface Area (m^2/g)	Iodine Number (mg/g)
	S_{BET}		V_{Micro}	V_{Meso}	V_{Macro}		
CCAC	556.473	0.476	0.175	0.266	0.128	4,026.220	932.428
MHAC	387.048	0.353	0.153	0.214	0.114	2,095,585	913

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RSAC	434.702	0.388	0.0155	0.224	0.115	432.702	834.62

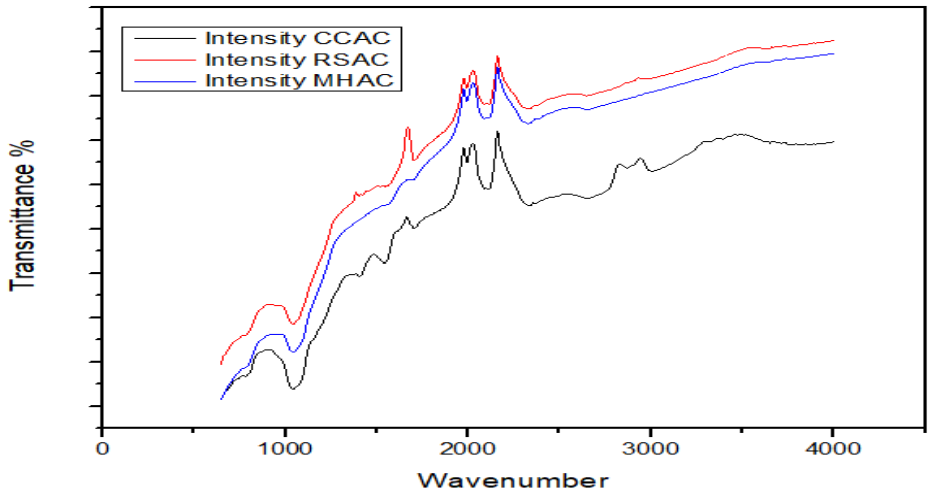

Figure 3.1: FTIR Spectrum for CCAC, MHAC and RSAC

3.2 Adsorption Study

3.2.1 Effect of Contact Time

In the contact time graph (figure 3.2), all the adsorbents show rapid increment in glyphosate adsorption with increase of time up to 60min and them slow down indicating equilibrium is achieved with CCAC achieving the highest removal (71.6 %) followed by MHAC (65.5%) and RSAC (61.2%). When the time was extended beyond the 60 min, there was no significant changes in the glyphosate adsorption on all the adsorbents, indicating that there were no more porous networks for further adsorption to take place (Lima-silva *et al.,* 2023). This result correlate to the findings of Kalsoom *et al.,* (2024) who noted pesticides uptake by the sorbents was too quick at initial time because of the presence of abundant vacant positions for sorption, but as the contact time prolongs, the adsorption rate was stabilizes. Thus, contact time of 60 min was found to be appropriate for maximum adsorption of glyphosate and was selected for the remaining experiments.

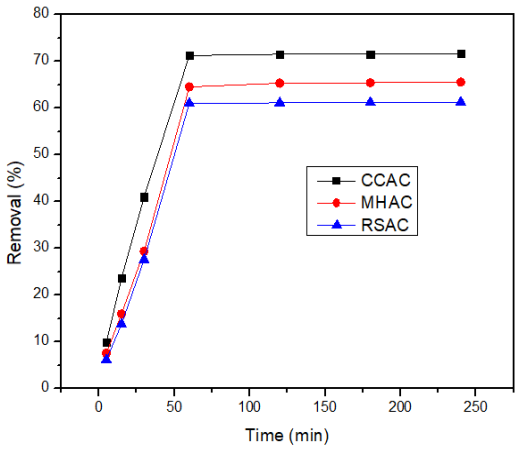

Figure 3.2 Effect of time on adsorption adsorption of glyphosate onto CCAC, MHAC and RSAC

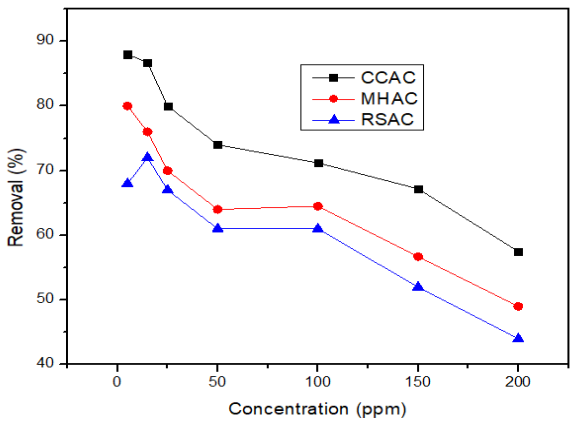

Figure 3.3 Effect of Concentration on glyphosate onto CCAC, MHAC and RSAC

3.2.2 Effect of Concentration

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The effect of glyphosate concentration on CCAC, MHAC and RSAC adsorbents is given in figure 3.3. All adsorbents showed an increase in removal of glyphosate at low concentrations, peaking before gradually declining at higher concentrations. CCAC maintained the highest glyphosate removal 88% at 5ppm prior to declining to 57.5% at 200ppm. The same trend was observed for MHAC 80% and 49%, RSAC 72% and 44% at 5ppm and 200ppm respectively. The optimum adsorption at the low adsorbate concentrations was due to the availability of vacant sites on the surface of the adsorbent (Nyamful *et al.,* 2021). Other findings reported that increase in concentration of the adsorbate brings about increase in competition of adsorbate molecules for available binding sites on the surface of the adsorbent (Ali *et al.,* 2020). More so, the decrease in glyphosate removal as the concentration increases indicated the surface approaches saturation hence, removal dropped sharply beyond a certain concentration (Benjelloun *et al.,* 2021). The higher uptake of glyphosate by CCAC, compared to the other adsorbent may be associated with its higher surface area (556.47 m²/g) providing abundant accessible adsorption sites for the glyphosate. The same trend was reported by (Njokua and Hameeda 2011) for the adsorption of 2,4-dichlorophenoxyacetic acid and Bromopropylate adsorption by Ourania *et al.,* (2010) onto CCAC.

3.2.3 Effect of Dosage

Figure 3.4 presents the effect of dosage on glyphosate adsorption for CCAC, MHAC, and RSAC. The removal of glyphosate increased rapidly by increasing of adsorbent dosage from 0.05-0.1g. However, no significant glyphosate removal was recorded as adsorbent dosage increased from 1-4g. Interestingly, both adsorbents CCAC (94.2%), MHAC (92.5%) and RSAC (91%) showcase remarkable removal efficiency. The higher removal efficiency recorded highlighted the presence of high surface area and number of available active binding sites for glyphosate to adsorbed (Mohammadi *et al.,* 2024). The slower removal recorded at 1-4g indicated that equilibrium is attained, hence, slower removal recorded could be due to particle aggregation and overlapping of active sites (Kuo *et al.,* 2023). The observed trend is similar to other findings who reported that increase in adsorbent dosage increases the active sites and surface area are abundantly exposed to the pesticides, leading towards reduction in competition between water molecules and pesticides, enhancing the capacity of adsorption (Nedjai *et al.,* 2021).

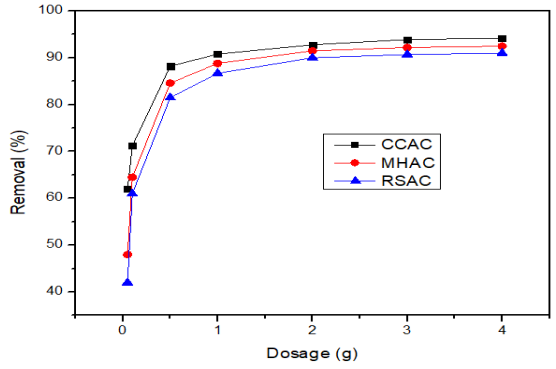

Figure 3.4 Effect of dosage on adsorption of glyphosate onto CCAC, MHAC and RSAC RSAC

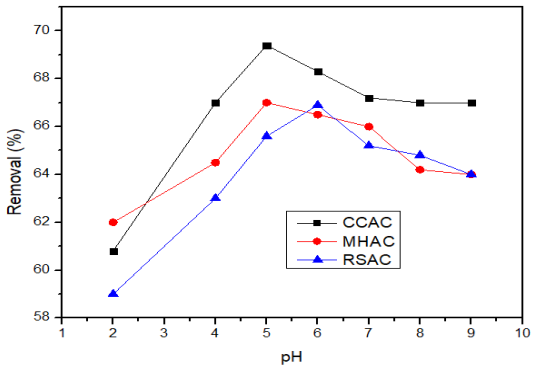

Figure 3.5 Effect of Ph on adsorption of glyphosate onto CCAC, MHAC and

3.2.4 Effect of pH

Figure 3.5 shows the effect of pH on glyphosate adsorption for CCAC, MHAC,

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and RSAC. The removal of glyphosate increased steadily from pH 2 to pH 5-6, reaching maximum adsorption of 69.4% for CCAC, 67.0% for MHAC, and 66.9% for RSAC. Beyond pH 6, removal efficiency decreased progressively across all adsorbents. The observed pH-dependent adsorption can be associated with changes in glyphosate speciation and adsorbent surface charge. At low pH (<4), glyphosate is mostly protonated due to its amine group (NH_2) and competes with hydrogen ions for adsorption sites, resulting in lower adsorption efficiency (Kumar *et al.,* 2022). Maximum removal occurred at mildly acidic pH (5-6), where glyphosate exists as a zwitterion, allowing stronger interactions with active adsorption sites (Okada *et al.,* 2016). At neutral to alkaline pH (>7), glyphosate becomes fully anionic due to deprotonation of its carboxylic (COOH) and phosphonic (PO_3H_2) groups, leading to electrostatic repulsion between the negatively charged adsorbent surfaces and glyphosate molecules (Ambursa *et al.,* 2022).

3.2.5 Effect of Temperature

The effect of temperature on glyphosate adsorption on to CCAC, MHAC and RSAC is shown in figure 3.6. CCAC achieves the highest removal across all temperatures peaking (77%) at 25 °C before steadily declining to 54% at 60 °C. MHAC shows similar behavior, with a maximum (71%) at 25 °C and a drop to 53% at 60 °C while RSAC consistently has the lowest removal, peaking (66%) at 25 °C and falling to 50% at 60 °C. The decline in adsorption with temperature suggests glyphosate is physisorption dominates, where van der Waals forces and hydrogen bonding are the primary interaction mechanisms between glyphosate molecules and the carbon surface with possible minor chemisorption contributions from functional groups (Lima-Silva *et al.,* 2023).

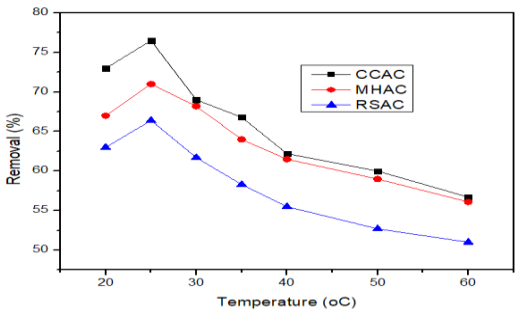

Figure 3.6 Effect of temperature on adsorption of glyphosate onto CCAC, MHAC and RSAC

3.3 Adsorption Isotherm

3.3.1 Langmuir isotherm Model

From the data presented in table 3.2 and figure 3.7, it can be observed that both adsorbents used for this study shows significant capacity in adsorption of glyphosate with Langmuir isotherm fitting the adsorption data well R2: 0.936, 0.983 and 0.965 for CCAC, MHAC and RSAC respectively with RSAC fitting the most. The value for qm obtained were CCAC 35.56 mg/g, MHAC 38.022 mg/g and RSAC 33.921 for glyphosate removal is higher than the maximum adsorption capacities 10.1883 mg/g reported by Kalsoom *et al.,* (2024) for removal of atrazine. The KL value calculated were CCAC 0.021mg/g, MHAC 0.0349 mg/g and RSAC 0.0175 mg/g hence indicating good interaction between glyphosate and CCAC, MHAC and RSAC at 25°C. The KL value in this study is closely in concordance with 0.040 mg/g, 0.037 mg/g, 0.020 mg/g and 0.036 mg/g reported by Sivasamy *et al.,* (2013) in adsorption of chromium ion. The dimensionless separation factor RL were CCAC 0.310, MHAC 0.222 and RSAC 0.362. Interestingly, all were >1, suggesting that the glyphosate adsorption onto CCAC, MHAC

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and RSAC is favorable (Al-Ghouti and Da'ana 2020). This further confirms the fitness of the data of Langmuir isotherm model.

3.3.2 Freundlich isotherm model

From the data presented in the table 3.2 and figure 3.8, it can be observed that Freundlich isotherm model provided the best fit to the equilibrium data R2 CCAC 0.990, MHAC 0.988 and RSAC 0.972, indicating that heterogenous multilayer adsorption dominates glyphosate uptakes across the adsorbents (Idoko and Muhammad 2020). The K_f (Freundlich adsorption capacity) obtained were CCAC 1.077, MHAC 2.254, RSAC 1.505 all were moderate and were higher compared to 0.63 mg/g and 0.0599 mg/g reported by Krupa and Hemangi (2019) For removal of sulfate and nitrile respectively. For coefficient n (adsorption intensity) CCAC 1.185, MHAC 1.337 and RSAC 1.131. Interestingly, all were >1 indicating adsorption was favorable (favorable (Al-Ghouti and Da'ana 2020).

Table 3.2 Adsorption isotherm parameters for the adsorption of glyphosate onto CCAC, MHAC and RSAC

Isotherm Model	Activated Carbon	Parameter	Numerical Values
Langmuir	CCAC	qm (mg/g)	35.561
		K_L (L/mg)	0.0216
		R_L	0.316
		R^2	0.936
Freundlich		n	0.715
		K_F (mg/L)	1.077
		R^2	0.990
Langmuir	MHAC	qm (mg/g)	38.022
		K_L (L/mg)	0.0349
		R_L	0.222
		R^2	0.938
Freundlich		n	0.670
		K_F (mg/L)	2.254
		R^2	0.988
Langmuir	RSAC	qm (mg/g)	33.921
		K_L (L/mg)	0.0175
		R_L	0.362
		R^2	0.965
Freundlich		n	0.779
		K_F (mg/L)	1.505
		R^2	0.972

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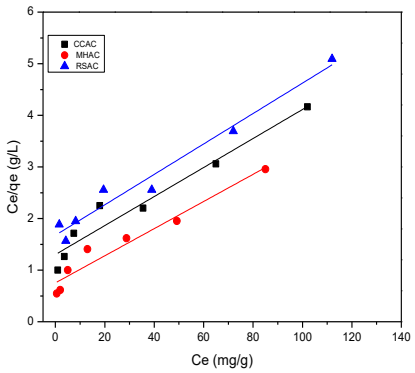

Figure 3.7 plot of Linearized Langmuir for the for the adsorption of glyphosate onto CCAC, MHAC, CCAC, MHAC and RSAC

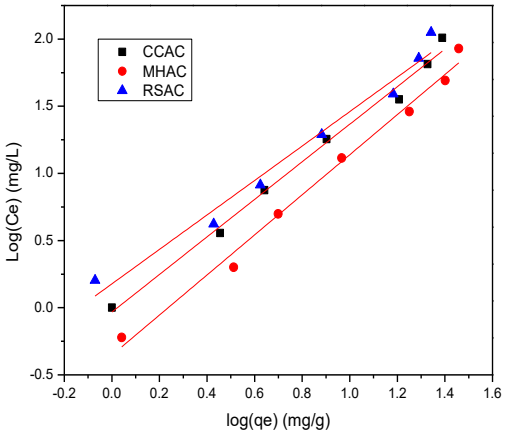

Figure 3.8 plot of Linearized Freundlich adsorption of glyphosate onto RSAC

3.4 Adsorption Kinetics

The adsorption kinetics of glyphosate onto CCAC, MHAC and RSAC were evaluated using the pseudo first order and pseudo second order models to elucidate the mechanism and rate controlling steps of the adsorption process. The corresponding kinetic parameters are presented in Table 3.3, while Figures 3.9 and 3.10 show the linearized plots of the models.

The pseudo first order model which assumes that the rate of adsorption is directly proportional to the number of unoccupied sites, showed relatively poor correlation coefficients (R^2 = 0.5803, 0.5514, and 0.5517 for CCAC, MHAC, and RSAC respectively). Additionally, substantial deviations between experimental adsorption capacity (qe) and calculated adsorption capacity qe(cal) were observed with percentage deviations of 47%, 16.7%, and 93% respectively. These discrepancies indicate that the pseudo first model inadequately describes glyphosate adsorption onto the studied adsorbents (Kumar *et al.,* 2022). In contrast, the pseudo second order model yielded significantly higher correlation coefficients (R^2 = 0.9621, 0.9508, and 0.9508 for CCAC, MHAC, and RSAC, respectively) and close agreement between experimental and calculated adsorption capacities, with maximum deviations of 5.1%, 5.3%, and 7.4%. This strong correlation suggests that the pseudo second model best describes the adsorption kinetics, implying that the rate limiting step is primarily chemisorption involving valence forces through electron sharing or exchange between glyphosate molecules and the surface functional groups of the activated carbons (Nguyen 2023). K_1 which presents rate at which adsorption occurs is higher in pseudo first order given compared to lower values obtained for pseudo second order hence, indicating second kinetic order is favorable in the adsorption of glyphosate (Kalsoom *et al.,* 2024).

Overall, the predominance of pseudo second order kinetics suggests that glyphosate adsorption onto CCAC, MHAC, and RSAC is governed by chemisorption interactions. This highlights the strong affinity of the adsorbents for glyphosate and confirms their suitability for the removal of glyphosate pesticides from aqueous systems (Aminu *et al.,* 2022).

Table 3.3 Adsorption Kinetics parameters for the adsorption of glyphosate onto CCAC, MHAC and RSAC

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Kinetics Model	Activated Carbon	Parameter	Numerical Values
Pseudo First Order	CCAC	qe(exp)	21.25
		qe(cal)	14.376
		K_1 min^{-1}	2.3056
		R^2	0.5803
	MHAC	qe(exp)	19.5
		qe(cal)	16.71
		K_1 min^{-1}	2.301
		R^2	0.5514
	RSAC	qe(exp)	25.2
		qe(cal)	13.025
		K_1 min^{-1}	2.3003
		R^2	0.5517
Pseudo Second Order	CCAC	qe(exp)	21.25
		qe(cal)	20.21
		K_2 min^{-1}	0.00114
		R^2	0.9621
	MHAC	qe(exp)	19.5
		qe(cal)	20.65
		K_2 min^{-1}	0.00172
		R^2	0.9508
	RSAC	qe(exp)	21.48
		qe(cal)	19.9
		K_2 min^{-1}	0.00106
		R^2	0.9508

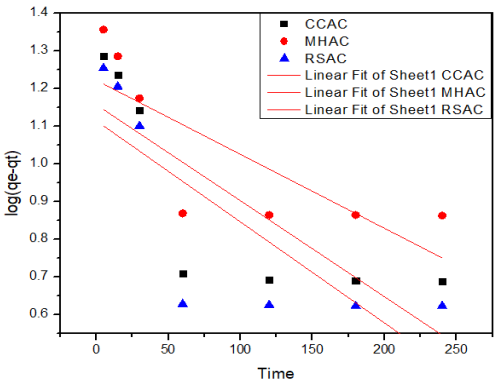

Figure 3.9 plot of Linearized Pseudo First Kinetic Order of glyphosate onto CCAC, MHAC and RSAC

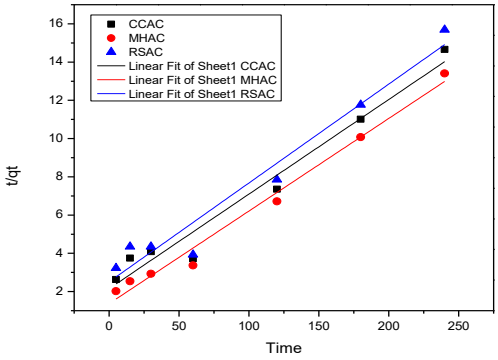

Figure 3.10 plot of Linearized Pseudo Fist Kinetic Order of glyphosate onto CCAC, MHAC and RSAC

4.0 Conclusion

This study demonstrated that activated carbons derived from corn cob (CCAC), millet husk (MHAC), and rice straw (RSAC) are highly effective and sustainable adsorbents for the removal of glyphosate from aqueous environments. Characterization results revealed that the presence of functional groups such as hydroxyl, carbonyl and carboxyl which plays vital roles in glyphosate intake. The adsorption efficiency was strongly influenced by contact time, pH, temperature, and adsorbent dosage, with optimal performance observed under mild acidic conditions. Equilibrium modeling showed that both Langmuir and Freundlich isotherms effectively described the adsorption

behavior, indicating the coexistence of monolayer and heterogeneous multilayer adsorption mechanisms. The pseudo-second-order kinetic model provided the best fit to the experimental data, suggesting that chemisorption governed the rate-controlling step of glyphosate uptake. Among the tested materials, CCAC exhibited the highest adsorption capacity (35.56 mg/g) and removal efficiency (94.2%), demonstrating superior performance compared to MHAC and RSAC.

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