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Adsorption-desorption characteristics of atrazine on activated charcoal and kaolin composite matrices and the In vitro performance of their CRFs

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ABSTRACT

The study reported in this paper examined the adsorption-desorption behaviour of atrazine on activated charcoal (Ac)/kaolin (K) composite matrices as a means of determining their suitability in preparing controlled release formulations (CRFs) of the herbicide atrazine. This is to devise a technique for protecting the environment from pesticide contamination. Adsorption experiments were carried out in buffered solutions, followed by desorption in soil to determine rates of atrazine release over time in the different media. The adsorption process was found to be independent of pH within the pH range of 5-8 studied. The adsorption data at pH 5, 6, and 8 (KH_2PO_4) and 24 °C for the different surfaces were fitted to the Langmuir and the Freundlich Isotherms. The regression coefficient, R^2 was used to determine the isotherm model that better fitted atrazine sorption on the composite matrices. The obtained R^2 values in the composite surfaces showed that the Langmuir isotherms were a marginally better fit than the Freundlich isotherms. The calculated favourable adsorption constant, R_L, all being less than unity in the surfaces, indicates that the composite matrices have a high affinity for atrazine.

Keywords: Slow release, Favourable, Herbicides, Environment, isotherm.

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INTRODUCTION

Weed infestation remains a major constraint to global food production and has led to intensive use of synthetic herbicides such as atrazine for the control of annual broadleaf and grass weeds in crops including maize and sorghum (Solomon et al., 1996). Atrazine is moderately soluble and relatively persistent, and both the parent compound and its metabolites are frequently detected in surface and groundwater at agriculturally impacted sites (Gfrerer and Lomniczi, 2003; Brauns et al., 2018; Dou *et al*., 2020; Wang *et al*., 2024). Because atrazine can leach through soil profiles and pose risks to aquatic ecosystems and human health, many regulatory agencies have introduced measures to limit its environmental contamination (U.S. EPA, 2020).

Controlled-release formulations (CRFs) of herbicides have been proposed as an effective approach to maintain weed control while reducing leaching losses and off-target exposure (Fernández-Piñas *et al.,* 2000; Li *et al*., 2024). In these systems, the active ingredient is associated with a carrier matrix, and release into the soil solution is governed by sorption–desorption and diffusion processes, which can prolong herbicidal activity and decrease the required application rate (Li *et al.,* 2024; Wang *et al*., 2025). For atrazine, alginate–bentonite or other clay–polymer CRFs have been shown to slow release into water and soils and to reduce its mobility in column experiments compared with conventional formulations (Cox *et al*., 1998; Cabrera *et al*., 2001; Lobo *et al*., 2003).

Activated carbon and clay minerals are widely used sorbents for organic contaminants because they combine high surface area, tunable surface chemistry, and relatively low cost (Li *et al*., 2019). Activated carbon has strong affinity for atrazine and related triazines, whereas kaolin is abundant, chemically stable, and commonly present in agricultural soils, making charcoal–kaolin composites attractive matrices for pesticide delivery (Abate *et al*., 2017; Araújo *et al*., 2025). Composite formulations that incorporate activated carbon into alginate–clay beads have already demonstrated enhanced control of herbicide release for other active ingredients, suggesting that similar systems could be designed to manage atrazine availability while limiting its downward transport (Fernández-Piñas *et al*., 2000; Cotterill and Wilkins, 1992). However, the specific relationship between adsorption–desorption characteristics of atrazine on activated charcoal–kaolin matrices and its subsequent release profile from CR beads has not been systematically investigated.

Therefore, the present work aims to develop controlled-release formulations of atrazine using activated charcoal–kaolin composite matrices and to relate their adsorption–desorption behaviour to in-vitro release performance. Building on previous research on clay–polymer CRFs of atrazine (Cabrera *et al*., 2001; Lobo *et al*., 2003), this study (i) characterizes atrazine adsorption and desorption on activated charcoal–kaolin composites, (ii) prepares atrazine-loaded CR beads based on these matrices, and (iii) evaluates their release kinetics in aqueous media simulating soil pore water. By establishing a mechanistic link between sorption properties and release behaviour, the work seeks to provide a basis for designing atrazine formulations that maintain effective weed control while reducing groundwater contamination and environmental pollution (Li *et al*., 2024; Wang *et al*., 2024).

MATERIALS AND METHODS

Materials

The following materials were used as obtained: Atrazine, anhydrous citric acid, ethylamine, chloroform, sodium alginate, potassium dihydrogen phosphate (KH_2PO_4) were obtained as analytical grade from Zayo Sigma, Jos, Nigeria, and were used as received, while calcium chloride, ethanol, distilled water, and activated charcoal were sourced from NAFDAC Zonal Laboratory, Agulu.

Methods

Atrazine stock solution

Atrazine (70.00mg) was dissolved in 20 ml ethanol in a reagent bottle in order to aid solubility of the herbicide in water. Using $1000\mu L$ Eppendorf pipettes, the following volumes of the 3.50 mg/ml stock solution were measured into a 25ml volumetric flask in each case, and the volume made up to the mark with ethanol.

Vol. of stock solution (ml)	5.71	4.29	3.57	2.86	1.43
Conc.of resulting atrazine solution (mM)	0.80	0.60	0.50	0.40	0.20

The absorbance of these solutions was measured using Cary 50 UV-visible spectrophometer by Varian, and the calibration curve plotted.

KH_2PO_4 buffer

A litre of 0.1M KH_2PO_4buffer pH 4.38 was made by weighing 6.80g (0.1mol) KH_2PO_4 into 500ml water in a 1-litre volumetric flask. This was agitated with a vortex mixer to make it dissolve. Aliquots of 18ml, 22ml and 26.5ml of 1M NaOH were added into the volumetic flask to adjust the pH to 5.0, 6.0 and 8.0, respectively. A pH meter was used to confirm the pH.

Atrazine derivatization with ethylamine

Into 2ml of 0.8mg/L atrazine solution in plastic screw capped bottles was added 1ml of 0.2M ethylamine. The bottles were immersed in boiling waterbath for 10 mins. The bath temperature was maintained between 50-100°C. The bottles were then cooled under running water and the required volume measured out for each atrazine concentration. In this derivatization procedure, ethylamine reacts with atrazine to yield 2, 4-(ethylamino)-6-isopropylamine-1, 3, 5-triazine (EAIA) which is amenable to quantitative uv-visible determination at 550nm, the wavelength of maximum absorbance. The obtained absorbances at 550nm were subjected to Beer-Lambert`s law given by the expression

$$A = \mathcal{E}bc \qquad (1)$$

where A is the absorbance (mg/g), b is path length (cm), $\mathcal{E}$ is molar absorptivity (M^{-1} cm^{-1}) and c is the concentration (mg/L). Equation (…1) was applied in calculating the equilibrium concentrations, C_e, reported in this work.

Adsorption isotherm

Adsorption-desorption experiments were done using the batch equilibration method as employed by Cruz-Guzmn *et al*., (2005) and described as follows: 70mg of Ac:30mg of K, 50mg of Ac:500mg of K and 30mg of Ac:70mg of K were separately weighed into five different 25ml conical flasks, and 2ml of the prepared buffer solution (pH 5.0, 6.0 or 8.0) was added to each sample in the conical flask, followed by 0.3ml atrazine solution of the different concentrations (0.2, 0.4, 0.5, 0.6, 0.8mg/L). These were placed on a mechanical shaker for equilibration for one hour at room temperature. After equilibration, the suspensions were centrifuged at 4000rpm for 10 minutes. Four ml of the supernatant was withdrawn for atrazine assay. Atrazine solution in buffer without adsorbent was agitated for 1 hour to serve as control. In all cases, the difference between initial and final concentration was assumed to be due to adsorption.

Desorption isotherm

Desorption was studied only at pH 5. Measurement of desorption rates started immediately after adsorption, the highest initial concentration point of the adsorption isotherms being regarded as the starting point of desorption, using the decanting method (Cruz-Guzmn *et al*., 2005). This method involves the separation of a mixture containing a liquid and solid into two distinct layers on standing. The ratio of solid/liquid phase was kept the same as in the adsorption experiments, but the 3ml supernatant withdrawn was replaced with 3ml of pH 5 buffer after shaking for 1hr, to make up to the required volume. The suspension was centrifuged, and 3ml supernatant withdrawn for determination of active ingredient concentration. This was repeated four times.

Preparation of controlled release formulations

Preparation of the controlled release formulations exploited the gelling properties of sodium alginate. The preparation of all controlled release formulations was carried out in a conical flask. 5.6g of the matrix (50mg of Ac:50mg of K) was weighed into a 250cm^3 conical flask, 10 ml of water added, and vigorously stirred by a magnetic stirrer for 5 minutes. A solution of the active ingredient (a.i), (i.e atrazine) was added, followed by 3g sodium alginate, with the stirring speed increased to double its initial value for 1 hour. The slurry formed was withdrawn with a 50ml syringe and deposited into about 200cm^3 2.5M calcium chloride solution, which was placed in a clean, dry petri dish. The beads formed were left in the calcium chloride solution in the dish for 30 minutes before filteration with filter paper. The beads were collected and placed on an aluminium foil at room temperature to dry. No beads were formed on the addition of $CaCl_2$ solution to the slurry formed with ethanol as solvent. Generally, percent composition of the slurry was about 10-15% matrix, 1-2% active ingredient (a.i), 1% algin, 2-3% additive, and water to make up 100%.

Determination of sorbed atrazine concentration

Atrazine itself is not amenable to convenient UV-Visible determination, as its wavelength of maximum absorption occurs at 225 nm. Rather, its concentration in the supernatant was determined spectrophotometrically after derivatization with ethylamine absorbance of the derivatized product was measured at 550 nm. In all cases, the difference between initial and final atrazine concentration was ascribed to sorption.

Batch release experiments

(a) Herbicide release in water

Weighed quantities of dry CR beads (two replicates of each sample) were added to 50 ml 1:1 buffer EtOH mixture at pH 5 in stoppered 150 ml borax bottles. The system was manually agitated end-over-end thrice and left to stand. At specific time intervals, the bottles were shaken in the same way, allowed to settle for 2 minutes, and 5 ml aliquots withdrawn, filtered, and analyzed for herbicide. Each aliquot withdrawn was replaced by 5ml of the buffer: EtOH mixture to maintain sink conditions.

(b) Desorption in soil using the soil column test.

Air-dried soil was ground to pass through a 2.0 mm sieve and then packed into four different PVC pipes with diameters of 10 cm which were labeled A (commercial herbicide formulation), Ac-50: K-50 to a depth of 10 cm. The column was saturated with tap water to its capacity and allowed to equilibrate for 24hrs. A commercial formulation of atrazine was dissolved in ethanol and applied to a control column (A). Three grams of the CR formulations was mixed uniformly with the soil in the other three columns. The set-up was saturated with tap water and covered with a thin layer of soil, which itself was covered with cellophane to prevent herbicide volatilization. It was then allowed to stand for two weeks within which period saturation with water was carried out two more times. At the end of the two weeks, each of the packed soil columns was extruded and cut into four sections of 2 cm each. Each section was placed in a beaker, and the herbicide extracted with ethanol. The extract was analyzed spectrophotometrically for atrazine.

UV-Visible Spectrophotometry

All UV-Visible measurements were undertaken using a Varian Cary 50 Spectrophotometer on cuvettes at a temperature of 25±0.1°C. The scanning was run from 600 to 200 nm at the rate of 1.00 nm/min, within which wavelenghth of highest intensity was obtained.

Data analysis

The calibration of the absorption of the derivatization product from atrazine, 2,4-(ethylamino)-6-isopropylamino-1,3,5-triazine, and the data gathered spectrophotometrically for adsorption, desorption, and release studies were all subjected to linear regression analysis to yield the parameters of interest and the accompanying correlation coefficients. The regression equations utilized were in conformity with the equation of a straight line

$$y = mx + c \tag{2}$$

where y (absorbance) is the dependent variable, which was plotted against changing x (the concentration of pesticide), which is the independent variable in the equation. The coefficient of x is m, which is the slope, while c is the intercept on the y-axis.

RESULT/EXPERIMENTAL

Table 1. Extinction coefficients and R^2 values obtained from the calibration curves at the various pHs.

Parameters	pH 5	pH 6	pH 8
Extinction coefficients, Ɛ,($M^{-1}cm^{-1}$)	9.66×10^4	8.34×10^4	9.79×10^4
R^2	0.974	0.970	0.984

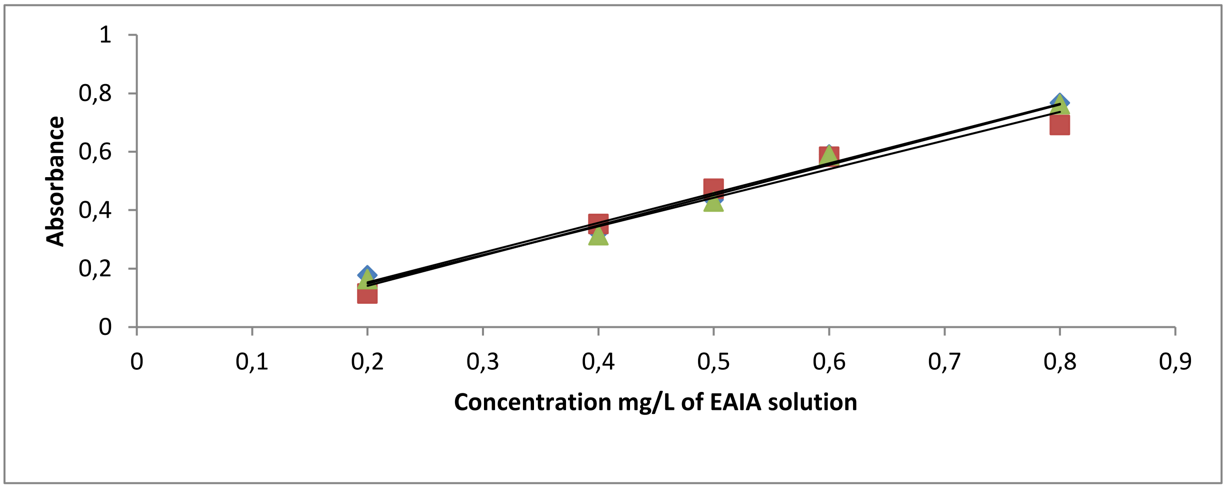

Figure1. Calibration curves of atrazine adsorption at different pHs.

Table 2. Freundlich constants for atrazine adsorptions on K-70:Ac-30, K-50:Ac-50, K-30:Ac-70 surfaces at pH 5, 6 and 8.

	K_F(mg g^{-1})	n	R^2	$\triangle G^{F}_{ads}$(Jmol^{-1})
pH 5				
K-70: Ac-30	1.138	1.883	(0.894)*	-319.207
K-50: Ac-50	1.172	1.425	0.980	-391.900
K-30: Ac-70	1.153	1.497	0.994	-351.541
pH 6				
K-70: Ac-30	1.081	3.729	(0.695)*	-192.322
K-50: Ac-50	1.245	1.433	0.980	-541.102
K-30: Ac-70	1.208	1.495	0.978	-466.606
pH 8				
K-70: Ac-30	1.084	2.481	0.999	-199.165
K-50: Ac-50	1.172	1.429	0.977	-391.900
K-30: Ac-70	1.169	1.172	0.978	-385.571

Because of the poor R^2 value obtained for the surface K-70: Ac-30 at pH 5 and 6, these data are not included in the analysis and subsequent discussion.

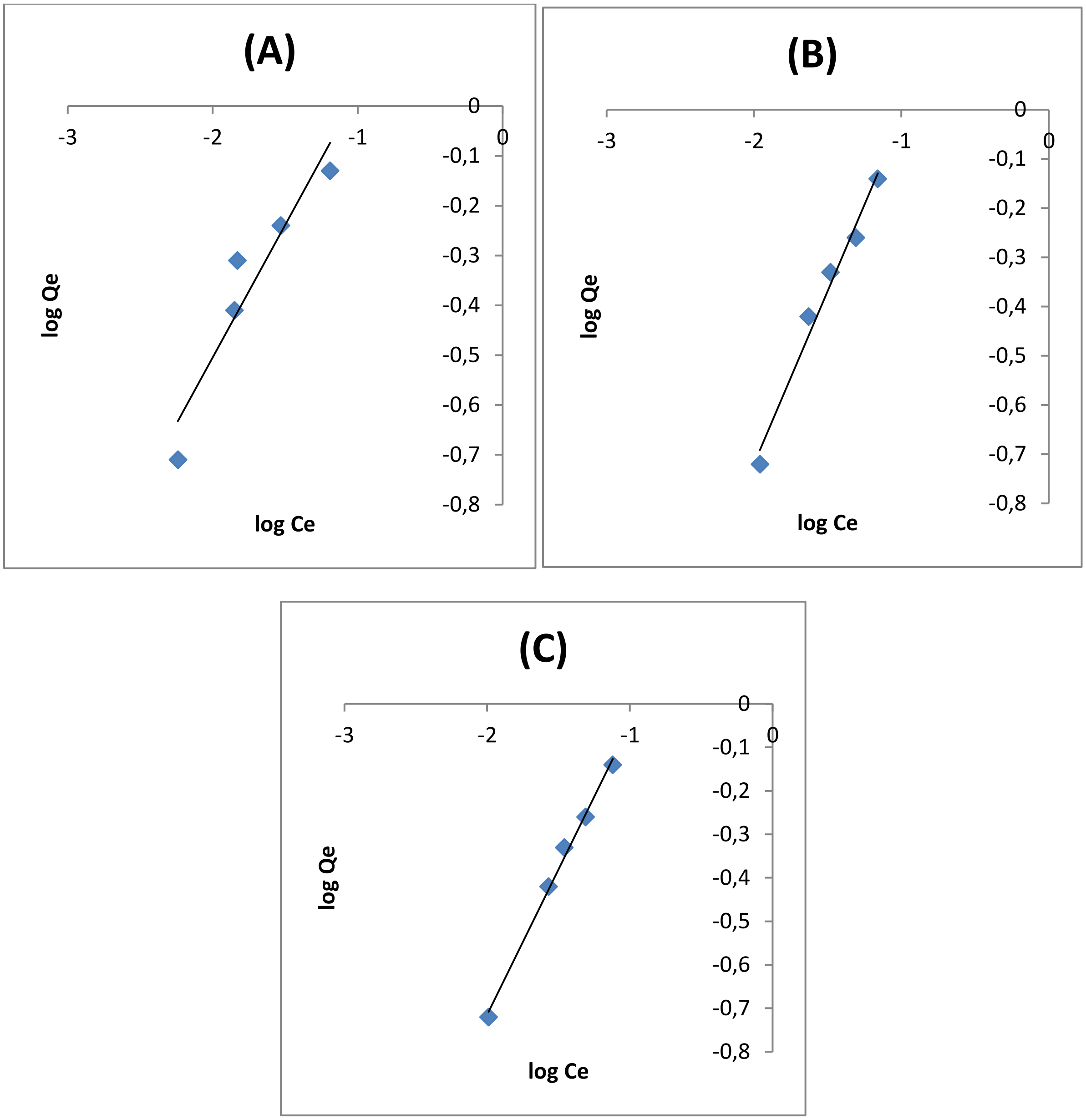

Figure 2. Freundlich plots for atrazine adsorption at pH 5 on (A) K-70:Ac-30 (B) K-50:Ac-50 (C) K-30:Ac-70 .

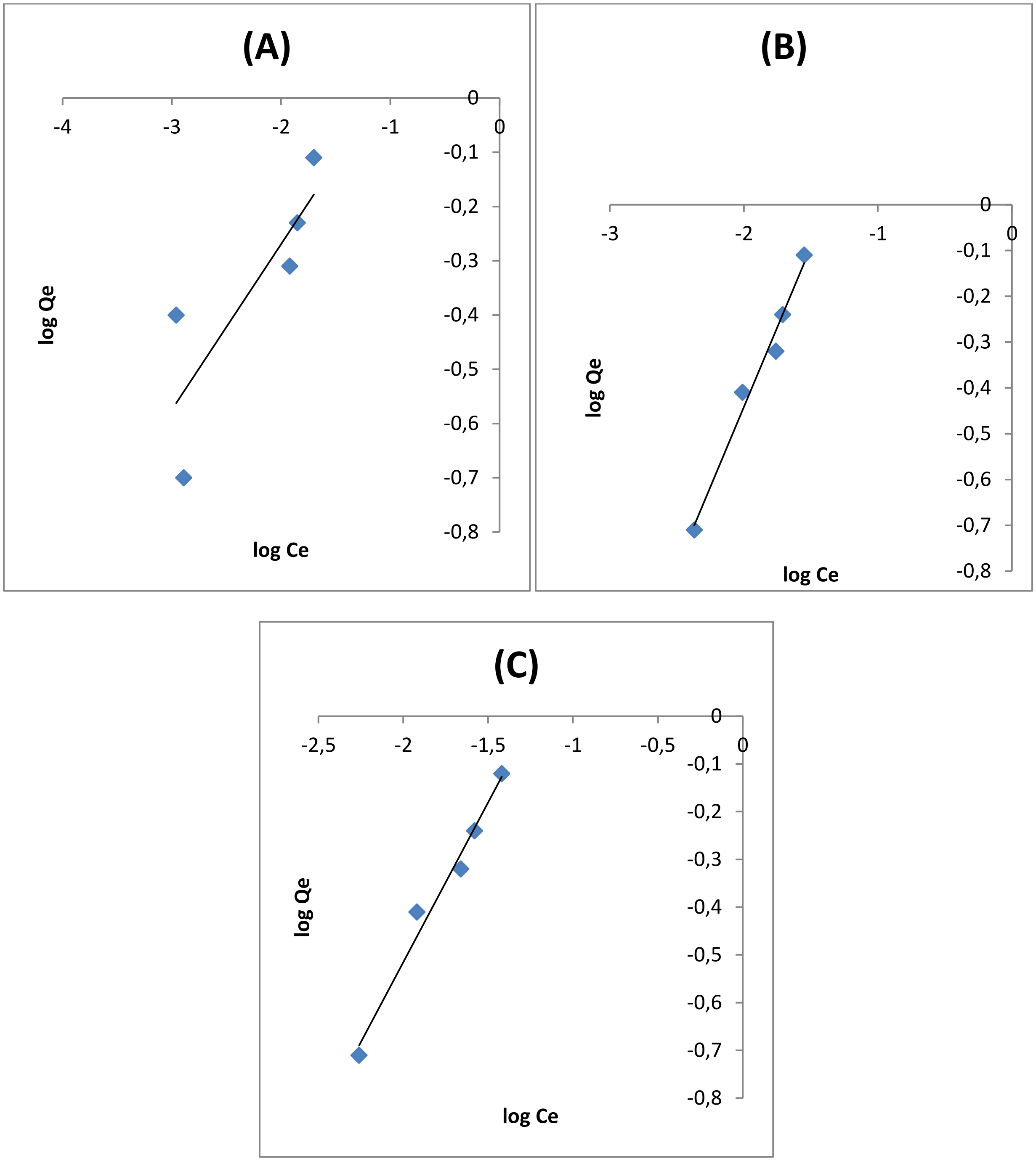

Figure 3. Freundlich plots for atrazine adsorption at pH 6 on (A) K-70:Ac-30 (B) K-50:Ac-50 (C) K-30:Ac-70.

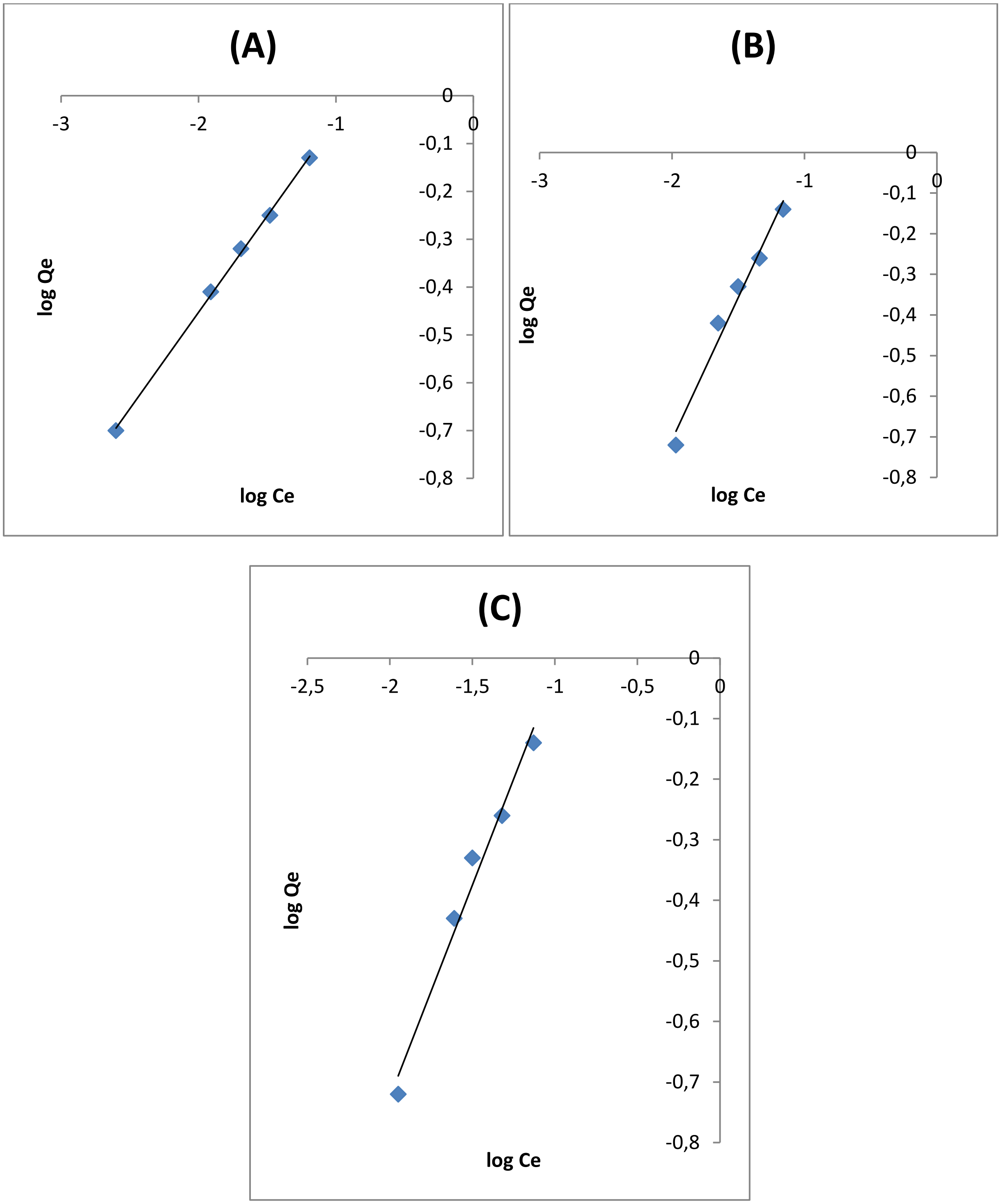

Figure 4. Freundlich plots for atrazine adsorption at pH 8 on (A) K-70: Ac-30 (B) K-50: Ac-50 (C) K-30: Ac70.

Table 3. Langmuir constants for atrazine adsorptions on the composite activated charcoal (Ac), kaolin (K) surfaces at pH 5, 6 and 8.

	K_L	b (Lg^{-1})	R^2	R_L	ΔG^L_{ads}(Jmol^{-1})
pH 5					
K-70: Ac-30	**1.195**	**0.029**	**0.978**	**0.983**	**-439.889**
K-50: Ac-50	**1.550**	**0.078**	**0.995**	**0.955**	**-1082.165**
K-30: Ac-70	**1.171**	**0.053**	**0.996**	**0.969**	**-389.792**
pH 6					
K-70: Ac-30	**(0.627)***	**(0.001)***	**(0.796)***	**(0.999)***	**(-1152.671)***
K-50: Ac-50	**1.323**	**0.024**	**0.991**	**0.986**	**-691.150**
K-30: Ac-70	**1.245**	**0.029**	**0.989**	**0.983**	**-541.102**
pH 8					
K-70: Ac-30	**0.653**	**0.006**	**0.975**	**0.996**	**-1052.343**
K-50: Ac-50	**1.580**	**0.076**	**0.994**	**0.956**	**-1129.500**
K-30: Ac-70	**1.534**	**0.078**	**0.996**	**0.955**	**-1056.543**

*Because of the poor R^2 value obtained for the surface K-70: Ac-30 at pH 6, the data is not included in the analysis and subsequent discussion.

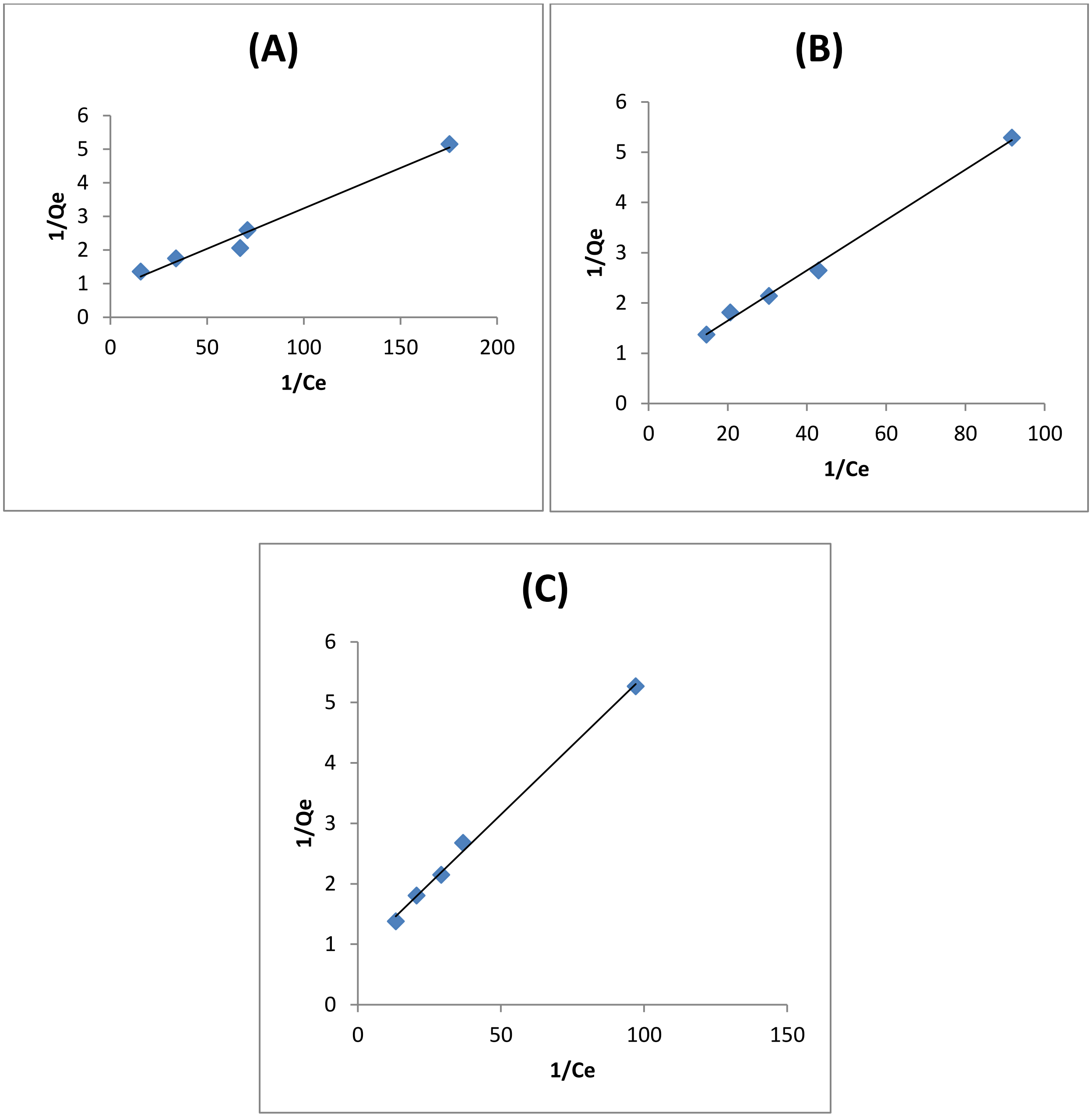

Figure 5. Langmuir plots for atrazine adsorption at pH 5 on (A) K-70: Ac-30 (B) K-50: Ac-50 (C) K-30: Ac-70.

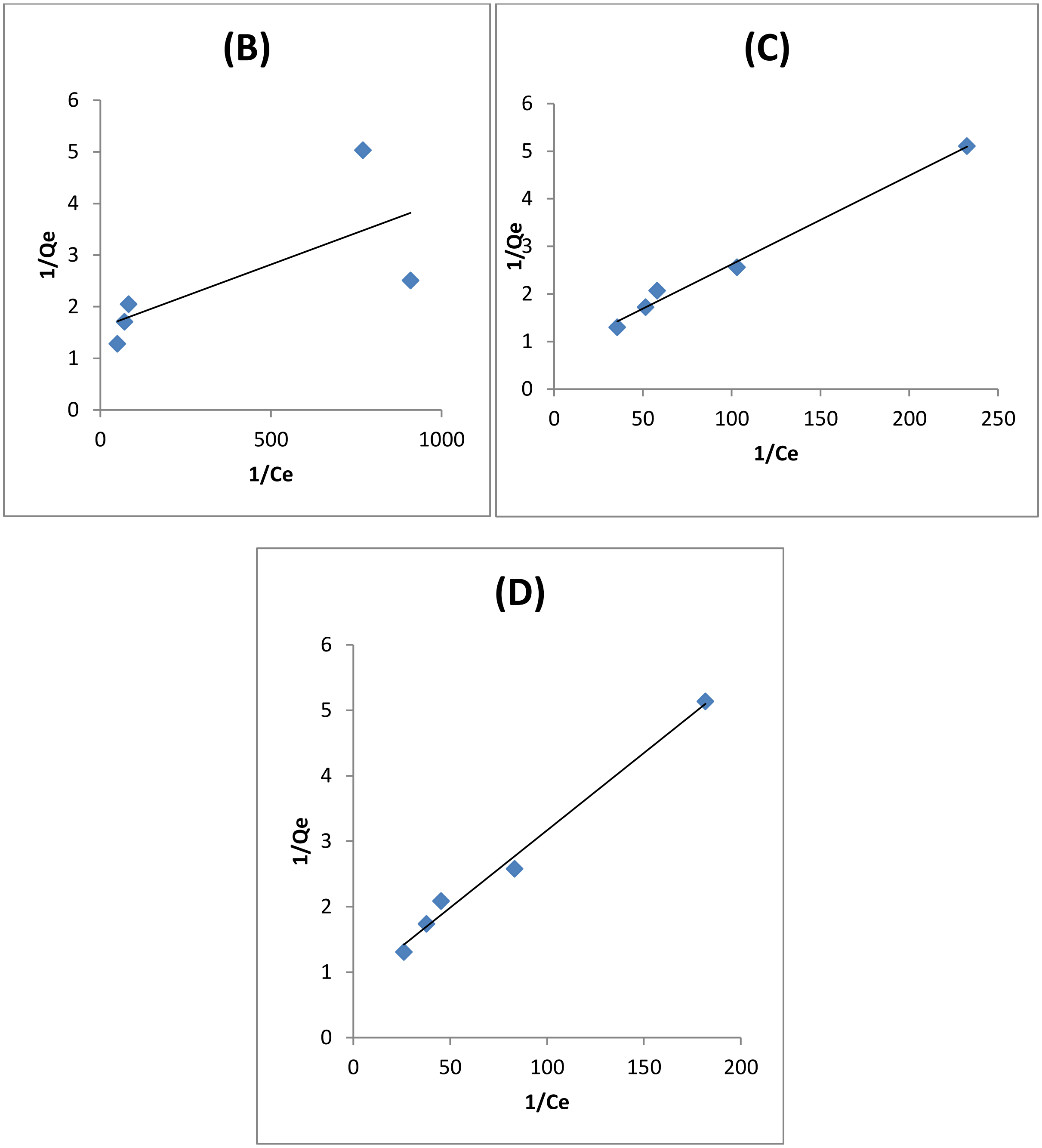

Figure 6. Langmuir plots for atrazine adsorption at pH 6 on (A) K-70: Ac-30 (B) K-50: Ac-50 (C) K-30: Ac-70.

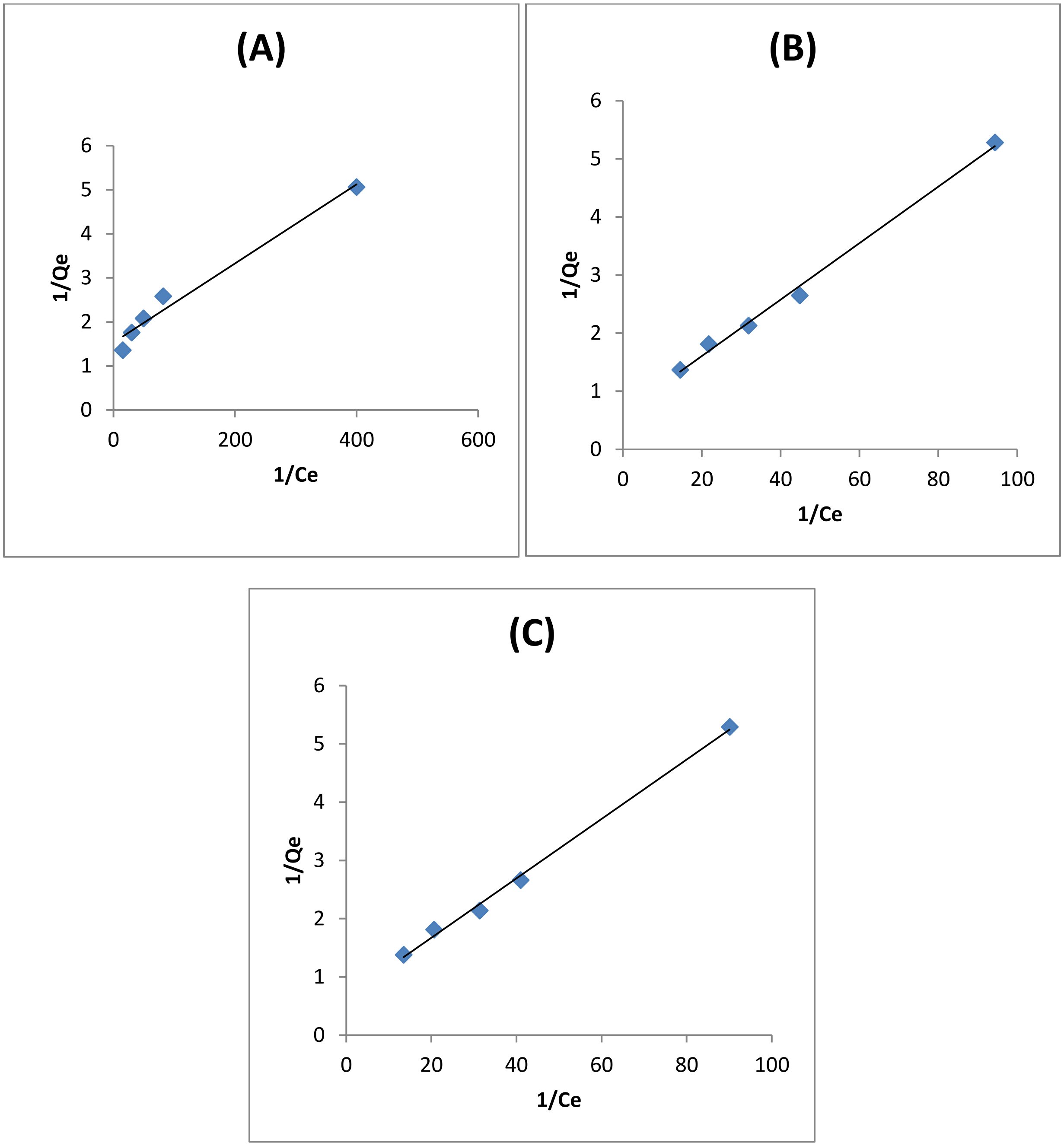

Figure 7. Langmuir plots for atrazine adsorption at pH 8 on (A) K100 (B) KC7030 (C) KC5050 (D) KC3070 (E) C100

Table 4. Data for the desorption of atrazine in soil.

	Herbicide Concentrations (mg/L)	
	A (commercial sample)	B (K-50: Ac-50)
Top (2cm)	0.062	0.321
Middle (4cm)	0.286	0.085
Middle (6cm)	0.233	0.018
Bottom (8cm)	0.192	0.000

K-30: Ac-70 and K-70: Ac-30 was not included in desorption in soil experiment as K-50: Ac-50 was used to monitor the migration rate of the composites.

DISCUSSION

Calibration of the analytical method was essential to reliably link spectrophotometric measurements to atrazine concentrations in the controlled-release formulations. Calibration curves for the atrazine derivative EAIA at different pH values were obtained by plotting absorbance at 550 nm, the wavelength of maximum absorption, against known EAIA concentrations, following the approach described by Onyido (2018). The linearity of these plots, together with similar extinction coefficients and high correlation coefficients at all pH values (Figure 1, Table 1), shows that EAIA solutions obey the Beer–Lambert law under the experimental conditions. Within experimental error, the extinction coefficient was independent of pH, so the values determined at each pH were used confidently to convert absorbance readings to atrazine concentrations in subsequent adsorption and release studies.

The adsorption data for atrazine on the activated charcoal–kaolin composites at pH 5, 6 and 8 were interpreted using the Freundlich and Langmuir isotherm models, which are widely applied to describe pesticide sorption on heterogeneous and approximately homogeneous surfaces, respectively (Xing, 2001; Foo and Hameed, 2010). For most composite formulations, both models provided acceptable fits, indicating that the surfaces possess a distribution of sorption sites but also approach monolayer coverage at higher concentrations. However, the Freundlich correlations for the K-70:Ac-30 composite at pH 5 and 6 were weak, suggesting that this surface does not fully conform to the heterogeneous sorption pattern assumed in the Freundlich model.

In contrast, the Langmuir plots were generally well behaved, with high correlation coefficients for all composites except K-70:Ac-30 at pH 6, indicating that a monolayer-type adsorption process predominates on most of the activated charcoal–kaolin matrices (Nnamonu *et al*., 2011). The calculated monolayer capacities and affinity constants were in the range reported for atrazine adsorption on other activated carbons and carbonaceous sorbents, where Langmuir behaviour is often observed (Gupta *et al*., 2011; Faur-Brasquet *et al*., 2005; Ahmed *et al*., 2015).

The relatively low affinity constants and the very small free energies of adsorption obtained in this study are characteristic of physisorption dominated by weak, non-covalent interactions, consistent with values typically reported for physical adsorption processes (Meroufel *et al*., 2013; Das *et al*., 2020). Similar coexistence of acceptable Freundlich and Langmuir fits has been reported for atrazine adsorption on waste activated carbon, highlighting that real sorbents can simultaneously exhibit surface heterogeneity and near-monolayer coverage (Goli *et al*., 2004).

The dimensionless separation factor derived from the Langmuir parameters yielded values close to, but below, unity for all composites and pH values, corresponding to marginally favourable adsorption according to the criterion proposed by Foo and Hameed (2010). This behaviour aligns with the intended function of the composites in controlled-release systems: atrazine should be retained strongly enough to avoid rapid leaching, but not so strongly that it cannot desorb and diffuse into the soil solution to provide effective weed control.

The soil-column experiments link the adsorption characteristics of the composites to the environmental performance of the controlled-release beads. After two weeks of simulated rainfall, the commercial atrazine formulation was detected at depths greater than 8 cm, indicating relatively fast movement through the soil profile and a heightened risk of leaching toward groundwater. In contrast, the K-50:Ac-50 composite formulation retained a larger fraction of atrazine in the upper soil layers, with concentration maxima closer to the surface horizon where weed seeds and seedlings are located. The observed order of surface retention, K-50:Ac-50 > commercial formulation, demonstrates that encapsulation in the activated charcoal–kaolin matrix slows downward migration and keeps atrazine concentrated near the soil surface.

These findings are consistent with previous work on alginate–bentonite controlled-release formulations of atrazine, where commercial products showed considerable mobility and were detected at depths approaching 20 cm, while CR granules released atrazine more slowly and mainly in the upper soil layers (Cabrera *et al*., 2001; Lobo *et al*., 2003). By analogy with these systems, the present results suggest that the activated charcoal–kaolin beads reduce the proportion of atrazine subject to rapid leaching and thus lower the potential for groundwater contamination, in line with the environmental goals highlighted in the introduction. At the same time, the marginally favourable and predominantly physical adsorption observed in the isotherm study indicates that atrazine can still desorb from the composite surfaces, supporting sustained release and maintaining herbicidal efficacy over time. Overall, the combination of spectrophotometric quantification, isotherm analysis, and soil-column testing demonstrates that activated charcoal–kaolin matrices provide a practical route to controlled-release atrazine formulations that better balance weed control with environmental protection.

CONCLUSION

The composite matrices K-70:Ac-30, K-50:Ac-50 and K-30:Ac-70 showed favourable, mainly Langmuir-type adsorption of atrazine, confirming that activated charcoal–kaolin surfaces are effective sorbent matrices for this herbicide. In soil columns, encapsulated atrazine in these composites remained concentrated near the surface, whereas the commercial formulation migrated deeper, indicating a higher leaching and groundwater-contamination risk for the conventional product. Overall, activated charcoal–kaolin composites with sodium alginate appear suitable for controlled-release atrazine formulations that combine good weed control with reduced environmental impact.

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