Efficient Removal of Acetamiprid Pesticide Using Date Core-Derived Activated Carbon and Korai Clay As A Good Sorbent

Huda Azhar Fadel [1*], Lekaa Hussein Khdaim [1]

Abstract

We used activated carbon derived from Iraqi date cores to study the efficacious removal of acetamiprid pesticide effluent. This activated carbon was made using chemicals and physical processes involving a 5M hosphoric acid solution. Various factors such as the concentration of acetamiprid pesticide, mixing duration, contact time, the quantity of activated carbon, temperature, and pH of the media were examined to understand how they influence the efficiency of acetamiprid pesticide removal. The results show that maximum adsorption of 0.05 g/L dosage, 150 mg/L dye concentration, and 3-hour contact time adsorption duration were optimal for 97.00- 99.00% removal efficiency. The adsorption equilibrium was performed using Freundlich, Temkin, Harkins-jura, Dupin, Elovitch, and Langmuir. Density function theory (DFT) was used to simulate the pesticide acetamiprid to determine the reaction origin. The results show that the Freundlich model with R2 values equal to 0.738 demonstrated better agreement with the adsorption experimental data. According to thermodynamic analysis, the pesticide is endothermic and spontaneous, indicating increased unpredictability at the adsorbent-adsorbent interface. It also indicates the pHZpc of the negatively charged carbon surface at pH = 2.0. Two models of pseudo-first and pseudo-second order were included in the kinetic analysis. The outcomes indicate that a pseudo-second-order model with R2 > 0.9998 fits the experimental data better.

Keywords: Acetamiprid, Adsorption, Kinetics, Pesticide, Pollution, Isotherms and DFT.

Significance | Iraqi date core-derived activated carbon effectively removes acetamiprid pesticide, crucial for combating water pollution, supported by comprehensive adsorption studies.

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1. Introduction

Water pollution is a serious problem that occurs when water's nature, quality, and properties are altered, making it unfit for human use (Anju, Ravi S, and Bechan 2010). This pollution can happen for different reasons, such as adding chemicals, bacteria, or even thermal or radiological sources. By 2025, half of the world's population is predicted to live in areas where water is scarce (Boretti and Rosa 2019). Water pollution can come from natural sources and human activities (Hong et al. 2020). Nutrient pollution, caused mainly by phosphates and nitrates from fertilizers used in farms and factories, is a big reason for contaminating freshwater.

Moreover, pollution is also caused by waste materials and industrial by-products. Pesticides, harmful chemicals used in agriculture to kill pests, can contaminate waterways and pose health risks to humans and wildlife (Ahmad et al. 2010, Sarhan et al. 2024, Adil et al. 2023). Overuse and incorrect application of fertilizers also contribute to water pollution. When these fertilizers break down, they can seep into the groundwater or get washed into rivers and streams by rainwater (Akhtar et al. 2021).

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One of the most commonly used pesticides is acetamiprid, used globally for pest control (Renaud et al. 2018). Although it's not highly toxic to humans or bees, it can still harm other animals if it gets into the water (Wijaya et al. 2014). Different methods like oxidation, photocatalysis, and adsorption remove pesticides from water. Bioabsorption, a type of adsorption, is particularly favored for its effectiveness and low cost (Hassaan, Eldeeb, and El Nemr 2022). However, the efficiency of these methods depends on various factors like the chemical composition of the contaminants and the conditions of the water (Bashir et al. 2019).

Innovative approaches are being explored to tackle pesticide contamination, such as utilizing phosphoric acid derived from tangerine peels to produce porous activated carbon for acetamiprid removal (TPAC) (Patel, Gupta, and Mondal 2023). Through meticulous analysis of particle size, surface area, and structural properties, this method demonstrates promising efficacy in adsorbing pesticides from water bodies. Moreover, Freundlich and Langmuir isotherms have been employed to evaluate the adsorption process, revealing exhaustive and accidental adsorption mechanisms with a theoretical capacity of 35.7 mg/g and an equilibrium time of 240 minutes. TPAC emerges as an eco-friendly and cost-effective solution for pesticide removal, offering the potential for recovery and reuse over multiple cycles (Mohammad et al. 2020).

Despite the significance of date palms, research exploring the conversion of date kernel residues into activated carbon remains scarce (Al-Fahdawi, 2019). Similarly, koura clay, commonly utilized in brick-making, presents variability in mineral composition based on geographical origin, influencing the properties of resulting bricks (Dill 2016; Konbr, Freewan, and Alshuk 2016). Understanding these variations is crucial for optimizing brick production processes.

The primary focus of recent studies is to develop strategies for water purification, with a particular emphasis on utilizing porous activated carbon derived from agricultural residues and clay for acetamiprid removal. By chemically activating Iraqi date kernel residues and kora clay using NaOH and HNO3, researchers have demonstrated the efficacy of these biosorbents in removing acetamiprid pesticides from aqueous environments (Al-Fahdawi, 2019). Impulse adsorption experiments have been conducted to evaluate removal capacity by investigating factors such as contact duration, mass, pH, and initial pesticide concentration. Results from thermodynamic, kinetic, and isothermal adsorption analyses provide insights into the efficiency of the process. However, addressing water pollution requires concerted efforts encompassing innovative technologies, sustainable practices, and scientific research. By leveraging natural resources such as agricultural residues and clay, coupled with advanced methodologies like adsorption, promising solutions for pesticide removal are being developed. However, further research and collaborative endeavors are imperative to effectively combat water pollution and safeguard global water resources for future generations.

2. Materials and Methods

2.1 Chemicals

The materials used in this research, including acetamprid (94%), hydrogen nitrate (16%), sodium hydroxide (98%), phosphoric acid (35%), and NaNO3 (98%), were all of excellent analytical quality (Scheme 1).

2.2 Prepare the adsorbent surfaces

2.2.1 Iraqi Date Cores (IDC) surface

Iraqi Date Cores (IDC) are collected from the Iraq city dried (IDC) in the open air for 48 hours at 80 °C in an oven after being water-rinsed; the material is subsequently crushed to create a fine, nutty powder. The (IDC) powder is physically activated with carbonation in an oven at 500 °C for two hours, and chemically activated with the addition of phosphoric acid at a five million concentration (1:1) for twenty-four hours. The powder is then dried, washed with hot water, and sifted to size through 600 µm(Konbr, Freewan, and Alshuk 2016). As shown in Scheme 2, Table 1.

2.2.2 Koura Clay surface

Koura Clay was ground and washed with distilled water several times to get rid of dust, salts and suspended impurities, and Next, as indicated in the following figure Koura Clay, the particles were filtrated, dried out, and the resulting clay was collected, ground once more, and filtered through a permeable sieve (200 µm). The results of chemical examination of Iraqi date cores turned into Koura clay and carbonated charcoal are listed in Table1.

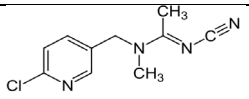
2.3 Evaluation of the efficiency of acetamprid removal in aqueous media

The batch method was used to evaluate the effectiveness of an activated carbon derivative in removing acetamprid from Iraqi date kernels. Several important experimental factors have been studied, including the pH of the solution under study (2-10), the amount of the drug labeled (10-150 mg/L) at a concentration of 500 mg/L, and the amount of activated carbon. (0.05 – 0.5 g) made from Iraqi date kernels, and the reaction time is (0.5 – 3.5 hours). 50 ml of 150 mg per liter of a drug called solutions and 0.05 grams of activated carbon were collected in a conical flask to test the effect of pH, temperature, duration of contact, and dose of activated charcoal on acetamiprid. The mixture was centrifuged after continuous stirring at a rate of 200 rpm. Next, the amount of acetamiprid still present in the solution was measured using UV visible spectrometry at 629 nm, which is its maximum absorption. The adsorption capacity of acetamiprid (qe, mg/g) was calculated using (Simonin 2016).

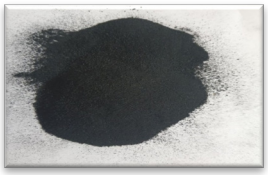
$$Qe=((Co-Ce)V)/m \quad \ldots\ldots\ldots\ldots\ldots (1)$$

where m (mg) is the amount of activated carbon, V (L) is the solution's volume of the acetamiprid, and C0 (mg/L) and Ce (mg/L)

Scheme 1. Chemical Properties of Acetamiprid

Molecular Weight	Chemical formula	IUPAC Name	Structure
222.67 g/mol	C10H11ClN4	N-[(6-chloropyridin-3-yl)methyl]-N'-cyano-N-methylethanimidamide	

Iraqi Date Cores



carbonated charcoal

Scheme 2. Iraqi Date Cores converting into carbonated charcoal

Table 1: The Chemical analysis of Iraqi Date Cores converting into carbonated charcoal and Koura Clay.

Koura Clay		activated carbon derivate from Iraqi Date Cores	
Constituent	**(Wt%)**	**Constituent**	**(Wt%)**
SiO_2	39.71	C	45.74
CaO	18.32	N	4.74
Al_2O_3	9.624	O	41.25
Fe_2O_3	6.905	P	8.26
MgO	5.063		
K_2O	1.343		
TiO_2	0.929		
Na_2O	1.357		

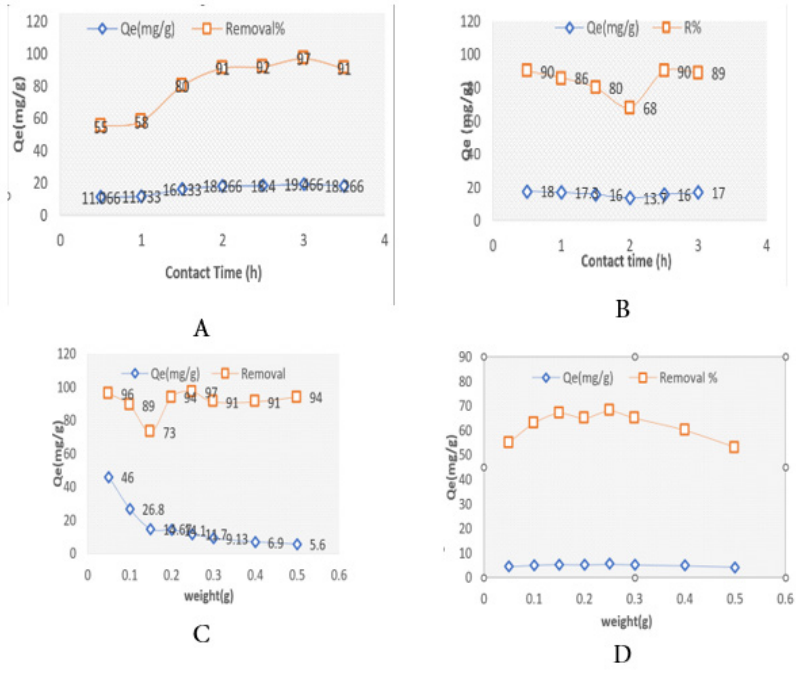


Figure 1. A) Effect of contact duration on the surface of activated charcoal made from Iraqi date kernels at a temperature of 318 K and pH = 7 for absorption of 20 mg/L of acetamprid.
B) Effect of contact duration on surface absorption of alkoura clay by 20 mg/L of acetamprid at 318 K and pH = 7
C) shows the extent to which surface weight affects the amount of pesticide that can be adsorbed at 318 K on carbonated charcoal made from Iraqi date kernels, when coal weights vary. The acid function is set at pH = 7.
D) Effect of surface weight on adsorption of 15 mg/L of acetamprid with different weights at Cora Clay temperature (318 K) and pH = 7.

Table 2. Thermodynamic function values for the pesticide Acetamiprid's adsorption on various surfaces

Values of thermodynamic	Activated charcoal	Koura clay
ΔG KJ.mol^{-1}	-0.1352	-5.4302
ΔH KJ.mol^{-1}	21.616	26.338
ΔS KJ.mol^{-1}	0.0684	0.0999

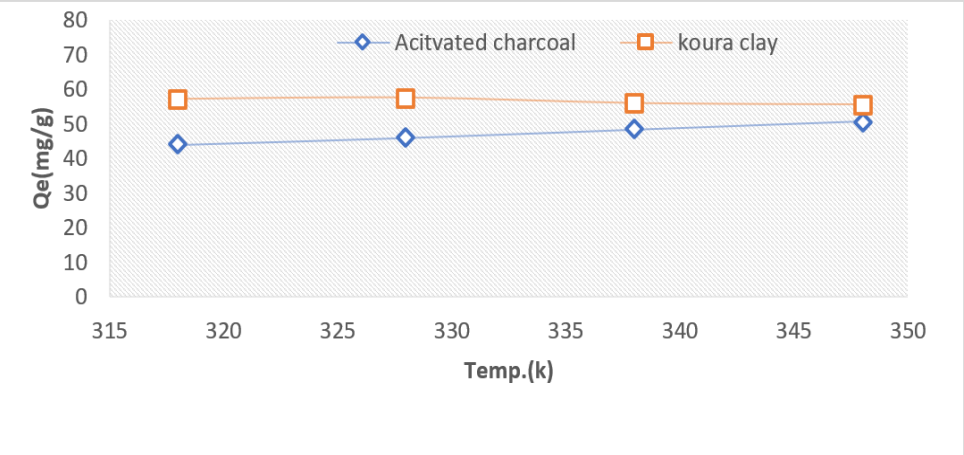


Figure 3. The impact of temperature on the adsorption of 20 mg/L of acetamiprid pesticide at varying weights, at 318 k temperature and pH = 7 acidic function

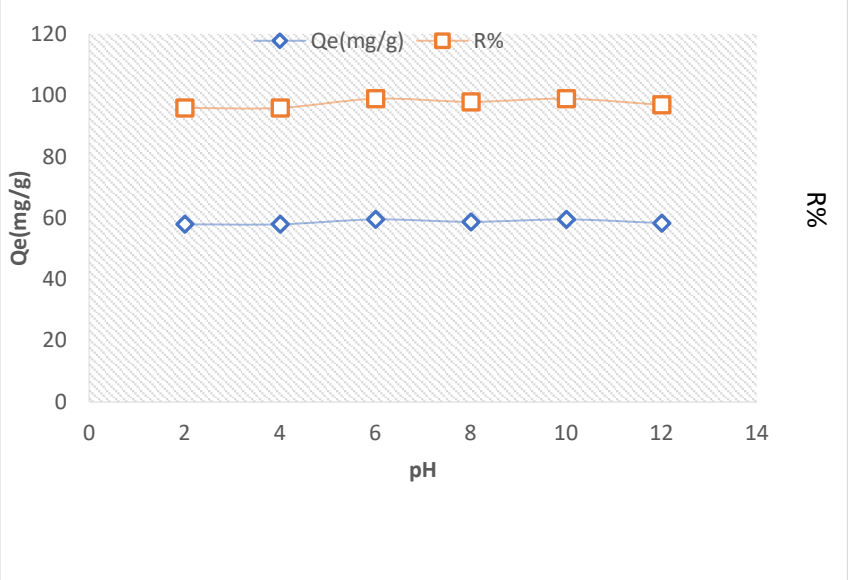


Figure 4. Effect of pH function of adsorption of 15 mg/L of acetamipride pesticide with 0.5 g of charcoal surface derived from Iraqi date kernels at 318 K.

indicate the original as well as equilibrium acetamiprid concentrations, respectively.

Statistical analysis

Data analysis was performed using IBM SPSS-29 (IBM Statistical Packages for Social Sciences, version 29, Chicago, IL, USA). The significance of differences between means for quantitative data was assessed using Student's t-test. Statistical significance was considered when the p-value was equal to or less than 0.05.

3. Results

3.1 The effect of contact time

This experiment was carried out to determine when the embedded acetamiprid will interact most effectively with the outermost layer of carbonated carbon made from Iraqi current cores(Ali 2018). Based on the results, the pesticide has a neutral lifetime of three hours, during which its ability to absorb increases in proportion of contact time until saturation occurs(Bonmatin et al. 2015). The relationship between the time (h) and the quantity of carbonated charcoal (mg/g) obtained from the powdered Iraqi date cores used as an adsorbent is explained in Figure 1A. This relationship is positive, meaning that increasing any one of the variables will increase the other. For initial acetamiprid levels of 150 mg/L, acetamiprid adsorption was measured at a given contact time with a different interaction time at (0.5-3.5 h). This is most likely because more adsorption surfaces were initially available to absorb pollutants. At initial concentrations of 150 mg/L, shaking was required for approximately three hours to achieve most of the maximum pollutant removal. The pollutant adsorption increased with increasing contact time and remained constant for 30 minutes after reaching equilibrium for different starting concentrations(Konbr, Freewan, and Alshuk 2016) (Suresh, Sugumar, and Maiyalagan 2011). The empty holes on the surface cause the initial high rate of absorption. Nevertheless, the rate of adsorption steadily decreases as soon as these gaps are filled by chemical ions, eventually depending on the transfer of electrons from the solution with water to the adsorbent surface(Suresh, Sugumar, and Maiyalagan 2011).

3.2 The effect of Surface Weight on adsorption Acetamiprid

This effect was measured by applying various weights to the charcoal surfaces made from Iraqi date cores, ranging from 0.05 to 0.5 g. However, it revealed that the surfaces used had the finest weights of 0.05 g.This is because, at these weights, the absorption of acetamiprid does not affect the increase in the number of adsorbing surfaces because all of the operational sites of a adsorption surface have reached saturation (the saturation limit)(Hadi and Khdaim 2023). The relationship among the outer weight of absorption and the quantity of adsorption (mg/g) for (318 k) and the acidic functional pH = 7 is depicted in Figure 1C. As the adsorbent weight rises, acetamiprid removal decreases because the active sites become saturated over time(Hadi and Khdaim 2023). The Figure shows that the adsorption amount decreases as the surface weight increases.

3.3 Temperature Effect

The efficiency of the adsorption process on the activated charcoal surface largely depends on temperature. These tests were run at various conditions (318, 328, 338, and 348 K) to observe how this variable affected the acetamiprid adsorption process's capacity on the surface. Figure 1D showed how temperature affected acetamiprid adsorption, and the results showed that acetamiprid adsorption increased as temperature increased. This could be due to increased pore size or surface activation at high temperatures, as adsorption is an endothermic process (Jawad and Khadim 2022).

3.4 Effect of pH

In the absence of accurate absorption, by calculating the relative charge of the surface as a feature of the ion concentration that determines the charge at different concentrations of the apathetic electrolyte, the zero-charge point (pzc) of colloid or suspension can be achieved (Lyklema 1984). Figure 3 and Supplementary Figure 7 shows zero charge point for acetamiprid pesticide absorption on the surface of charcoal derived from Iraqi date kernels 15 mg / l of pesticide with 0.5 g of the charcoal surface of charcoal derived from Iraqi date kernels with a grade of 318 A and the range of the acid function 2-12 and the results indicate that the surface charge 10, this was confirmed by the results of the effect of acid function on the adsorption of the pesticide, as in Figure 4 (Sumanjit and Kaur 2007).

Adsorption equilibrium

The relationship between the amount of substance adsorbed at a constant temperature and its concentration in the equilibrium solution is called the adsorption isotherm, and it is important from a theoretical and practical point of view. Analyzing isotherm data by fitting it to different isotherm models is an important step in finding the appropriate model that can be used for design purposes and the applicability of isotherm models to adsorption. The study was conducted by Correlation coefficients R values. A range of isotherms Models have been tested. Freundlich, Langmuir, and TempkinDubinin Radushkevich (D-R).

Freundlich Isotherm Model

Freundlich isotherm is derived to a model of the multilayer adsorption, and for the adsorption on heterogeneous surfaces, the linearized form of the Freundlich equation is given by (Ji LiangLiang et al. 2010):

Log qe = log kf + 1/n log Ce

where kf and n are Freundlich constants, QE is the range of absorption of the pesticide per unit mass of adsorbent (mgg-1), and Ce is the equilibrium concentration of the pesticide (mgL-1). The log qe vs. log Ce chart will give the n and kf values of slope and itercept, respectively. The slope (Har and Sathasivam 2009) of 1/n between 0 and 1 measures the intensity of adsorption or

heterogeneity of the surface, becoming more homogeneous as its value approaches zero, while kf represents the amount of adsorption on the adsorbents. The values of the Freundlich constants with correlation coefficients are shown in Table 2 and Supplementary Figure 9. The results showed better suitability of experimental data for pesticide absorption, less agreement with (D-R), and less suitability with Langmuir and Tempkin.

Langmuir Isotherm Model

To understand the adsorption isotherm , the Langmuir equation is perhaps the most widely used model due to its simplicity and Strong theoretical reasoning (Sohn and Kim 2005). This model suggests monolayer sorption on a homogeneous surface without interaction between sorbed molecules(Saadi et al. 2015). In addition, the model assumes uniform energies of sorption on to the surface and no transmigration of the sorbate. The linearized form of the Langmuir isotherm equation is represented as(Ji LiangLiang et al. 2010):

Ce /qe = 1/kL qm + Ce /qm

where quantitative facilitation (mgg-1) is the amount absorbed per unit mass of adsorbent corresponding to the full coverage of sites, CE (mgl-1) is the concentration of pesticide equilibrium in solution, qm (mgg-1) is the single-layer adsorption capacity of the adsorbent and KL (lmg-1) is the adsorption energy. The relevant parameters are summarized in Table 2, and the Langmuir linear equation is shown in Supplementary Figure 10. The results reveal that the Langmuir model is unable to describe experimental data correctly.

Tempkin Isotherm Model

Tempkin isotherm assumes that heat of adsorption decreases linearly with the adsorption onto the surface at a particular temperature, and the adsorption is characterized by uniform distribution of binding energies(Imran Din et al. 2013)(Hameed, Tan, and Ahmad 2008). The tempkin has generally been applied in the following linear form:

Qe = B ln A+ B ln Ce

Where, B = RT/b

The qe vs ln ce chart makes it possible to determine the constants A and B. Table 2 Tempkin constants and correlation coefficients are listed in Supplementary Figure 11, this isothermal diagram is shown. The Tempkin equation can be used to describe a pesticide's absorption on the coal surface.

Dubinin - Radushkevich (D-R) Model

This isotherm model was chosen to estimate the characteristic porosity of the biomass and the apparent energy of adsorption(Maneerung et al. 2016). In general (D-R) isotherm model is subjected to experimental data to determine the nature of adsorption / biosorption processes either physical or chemical process, and this model is applicable at low concentration and can be used to describe sorption on both homogeneous and heterogeneous surfaces(Itodo and Itodo 2010)(Qok 2013). The linear form of (D-R) isotherm equation is(Ji LiangLiang et al. 2010):

ln qe=ln qm – KD R ε2

ε = RT ln [1+ (1/Ce)]

qm is the capacity of the single layer (D-R) (mgg-1), K is constant associated with adsorption energy, ε is polany absorption potential, R is gas constant, T is absolute temperature, Ceis equilibrium concentration (Beşel Düzgün 2014). A chart in qe vs ε2 is shown in Supplementary Figure 12. The values of constants (D-R) and correlation coefficients are listed in Table 2. The data points in this model appear to be good at compatibility with experimental data for pesticide absorption, and appear to be less consistent with the Freundlich and Tempkin equations but less poor than compatibility with the Langmuir equation.

3.4 Thermodynamic Parameters

The following formulas were used to calculate the enthalpy (ΔH), entropy (ΔS), and thermodynamic parameters(Khadim 2022):

ln Keq = ΔS0/R – ΔH0/RT …… (1)

where T is the temperature in degrees Celsius (Kelvin), R is the universal constant for gases (8.314.10-3 kJ/mol. K-1), K is the constant of the Vant Hoff equation, and ln Xeq is a natural logarithm for the largest amount adsorbed (mg/g).

ΔG°= – RT lnK …… (2)

ΔS°= ΔH – ΔG° / T …… (3)

l Kc against 1/T's Van't Hoff plot's slope and slope were used to calculate ΔH◦ and ΔS◦. (Supplementary Figure 13, Table 2). Since an increase in 1 / T causes a decrease in Ln Keq, the relationship between the two quantities (1 / T and Ln Keq) is negative. The findings demonstrated that a positive value for ΔH◦ indicated the endothermic character of the adsorption, and a negative value for ΔG indicated a spontaneous reaction(Shukla et al. 2020), as shown in Supplementary Figure 14 and Table 2.

Kinetic of Adsorption

The fictitious first-order equation was used to explain the kinetics of adsorption for the liquid-solid structure during this investigation(Shukla et al. 2020):

ln⁡(q_e- q_t)= 〖ln⁡q〗 _e– (K_1-K_(-1))t …….. (9)

In this example, qt is the amount of adsorbent material present at different times, K1 is the constant frontal contact velocity (min-1), and K-1 is the constant reverse interaction speed (min-1). Drawing ln(qe-qt) vs. t results in a straight line whose cross has a y-axis equal to lnqe and a slope equal to (K1+K-1). Using the following equation, define pseudo-second order(Konbr, Freewan, and Alshuk 2016):

t/q_t =1/(〖K_2 q〗 _e^2)+1/q_e t …….. (10)

Where K2 is the constant of sorption velocity of a pseudo-second order (g.mg -1.min -1), and the following equations are used to obtain K2, qe from the drawing of t/qt vs. t:

K2=Slop^2/intercept …….. (11)

qe=1/slope ……. (12)

4. Discussion

The study conducted to assess the effect of contact time on acetamiprid adsorption onto carbonated carbon derived from Iraqi date cores provides valuable insights into the kinetics of the adsorption process. The results indicate that the adsorption capacity increases with contact time until saturation is reached. This observation aligns with previous studies that suggest an initial rapid absorption phase followed by a plateau as saturation occurs. However, it's essential to critically examine some aspects of the findings and their implications.

Firstly, while the experiment identifies a neutral lifetime of three hours for acetamiprid, it's crucial to consider the practical implications of this timeframe. In real-world scenarios, such as water treatment processes or environmental remediation efforts, three hours may not always be feasible or sufficient. Therefore, understanding how this timeframe translates into practical applications and whether longer exposure times would significantly enhance adsorption efficiency warrants further investigation.

Moreover, while the relationship between contact time and adsorption capacity is well-established, the mechanisms underlying this phenomenon require deeper exploration. The mention of empty holes on the surface causing initial rapid absorption, followed by a decrease as these gaps fill up, provides valuable insight into the surface dynamics. However, a more detailed analysis of the molecular interactions between acetamiprid molecules and the adsorbent surface would enhance our understanding of the process.

Additionally, the experiment highlights the importance of surface weight on adsorption efficiency. While it's intuitive that increasing surface weight would provide more adsorption sites, leading to higher efficiency initially, the observation of decreased adsorption at higher weights due to surface saturation raises questions about the optimal weight-to-surface area ratio. Further investigation into optimizing this ratio could lead to more efficient adsorption processes.

Furthermore, the temperature effect on acetamiprid adsorption underscores the need to consider environmental factors in adsorption processes. However, the specific mechanisms driving increased adsorption at higher temperatures need clarification. Is it solely due to increased pore size or surface activation, or are there other factors at play? Understanding these mechanisms could inform the design of adsorption systems tailored to specific temperature conditions.

Regarding the adsorption equilibrium analysis, while the Freundlich model appears to fit the experimental data best, the discrepancies with other models, such as Langmuir and Tempkin, raise questions about the underlying assumptions of each model. Further exploration of these assumptions and their applicability to real-world scenarios would enhance the predictive power of adsorption models.

Finally, the determination of thermodynamic parameters and kinetic analysis provide valuable insights into the energetics and kinetics of the adsorption process. However, a critical examination of the assumptions made in calculating these parameters and their implications for practical applications is necessary.

while the experiment provides valuable insights into the kinetics and thermodynamics of acetamiprid adsorption onto carbonated carbon, further research is needed to elucidate the underlying mechanisms, optimize process parameters, and translate these findings into practical applications for water treatment and environmental remediation.

5. Conclusions

In conclusion, the findings from our study shed light on the behavior of the pesticide adsorption process and its thermodynamic aspects. The adherence to the multilayer Freundlich isotherm suggests a specific pattern in the adsorption process. Moreover, the calculations of thermodynamic functions, including Free energy ΔG, enthalpy ΔH, and entropy ΔS, provide insights into the energy changes and randomness of the system during adsorption. The positive enthalpy ΔH value indicates an endothermic process, while positive entropy values signify increased randomness with higher temperatures. The negative ΔG value also implies automatic adsorption under experimental conditions, with a resultant heat of adsorption comparable to physical adsorption. These findings contribute to a deeper understanding of pesticide adsorption behavior and provide valuable implications for further research in environmental remediation strategies.

Author contribution

H.A.F., L.H.K. analyzed data, designed experiments, wrote the manuscript. All authors read and approved for publication.

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Competing financial interests

The authors have no conflict of interest.

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