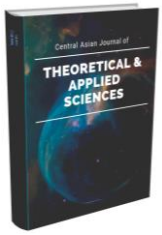

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Article

Thermodynamic and Kinetic Study of The Adsorption of Some Amines on The Surface of Bay Leaves

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Abstract: This research conducted a thermodynamic and kinetic investigation of the adsorption of environmentally detrimental organic amino compounds, such as aniline and diphenylamine, from aqueous solutions onto bay leaves as an adsorbent. The study examined the influence of various factors on adsorption, including initial adsorbate concentration, pH, and temperature. Additionally, adsorption isotherms were determined. The findings revealed that the interaction between the amino compounds and the bay leaf surface was rapid, reaching equilibrium within 50-70 minutes. Furthermore, adsorption increased with an increase in adsorbent dosage. Temperature and pH impacted adsorption significantly; higher temperatures and acidic conditions (pH 7) favored increased adsorption. The adsorption isotherms indicated a non-spontaneous process. Correlation coefficient analysis revealed a strong resemblance between the experimental data and the Freundlich isotherm while deviating from the Langmuir isotherm. Regarding adsorption kinetics, the study concluded that the process followed a pseudo-second-order model.

Keywords: Adsorption, Laurel, Isotherms, Thermodynamics

1. Introduction

Pollution, a pressing global challenge, necessitates effective remediation strategies. Human activities significantly contribute to pollution, exacerbating its severity [1]. Environmental pollution encompasses various forms, including soil contamination caused by chemical accumulation [2], air pollution from forest fires and toxic volatile compounds, groundwater pollution, and wastewater pollution from industrial discharges [3]. Traditional treatment methods, such as sedimentation, electrochemical oxidation and reduction, biological treatment, coagulation, and adsorption, have been employed to address these pollutants [4]. Adsorption, a widely used technique, has been the focus of extensive research, primarily on conventional, non-polar carbon surfaces. However, advancements in analytical methods and adsorption studies have enabled investigations into alternative surfaces, including bay leaves [5]. The bay plant, an evergreen conifer reaching 41 feet in height, exhibits dense, pointed, lanceolate leaves characterized by a vibrant green hue, especially in youth, and a distinct aromatic fragrance. Bay leaves contain bioactive compounds, such as flavones, luteolin, alkaloids, glycosides, and tannins, making them suitable as adsorbent materials [6].

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2. Materials and Methods

Apparatus used and chemicals, see Table 1 and 2.

Table 1. Laboratory equipment used.

Device Name	Its type and origin
UV/Vis Spectrum	UV.Visible, Spectrophotometer Shimadzu UV.Visible, Japan . 1700
Physiometric Instrument	HANA , PH-meter,112 Instrument , Romania
Distilled Water Instrument	Gell (Gasell Schaft fur labortechink) MbH ,W.Germany
Electric Sensitive Balance	Sartorius median. Electric Balance Germany
Electric Oven	Oven Memort / Germany
Electric Shaker with Water Bath	ShakingIndueator.GCA./Percision Scientific Chicago,U.S.A
Centrifuge	Centrifuge,Megafuga1.0/Herouse Sepatech

Table 2. Chemical compounds used.

Name of the substance	Chemical formula
Diphenylamine	$C_{12}H_{11}N$
Aniline	C_6H_7N
Ethanol	CH_3-CH_2OH

Preparation of Standard Organic Amino Acid Solutions

Stock solutions of the organic amino acids were prepared by dissolving 0.1 grams of each compound in a minimal volume of ethanol, followed by dilution with distilled water to a final volume of 1000 mL, resulting in a concentration of 100 ppm. Diluted solutions were subsequently prepared by taking appropriate volumes of the stock solutions and diluting them with distilled water to achieve concentrations ranging from 10 to 30 ppm. These solutions were allowed to stand undisturbed for an entire day to ensure complete homogeneity.

Determination of Maximum Absorption Wavelength (λmax) of Amino Compounds

To determine the λmax of the organic amino compounds, UV-visible spectrophotometry was employed. Solutions of the compounds were analyzed within a spectral range of 200-850 nm using a quartz cuvette with a path length of 1 cm. The absorbance values were recorded at the determined λmax for each compound as a function of concentration. Subsequently, a standard calibration curve was constructed by plotting the absorbance against concentration, as illustrated in Figure 1 and Table 3.

Table 3. Values of λ max for the two compounds under study.

The material	λ max
Aniline	286
Diphenylamine	380

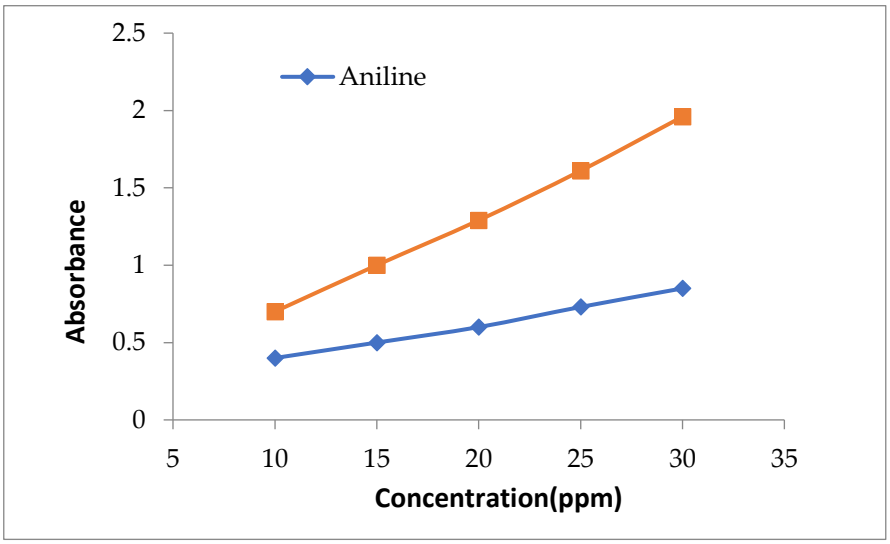

Figure 1. Calibration curve for the two compounds under study.

Determination of Optimal Adsorbent Dosage

To establish the optimal adsorbent dosage, varying amounts of bay leaves, ranging from 0.05 to 2 grams, were added to 30 mL of a 20 ppm amino acid solution in 50 mL volumetric flasks. The mixtures were agitated on a laboratory shaker for 60 minutes, and the absorbance of the resulting solutions was measured. The adsorbent dosage that yielded the lowest absorbance, indicating maximum adsorption, was determined to be 0.01 grams of bay leaves.

Determination of Equilibrium Time

To establish the equilibrium time for the adsorption process, twenty 50 mL volumetric flasks were filled with 30 mL of 20 ppm solutions of both amino compounds. Each flask contained 0.01 grams of bay leaves. The flasks were placed on a laboratory shaker at 25°C, and samples were withdrawn at predetermined intervals. The absorbance of these samples was measured to monitor the change in concentration over time. The optimal equilibrium times were determined to be 50 minutes for diphenylamine and 70 minutes for aniline.

Determination of Adsorption Isotherms

To investigate adsorption isotherms, six solutions of aniline and diphenylamine were prepared at concentrations ranging from 10 to 30 ppm. Thirty milliliters of each concentration were placed in contact with 0.01 grams of bay leaves in 50 mL volumetric flasks. The flasks were agitated on a laboratory shaker until equilibrium was reached, as determined previously. The solutions were then centrifuged, filtered, and analyzed using UV-visible spectrophotometry. The equilibrium concentration (Ce) was determined from the calibration curves. The amount of adsorbed material (Qe) was calculated using the following equation [7]:

$$Qe = (C_0 - Ce) * Vsol / M \quad \text{……..(1)}$$

where:

Qe: amount of adsorbent (mg/g)

C_0: initial concentration of adsorbent (mg/L)

Ce: residual concentration of adsorbent at equilibrium (mg/L)

Vsol: total volume of adsorbent solution (L)

M: weight of adsorbent (g)

Freundlich Isotherm

The adsorption data were analyzed using the Freundlich isotherm, represented by the following equation:

$$\log Qe = \log Kf + (1/n) * \log Ce \quad \text{……..(2)}$$

The linear relationship between log Qe and log Ce, as depicted in Figure 3, indicates the suitability of the Freundlich model for describing the adsorption of these amino compounds by bay leaves.

Langmuir Isotherm

The Langmuir isotherm is expressed by the following equation:

Ce/Qe = 1/Kf * Ce + 1/Qmax ……..(3)

The adsorption data for the two amino compounds were analyzed using the linear form of the Langmuir equation. A plot of Ce/Qe versus Ce resulted in a linear relationship, as shown in Figure 4, indicating the applicability of the Langmuir model to describe the adsorption of these.

Effect of temperature on adsorption

To evaluate the effect of temperature change on the adsorption process of the two studied materials, a series of experiments were conducted by changing the temperature of the system. These experiments included determining the adsorption isotherms at different temperatures, which are (25, 35, 45, 55) degrees Celsius.

The effect of acidity on adsorption

The adsorption of aniline and diphenylamine to the surface of bay leaves was studied in the same way as mentioned above, but at pH values of (5, 7, 9), and the acidity of the solution was adjusted using standard buffer solutions and using a (pH-meter) device.

Thermodynamic study

The thermodynamic study includes calculating the values of ΔS°, ΔG°, and ΔH°. The values of enthalpy ΔH° were calculated through the van't Hoff equation (8) by plotting $\log Qe_{max}$ versus the reciprocal of temperature (1/T).

$$\log Qe_{max} = \frac{-\Delta H^{\circ}}{RT} + \text{Con} \quad \ldots\ldots\ldots\ldots\ldots (4)$$

Where:

$\log Qe_{max}$: Logarithm of the greatest adsorbed quantity.

Con: constant of the van't Hoff equation.

T: temperature (Kelvin).

R: the general gas constant.

A linear relationship is obtained, and from the slope of the lines in Figure (9), the value of ΔH° is calculated, since the value of the slope is (- ΔH° / 2.303R).

The value of the change in free energy °ΔG is also calculated using the equation:

ΔG° = - RT Ln (Qe/Ce) ……(5)

The values of entropy change ΔS° are calculated from the equation:

ΔG° = ΔH° - TΔS° ……(6)

Kinetic study of adsorption

The research included the application of two kinetic models to the practical data for the adsorption of the studied systems, which are the false first and second-order equations. This study was completed at different concentrations of each substance and a constant temperature of 298 °C using bay leaves and at the natural acidity function. The false first order model or what is called the (Lagergren) equation was applied, which is:

ln (Qe – Qt) = lnQe – (k1+ k-1)t ……(7)

The drawing was done by drawing the relationship between ln (Qe – Qt) versus time, which is supposed to give a linear relationship with a slope of (-k1) and intersect the y-axis at the value of (lnQe). For the studied system to be subject to the first-order law, the linear relationship with the correlation coefficient (R2) must be close to the value of (1) throughout the adsorption process. In addition, the value of qe calculated from the graph must agree with the practical value calculated when the studied system reaches the state

of equilibrium, and any defect in achieving one of these two conditions makes it impossible for this kinetic model to apply to the practical adsorption data.

The false second-order equation model was also applied to the experimental data for the adsorption of the two substances on bay leaves under the same optimal conditions adopted when applying the false first-order equation of temperature and concentration of the adsorbent substance mentioned previously. The theoretical adsorption capacity value was calculated from the graph and the value of the velocity constant (k2) from the slope and the straight line segment was obtained from drawing the relationship between t/Qt versus time t from the following equation:

$$\frac{t}{Q_t} = \frac{1}{k_2 Q_e^2} + \frac{1}{Q_e} t \ldots\ldots\ldots\ldots\ldots (8)$$

The conditions for applying the false second-order equation to the adsorption system are similar to those mentioned in the case of applying the first-order equation in terms of the conditions for the availability of the strength of the linear relationship and the agreement between the value of the adsorption capacity Qe calculated practically and that calculated from the graph.

3. Results and Discussion

Adsorption isotherms

When studying the adsorption of aniline and diphenylamine compounds on the surface of bay leaf, the adsorption isotherms were obtained as in the figures below at a temperature of (25) ℃ and an acidity function (pH=7). Figure 2 and 3 shows that the general form of the adsorption isotherms follows the Freundlich isotherms, which indicates that the surface of the adsorbent is a heterogeneous surface and different physical forces arise on this surface, including the hydrogen bonding forces that occur between the active functional groups in the adsorbed amino compounds and the hydroxyl groups and the van der Waals forces present on the surface of the bay leaf [8].

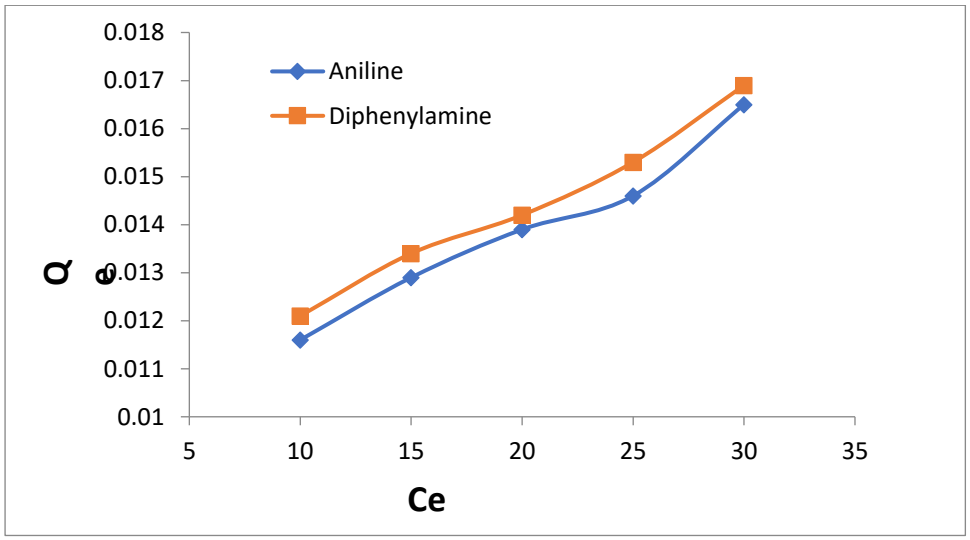

Figure 2. Adsorption of the two compounds (aniline and diphenylamine) used at a temperature of (25)℃ and pH = 7.

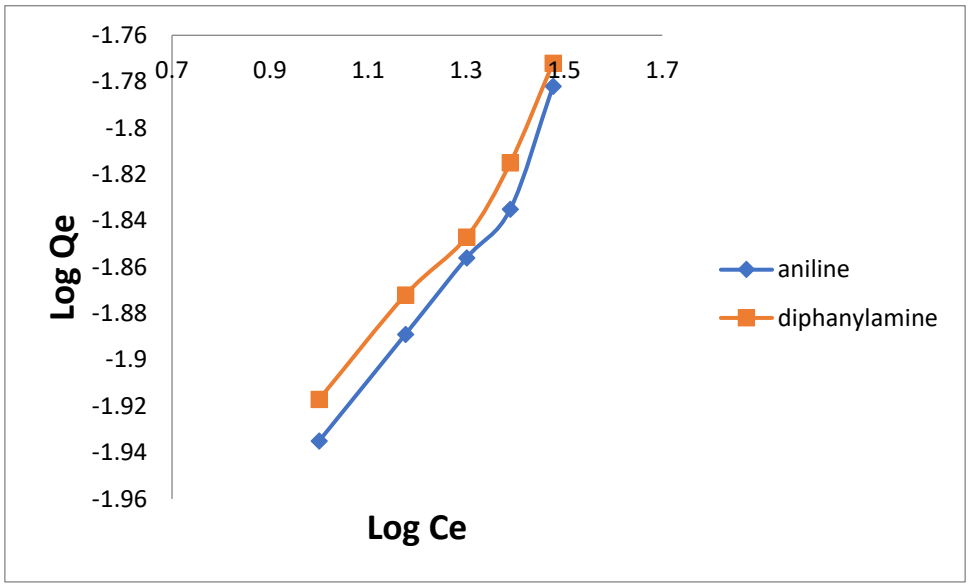

Figure 3. Freundlich lines for the adsorption of the two amino compounds at a temperature of (25)°C.

The experimental Freundlich constants and the correlation coefficient were calculated from the data of these lines, as shown in Table 4 and Figure 4.

Table 4. Freundlich experimental constants and correlation coefficient.

Compound	K_f	n	R^2
Aniline	0.00572	3.134	0.9671
Diphanylamine	0.00612	3.444	0,9713

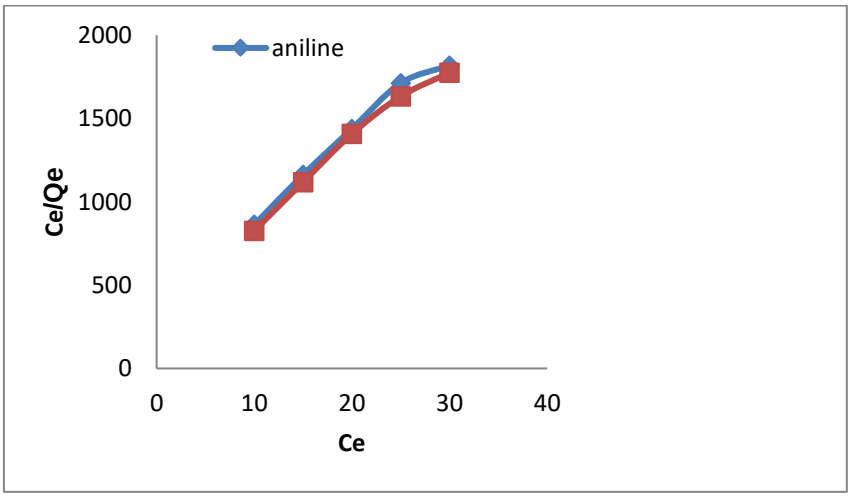

Figure 4. Langmuir lines for the adsorption of the two amino compounds at a temperature of (25)°C.

The substituted groups have an effect on the aromatic ring in its adsorption on the surface of bay leaves and it was found that its adsorption capacity increases according to the following order: Diphenylamine < Aniline The absence of a substituted group on the aniline ring makes it more stable as the orientation of the molecule is perpendicular to the surface and thus occupies a small surface area, while the compensation is with a driving group such as (-CH3) as this group works to reduce the electronic affinity of the ring, which

reduces the stability of the complex formed on the surface and thus a slight decrease in its adsorption capacity [9].

The electron-withdrawing substituent group such as (-NO2) leads to a greater decrease in adsorption. This is due to the inductive effect of this group, as it works to reduce the electronic density of the nitrogen atom and increase it on the two oxygen atoms, i.e. increasing the electronegativity of the two oxygen atoms of the nitro group, and then causing repulsion with the negative surface sites, which leads to making the complex formed with the surface less stable. However, the nitro group tends to form a hydrogen bond with water, which increases the ability of the molecule to bind to water and dissolve in it more than its tendency to bind to the surface [10].

Effect of temperature on adsorption

The effect of temperature on the adsorption of the two amino compounds on the surface of bay leaf was studied at a range of experimental temperatures (25, 35, 45, 55)°C. Figures 5 and 6 show the adsorption isotherms related to these temperatures.

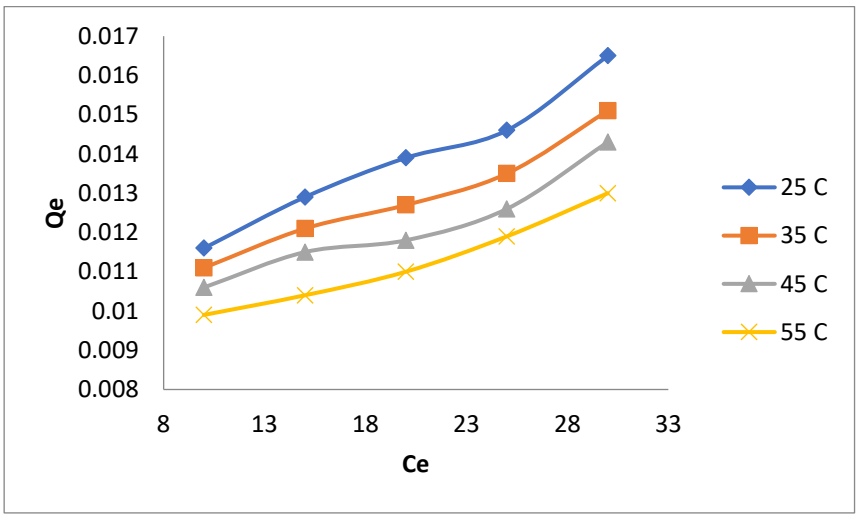

Figure 5. Aniline adsorption isotherm at different temperatures.

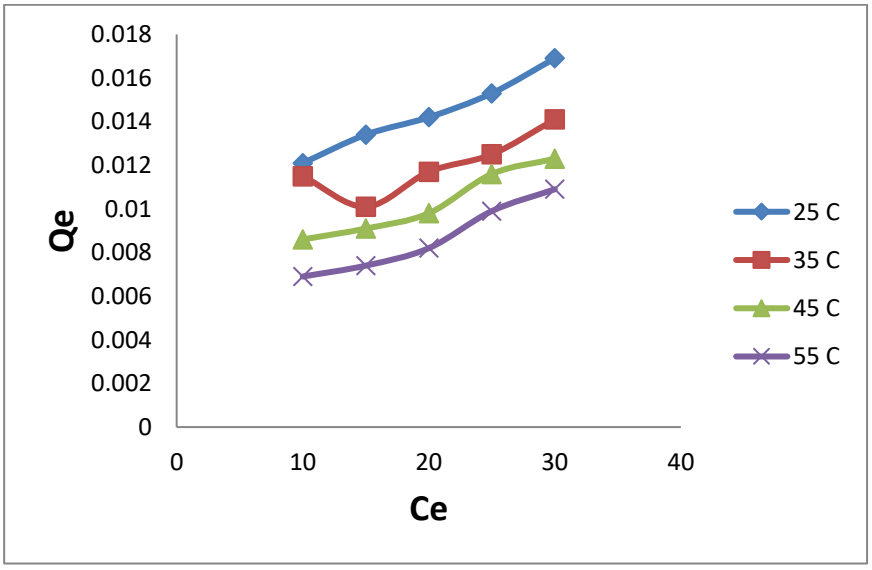

Figure 6. Adsorption isotherm of diphenylamine at different temperatures.

The experimental results showed that the adsorption of these two compounds decreases with increasing temperature, i.e. the adsorption process is exothermic, and this

is consistent with the thermodynamic study of the adsorption process, as increasing the temperature leads to an increase in the kinetic energy of the molecules adsorbed on the adsorbent surface, which leads to an increase in the probability of their separation from the adsorbent surface and their return to the solution [11]. The temperature affects the solubility of the adsorbed substance, so if the solubility increases with increasing temperature, this leads to a decrease in adsorption [12].

Effect of pH on adsorption isotherm

The effect of pH on the adsorption of the two amino compounds used in this study on the surface of laurel leaves was studied at different pH values (pH= 5, 7, 9). The results were as shown in Figures 7 and 8, that the adsorption amount of the two compounds is at its highest value when the solution is neutral (pH= 7) and decreases when the solution is acidic (pH= 5) and decreases more when the solution is basic (pH= 9), according to the following order, see Figure 7 and 8.

pH=9<5<7

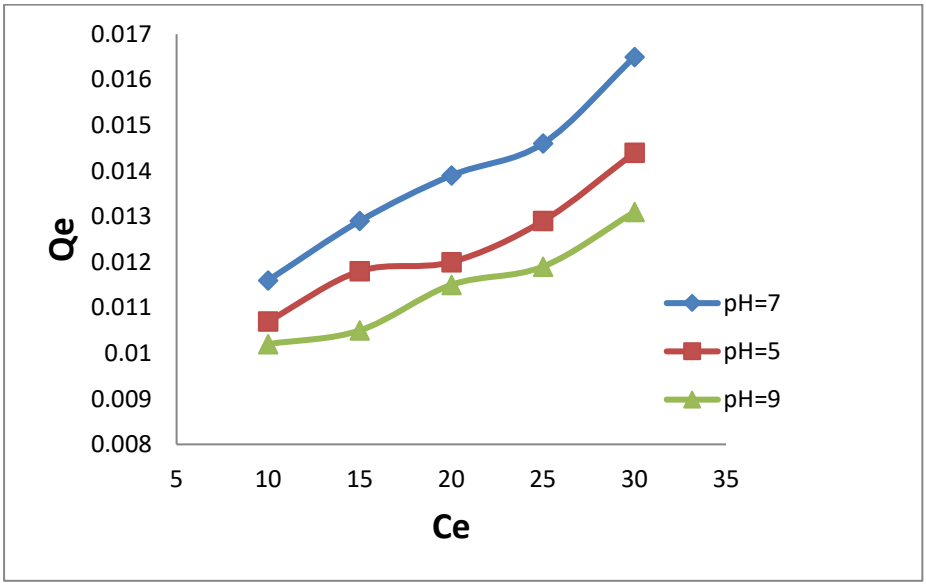

Figure 7. Effect of solution acidity on aniline adsorption at 25°C.

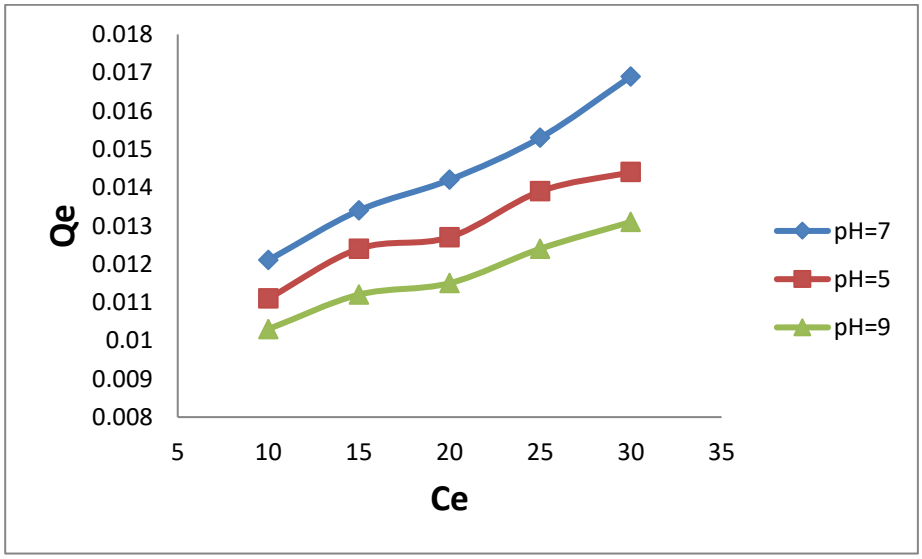

Figure 8. Effect of solution acidity on the adsorption of diphenylamine at a temperature of 25°C.

These results can be explained by the fact that in a neutral medium, the probability of hydrogen bonding between the hydroxyl group on the surface and the active (NH2) group is greater, i.e. the tendency of amino molecules to bind to the surface is greater than their tendency to bind to solvent molecules. In an acidic medium, the amino group acquires a proton and turns into a positively charged anilinium ion, and the surface carries positive and negative charges, so electrostatic attraction occurs between the different charges only, and thus the amount of adsorption decreases [13]. In a basic solution, the surface acquires more negative charges under the influence of this solution, and the amino molecules also carry negative charges, so electrostatic repulsion occurs between the negative charges, and this is the reason why the amount of adsorption decreases greatly [14].

Thermodynamics of Adsorption

Adsorption is a phenomenon that can be either endothermic or exothermic depending on the adsorbent and the nature of the adsorbed molecules (15). The thermodynamic functions that must be considered to determine the process are the changes in standard enthalpy ΔH°, standard free energy ΔG°, and standard entropy ΔS°, see Figure 9 and Table 5.

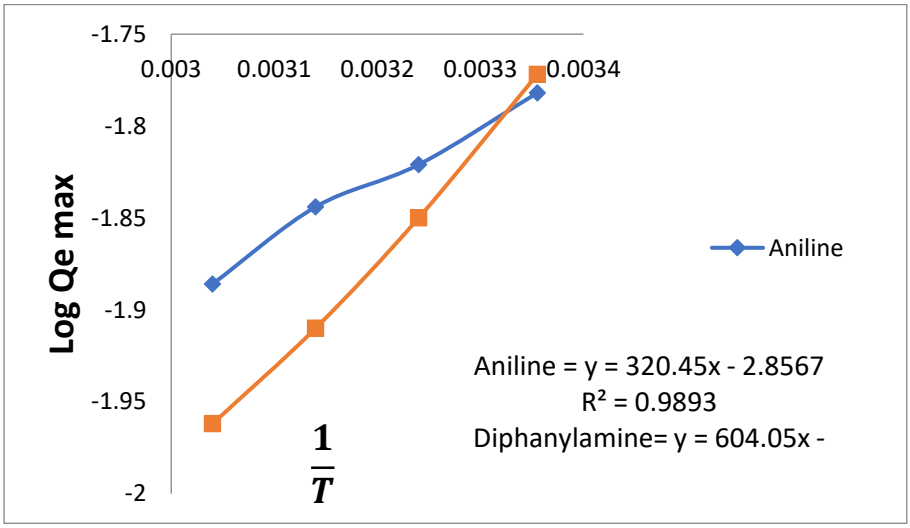

Figure 9. shows the relationship between the logarithm of the maximum excellent quantity and the reciprocal of the temperature for the adsorption of the two amino compounds.

Table 5. Shows the thermodynamic values of the two amino compounds on the surface of bay leaf at a temperature of 25°C.

Compound	ΔH° (KJ/mol)	ΔG° (KJ/mol)	ΔS° (J/mol.K)
Aniline	-6.133	18.595	-20.64
Diphanylamine	-11.561	18.551	-101.04

The negative values of the standard enthalpy ΔH° indicate that the adsorption process of the two amino compounds on the surface of the bay leaves is exothermic and low ΔH<40Kj/mol and its value falls within the heat of physical adsorption. It also indicates a decrease in the mutual interaction between the adsorbed molecules and the surface with increasing temperature due to the separation of the bonds between the adsorbed surface and the adsorbed molecules [15].

The positive values of the free energy ΔG° indicate that the adsorption of the two amino compounds on the surface is non-spontaneous under these experimental conditions, as shown in previous studies [16]. As for the values of the change in entropy ΔS°, they are an indication that the adsorbed molecules are arranged on the surface as a result of their association with the adsorbed surface, i.e. the adsorbed and intermingled

molecules are less organized than their form in the solution when the two processes of adsorption and desorption occur together [17].

Kinetic study of adsorption

The false first-order equation, or what is called the Lagergren equation, was relied upon to extract the adsorption rate constant, as this method depends on the difference between the amount of the adsorbed substance at equilibrium (Qe) and its amount at time (Qt), as the results obtained were treated according to this equation, see Figure 10 and 11.

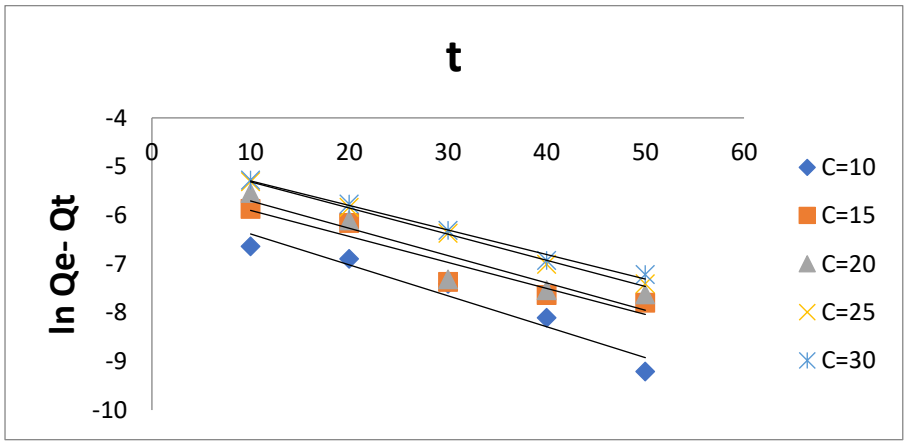

Figure 10. Lagergren lines plotted for the adsorption of aniline at different concentrations and at constant temperature.

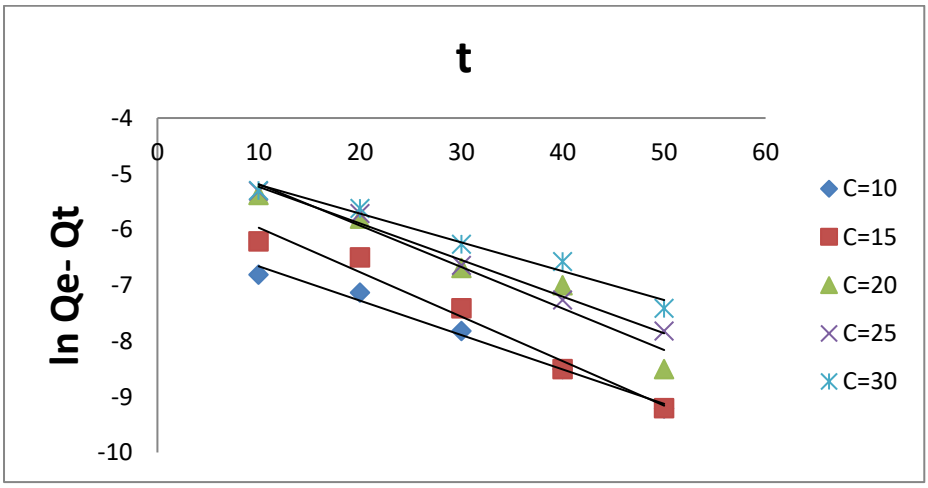

Figure 11. Lagergren diagram for the adsorption of diphenylamine at different concentrations and at constant temperature.

Table 6. Values of the adsorption rate constant, the amount of adsorbed material, and the correlation coefficient at different concentrations of aniline. False first-order equation.

Concentration(ppm)	Original Qe	k_1	Calculated Qe	R^2
10	0.0118	0.0617	0.00237	0.985
15	0.0148	0.0798	0.00568	0.973
20	0.0184	0.0744	0.0117	0.941
25	0.0186	0.0657	0.0102	0.988
30	0.0195	0.0517	0.00926	0.97

Table 7. Values of the adsorption rate constant, the amount of adsorbed material, and the correlation coefficient at different concentrations of diphenylamine by applying the false first-order equation.

Concentration(ppm)	Original Qe	k_1	Calculated Qe	R^2
10	0.0124	0.0635	0.00318	0.940
15	0.0159	0.0534	0.00467	0.901
20	0.0175	0.0561	0.00582	0.882
25	0.0188	0.0537	0.00842	0.996
30	0.0193	0.0504	0.00831	0.990

When matching the obtained practical results with the final form of the false first-order equation numbered 7 and as shown in Figures 10 and 11 and Tables 6 and 7, we notice the inadequacy of this model for the studied adsorption systems through the relatively low values of the correlation coefficients as well as the divergence of the values of the adsorption quantity (Qe) calculated through the drawing and comparing them with the original values.

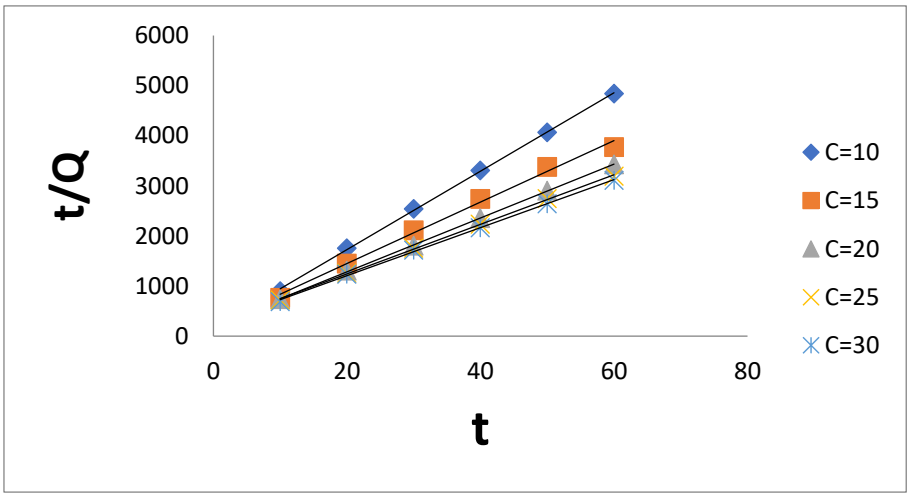

Figure 12. Pseudo-second order lines for the adsorption of aniline at different concentrations and at constant temperature.

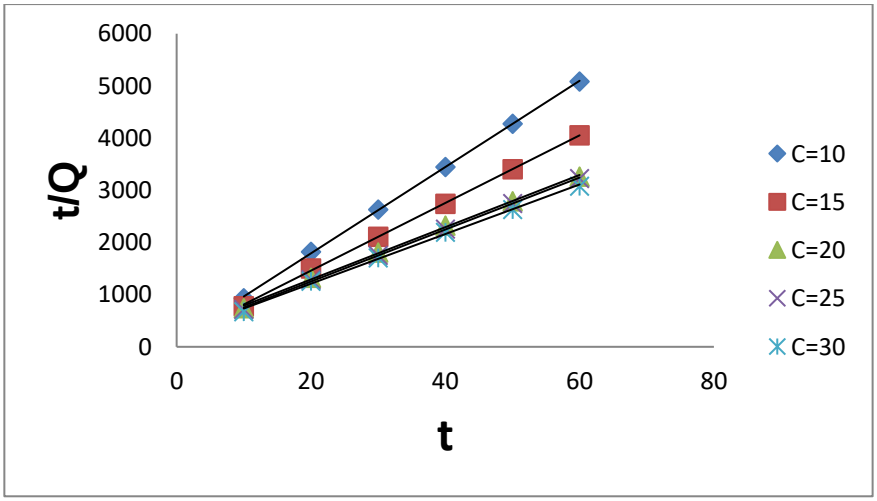

Figure 13. Pseudo-second order diagrams for the adsorption of diphenylamine at different concentrations and at constant temperature.

Table 8. Values of the adsorption rate constant, the amount of adsorbed material, and the correlation coefficient at different concentrations of aniline by applying the false second-order equation.

Concentration(ppm)	Original Qe	k_1	Calculated Qe	R^2
10	0.0118	50.50	0.0120	0.999
15	0.0148	25.83	0.0154	0.999
20	0.0184	8.71	0.0199	0.998
25	0.0186	9.80	0.0200	0.999
30	0.0195	8.77	0.0210	0.997

Table 9. Values of the adsorption rate constant, the amount of adsorbed material, and the correlation coefficient at different concentrations of diphenylamine by applying the false second-order equation.

Concentration(ppm)	Original Qe	k_1	Calculated Qe	R^2
10	0.0124	37.87	0.0127	0.999
15	0.0159	16.92	0.0163	0.994
20	0.0175	13.86	0.0186	0.999
25	0.0188	9.68	0.0202	0.999
30	0.0193	9.27	0.0208	0.999

As for the false second-order equation numbered 8, which was used to analyze the adsorption kinetics of the studied systems, it gave a straight line when drawing the relationship, and the values of the amount of adsorbed material were calculated from the slope and the values of the adsorption rate constant from the values of the intercept. We note that this model largely matches the practical results obtained for the two adsorption systems, as the correlation coefficients R2 are all very close to 1.00 and greater than 0.99.

4. Conclusion

This study demonstrated that bay leaves (laurel) can serve as an effective, natural, and low-cost biosorbent for the removal of toxic organic amines such as aniline and diphenylamine from aqueous solutions. Through thermodynamic and kinetic evaluations, it was found that the adsorption process was influenced by multiple factors including adsorbent dosage, pH level, temperature, and initial concentration of the adsorbate. Maximum adsorption was achieved at neutral pH (7) and lower temperatures (25°C), indicating that the adsorption process is exothermic and more favorable in slightly acidic to neutral conditions. Equilibrium times were relatively short (50–70 minutes), and the optimal adsorbent dosage was determined to be 0.01 g.

The adsorption isotherms were best described by the Freundlich model, suggesting that the surface of bay leaves is heterogeneous, and the adsorption is multilayered in nature. In contrast, the Langmuir model, which assumes homogeneous monolayer adsorption, showed lower correlation. Thermodynamic parameters supported these findings: $\Delta H°$ was negative, confirming an exothermic process, while positive $\Delta G°$ values indicated that the adsorption was non-spontaneous under the tested conditions. Entropy change ($\Delta S°$) values suggested a more ordered system upon adsorption due to interaction between amine molecules and the functional groups on bay leaf surfaces.

Kinetic analysis revealed that the adsorption followed a pseudo-second-order model, as evidenced by the high correlation coefficients ($R^2 > 0.99$) and close agreement between calculated and experimental adsorption capacities. This indicates that chemisorption is likely the rate-controlling mechanism, involving valence forces through electron sharing or exchange. Overall, this study highlights the sustainability, efficiency,

and environmental compatibility of using bay leaf powder as a biosorbent material for water purification applications involving organic pollutants.

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