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Some parameters influencing the uptake of industrial Acid blue 113 dye using chitin as natural adsorbent: Equilibrium and Isotherm studies

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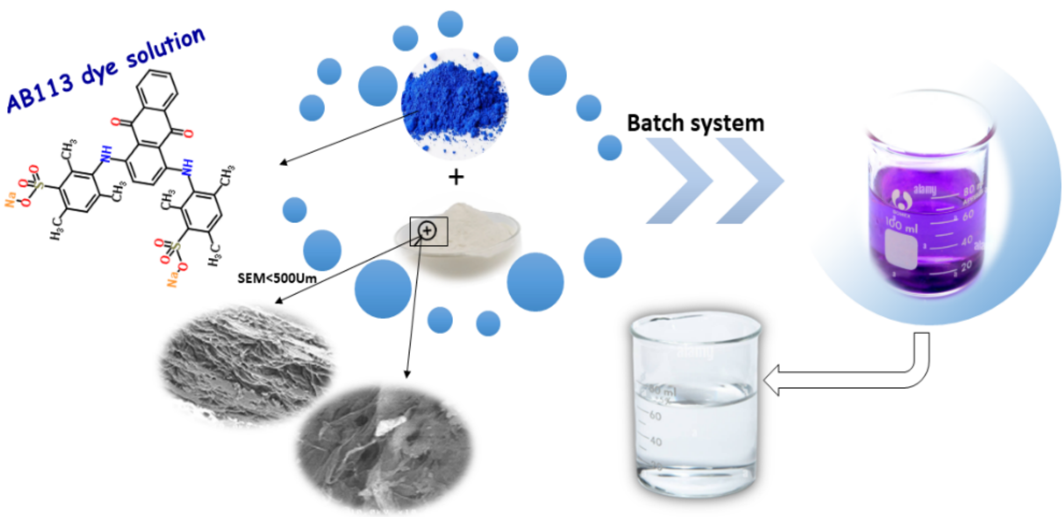
Abstract: In the present study, we aimed to evaluate the potentiality of chitin as eco-friendly adsorbent for the removal of industrial dye (Acid Blue 113) from aqueous solutions using the static system under different experimental conditions. The sorption properties of the adsorbent used for the removal of an anionic dye Acid Blue 113 were studied and the results obtained showed that, at initial concentrations of 20 and 100 mg/L at room temperature, the chitin microparticles had a removal percentage higher than 85%. The effect of different environmental factors on the adsorption of AB113 dye was also studied. The results showed that the isotherms were well described by the Langmuir model justifying a monolayer and homogeneous adsorption. Scientifically, it can be concluded that chitin, as an environmentally friendly adsorbent, could potentially be used for the removal of industrial anionic dyes from aqueous solutions.

Keywords: Adsorption, Industrial anionic dyes, Acid bleu 113, low-cost, Chitin



1. Introduction

Dyes are widely used in textile, paper, rubber, plastic, leather, cosmetic, and pharmaceutical and food industries. These pose an aesthetic and health problem because many of dyes are toxic. Many various physicochemical techniques have been developed to remove industrial anionic dyes from both water and industrial wastewater (Saravanan *et al.,* 2021, Khan *et al.,* 2018; Medjahed *et al.* 2013), including precipitation, coagulation, flocculation, reverse osmosis, filtration, ozonation, and adsorption process (Akar *et al.,* 2006, Ghaedi *et al.,* 2012, Chiban *et al.,* 2013, Gupta *et.al.,* 2016, Aziam *et al.,* 2016, Chiban *et al.,* 2011a). Adsorption is one the method for separation of mixtures on a laboratory and industrial scale where it is a surface phenomenon that can be defined as the increase in concentration of a particular component at the interface between two phases (Dabrowski, 2001, Noll *et al.,*1992). Adsorption of dyes depends on the properties of the dye and the surface chemistry of the adsorbent (Noroozi *et al.,* 2007).

Different adsorbents derived from dried plants and agricultural solid wastes have been used for dye removal from wastewater and many studies of dye adsorption by agricultural solid wastes have been published (Chiban, 2011b, Akartasse *et al.* 2023; Aaddouz *et al.* 2023). Chitin may be part of these materials due to its intrinsic properties, its abundance and low cost. It is mainly extracted from the shells of crustaceans such as crabs, shrimp and lobsters (Abidar *et al.,* 2015, Morghi *et al.,* 2015). Chitin is the second most abundant polymer in nature after cellulose.

Chitin derivative have gained wide attention as effective bio adsorbent due to low cost and high contents of amino and hydroxyl functional groups which show significant adsorption potential for the removal of various aquatic pollutants such as industrial anionic dyes. The AB113 dye chosen as a model compound for the present study finds vast applications as a coloring agent of the textile industry. The aim of this work is to evaluate the potentiality of chitin as low-cost adsorbent for the removal of AB113 from aqueous solutions using the batch equilibration technique. The effect of some environmental parameters such as adsorbent dose, contact time, initial dye concentration, solution pH, temperature on adsorption capacity of AB113 dye onto chitin were investigated.

2. Materials and methods

2.1. *Materials*

The organic pollutant studied in this work is an anionic dye with the chemical formula $C_{32}H_{21}N_5O_6S_2Na_2$ (C.I. N° 26360, CAS N° 3351-05-1 and M. W= 681.65 g/mol) were purchased from from Sigma-Aldrich (Fig. 1). A stock solution of 1000 mg. L^{-1} in industrial anionic dye was prepared by dissolving accurately weighed quantity of AB113 dye in double distilled water. The pH of the test solution was adjusted using reagent grade dilute chloridric acid and sodium hydroxide.

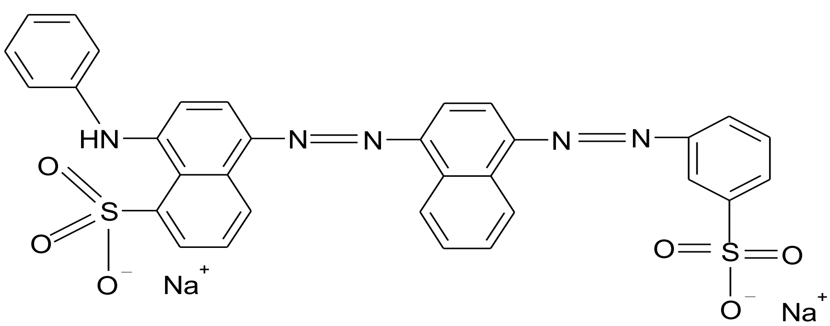


Fig. 1: Molecular structure of AB113

The adsorbent used in this study, chitin, is a linear beta 1,4-linked polymer of N-acetyl-D-glucosamine (Fig. 2). It differs from the other polysaccharides by the presence of nitrogen in addition to carbon,

hydrogen and oxygen in the macromolecular chain (Erradi et Jaafari, 2022, Tokura et Tamura., 2007). Commercially available chitin (CAS N° 1398-61-4 and M.W. = 400,000 g.mol^{-1}) was obtained in the form of flakes from Loba Chemie Pvt., Mumbai, India. Chitin powder was sieved to give different particle sizes less than 500 µm. The commercial chitin was used in adsorptions experiments without further purification.

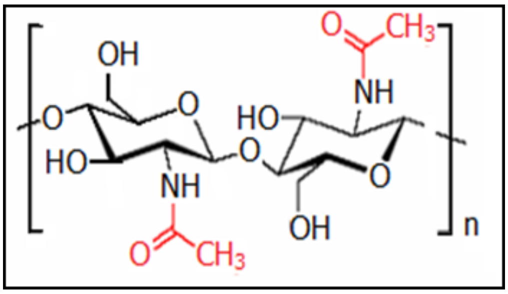


Fig 2: Chemical structure of Chitin

2.2. Instrumentation

The instrument used for the determination of AB113 dye concentration was a Tech Comp UV 2300 Spectrophotometer at the respective λ_{max} value, which is 566 nm for AB113.
The pH values of solutions before and after adsorption were measured by a Mettler-Toledo meter (MP120). The surface structure, morphology and the elements of chitin particles were measured by scanning electron microscope coupled with energy dispersive X-ray analysis (SEM/ EDX, HITACHI S-4500, IEMM, Montpellier-France).

2.3. Static adsorption experiments

Adsorption experiments were made by a static technique at room temperature (except when the effect of temperature was studied). Known amounts of chitin were placed in different stoppered Erlenmeyer glass flasks of 100 mL capacity containing 40 mL of acid blue 113of known concentration and pH. The agitation speed was kept constant for each run to ensure equal mixing. After different contact times (t), the final samples were centrifuged at 5000 rpm for 10 min and the supernatant was filtered through a 0.45µm membrane filter and the filtrate was analyzed. The residual concentration of dye solution was determined using UV-Visible.

2.4. Apparent capacity measurements

The AB113 dye concentration removed (C_r, mg/L) from the aqueous solution was calculated as a difference between the initial concentration (C_0, mg/L) and the concentration at different contact time (C_t, mg/L). The percentage removal (%) of AB113 dye and the uptake capacity (q_t, mg/g) per mass unit of the chitin at time't' and were calculated as follows (Nguyen *et al.,* 2021, Aziam *et al.,* 2016):

$$\textbf{\% removal} = \frac{(C_0 - C_t)}{C0} * 100 \quad \textbf{(Eq. 1)}$$

$$q_t = (C_0 - C_t) \times \frac{V}{m} \quad \textbf{(Eq. 2)}$$

where C_o (mg/l) is the initial concentration of AB113 dye in aqueous solutions; C_t (mg/l) is the concentration of AB113 dye left in aqueous solutions at time't'; q_t (mg/g) is the uptake removed per mass unit of the chitin at time't'; m (g) is the weight of the chitin and V (L) is the working solution volume.

3. Results and discussion

3.1. Characterization of the adsorbent

A detailed characterization of chitin adsorbent has been done by different techniques as it was described in our previous report (Abidar *et al.,* 2015, Morghi *et al.,* 2015). The scanning electron microscope (SEM) of chitin (Fig. 3) shows a homogeneous surface, microscopic multilayer superposables with net interstices between sheets and a high porosity. The structure of chitin possesses several functional groups to create affinities (hydrogen bonds, electro-static interaction ...) with several chemical entities with pollution effects as evidenced by its IR spectrum (Table not shown) which is consistent with literature data (Abidar *et al.,* 2016, Brugnerotto *et al.,* 2001).

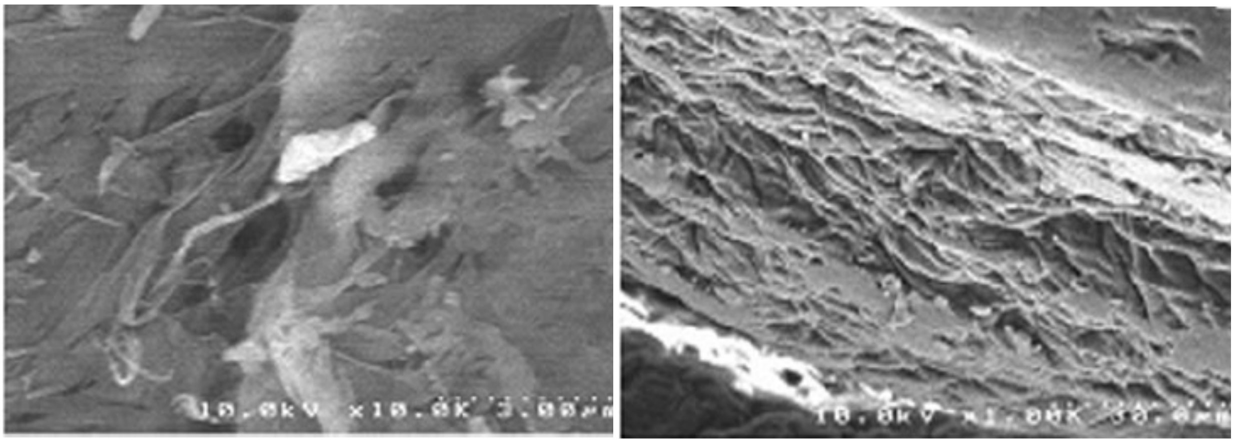


Fig. 3. SEM chitin, particle size < 500 µm

The solution pH is a significant parameter which affects the dye adsorption process. It also alters the surface charge of the adsorbent, the ionization extent of different pollutants, as well as the structure of dyes. The effect of pH on AB113 adsorption can be illustrated on the basis of zero point charge of the adsorbent surface. The presence of various ligands such as carboxyl, phosphate, and amino group on lignin- and cellulose-based materials in the ionic state contributes to the reaction with industrial dyes. The zero point charge, pH_{zpc} of chitin, was found to be 6.7 (Figure not shown). At pH_{zpc}, the acidic and basic functional groups no longer contribute to the pH of the solutions. At pH values of the solution below the pHzpc, the predominate chitin surface species is positively charged and can attract anionic dyes from the solution, while at pH values above the pH_{zpc}, the predominate surface species of chitin is negatively charged and can attract cationic dyes from the solution. Thus, at pH < 6.7, the chitin surface has a high positive charge density, so the uptake of negatively charged AB113 dye would be high. At pH > 6.7, the chitin surface has a high negative charge density, so the uptake of negatively charged AB113 dye would be low.

3.2. Effect of adsorbent dosage on dye removal

Adsorbent dosage is an important parameter because this factor determines the capacity of an adsorbent for a given initial concentration of the adsorbate (Chiban, 2011b, Benhima *et al.,* 2008). The effect of adsorbent dosage on the removal of AB113 at C_0 = 20 mg/L and 100 mg/L was studied by shaking different chitin masses, while other conditions remained the same as the adsorption studies. Fig. 4 shows the removed concentration of AB113 as a function of adsorbent dosage. It is found that the concentration selected stabilizes at a maximum value corresponding to a ratio (m/V) equal to 2.5 g/L (0.1g/40mL). This result shows that 0.1 g of chitin with 40 mL of solution is sufficient to achieve the maximum adsorption for AB113 dye. For higher adsorbent concentration of 2.5 g/L, the removal

efficiency becomes almost constant for the removal of AB113 onto microparticles of chitin. In order to achieve a maximum adsorption of the acid blue 113, the m/V ratio of 2.5 g/L was selected for further studies.

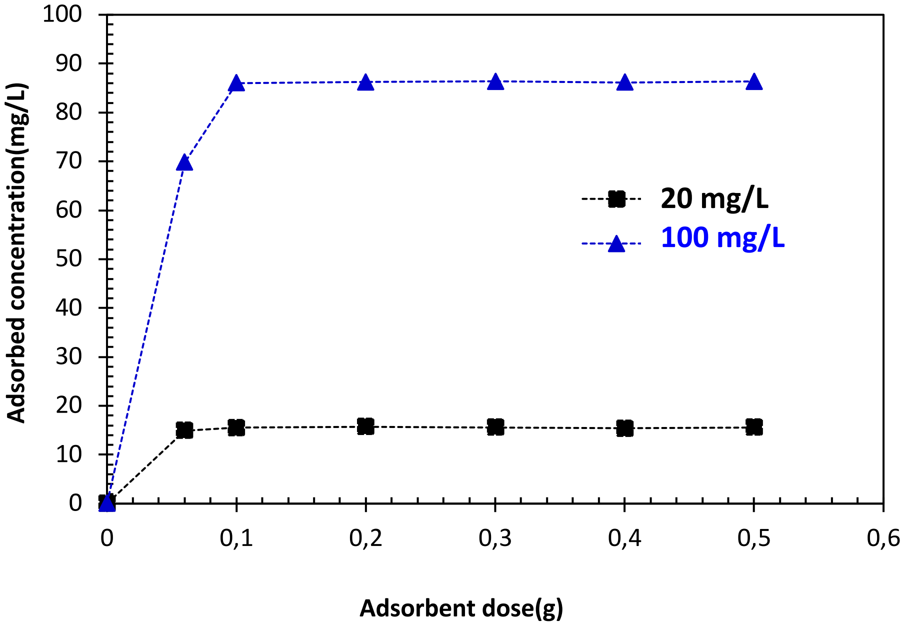


Fig. 4. Effect of chitin amount on the removal of AB113: T=23±2°C, pH= 6.3 and t_c= 6h.

3.3. Effect of contact time on dye removal

The effect of contact time on the adsorption of Acid blue 113 dye was performed for two initial concentrations: C_i=20 mg/L and C_i= 100mg/l. The impact of contact time on the adsorption of industrial dye solution containing 20 mg/l and 100 mg/l of acid blue at room temperature (23 ±2 °C) and initial pH value 6.3 was shown in Fig 5.

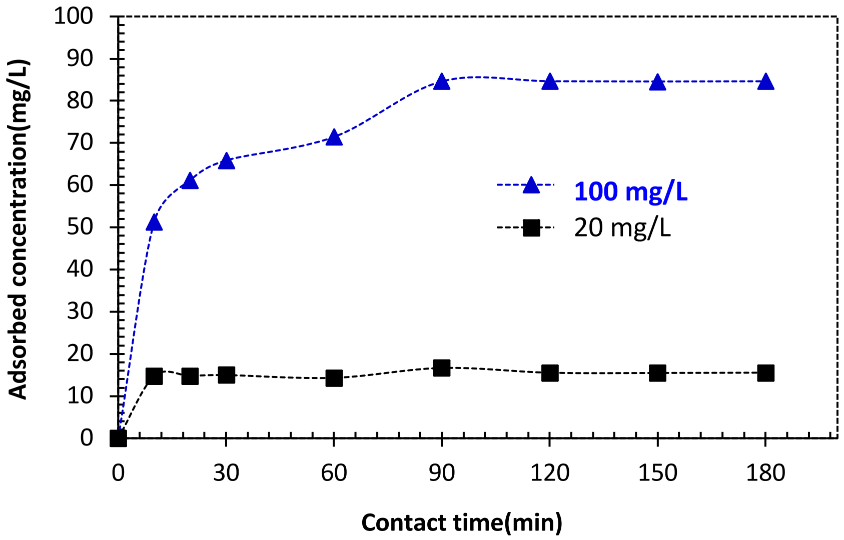


Fig.5. Effect of contact time on the removal of acid blue 113 onto chitin m/V=2.5 g/L, pHi=6.3 and T=23°C

It is noted that the uptake adsorbed of AB113 dye retaining by chitin surface increases with contact time. This adsorption is very fast in the first 15 min. Then, the adsorption equilibrium is reached after a contact time of about 60 min for both initial concentrations (20 and 100 mg/L). The percentage

removed of Acid blue 113 dye at equilibrium is about 77 % and 80 % for 20 and 100 mg/L, respectively. This result could be due to the high availability of free active sites at the beginning of adsorption process (Aziam et al., 2016, Abidar et al., 2015, Morghi et al., 2015, Ahmadou et al., 2023). This study revealed that the contact time is an important parameter for the adsorption of acid blue 113 on chitin. So, taking into account these results, the contact time was fixed to 90 min for the rest of the batch experiments.

3.4. Effect of initial concentration on dye removal

The effect of the initial concentration on the adsorption of AB113 dye was investigated at different concentrations ranging from 20 mg/L to 300 mg/L (Fig 6). Analysis of this curve shows that the retained uptake of AB113 increases with the initial dye concentration of the solution. The retained uptake continues to increase even for high initial concentrations (300 mg/L) without reaching the level corresponding to saturation.

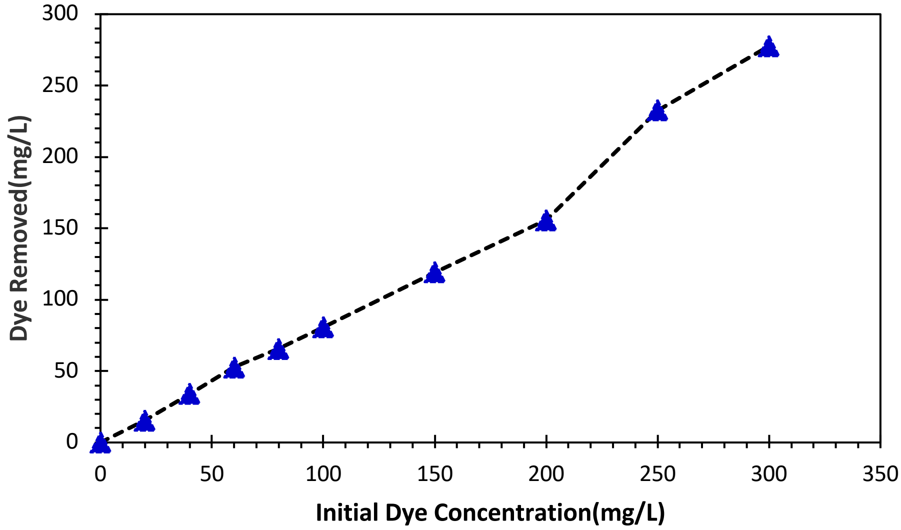


Fig 6. Effect of initial dye concentration on AB113 adsorption using chitin: R = 2.5 g/L; T=25°C ; pH= 6.3; Tc = 90 min.

At equilibrium (after 90 min of contact time) the adsorption capacity of chitin increased with the initial AB113 dye concentration in the solution which may be due to more availability of acid dye molecules and the active sites of the adsorbent have not reached saturation even at initial dye concentrations more than 300 mg/L. The higher amount of dye adsorption at higher concentrations is probably due to increased diffusion and decreased resistance to dye uptake (Boukarma et al., 2021). These results indicate a great potential application of chitin particles, as a bio-adsorbent to the treatment of industrial wastewaters.

3.5. Effect of initial pH on dye removal

The effect of solution pH on the adsorption of AB113 was carried out by prepare adsorbent-adsorbate solution with different pH then shaken together until equilibrium time. The pH of the dye solutions was adjusted over the range pH 2 to pH 12 using 0.1 M of HCl and NaOH solutions. The results presented in Fig 7 indicate that the maximum adsorption capacity of AB 113 considerably affected by the initial pH of solution. From these results, the AB113 uptake increased slightly from pH 2 to 6 and then after decreased with further increase in the pH up to 12. The optimum pH solution of the AB113

adsorption was found to be about 6, indicating that the adsorption of anionic AB113 dye onto chitin at pH value less than $pH_{ZPC} = 6.7$ is favorable. These results showed also that AB113 uptake is lower at higher pH values ($pH > pH_{ZPC}$). The effect of initial pH on the dye adsorption may be due to the chemical and electrostatic interactions between the adsorbent surface and the dye molecules (Aziam et al., 2016).

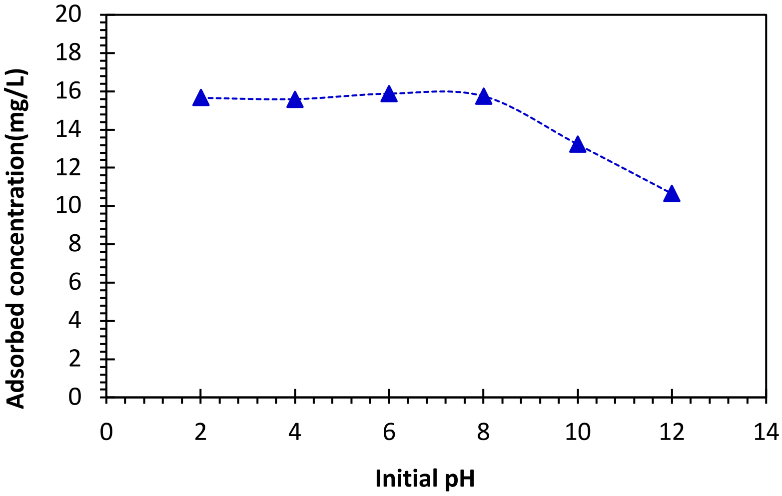


Fig.7. Effect of initial pH on adsorption of AB113: Ci= 20 mg/L, m/V=2.5 g/L, T=23±2°C, and t = 90 min.

3.6. Effect of temperature on dye removal

The temperature of the dye solution played an important role in the whole adsorption process and particularly on the adsorption capacity. The effect of temperature on the adsorption of AB113 dye by using chitin as adsorbent was studied in the temperature range from 25 to 40°C. The results are illustrated in Fig 8. It was observed the increase of adsorption capacity of AB113 with increasing temperature; this result indicates that the adsorption process nature is an endothermic.

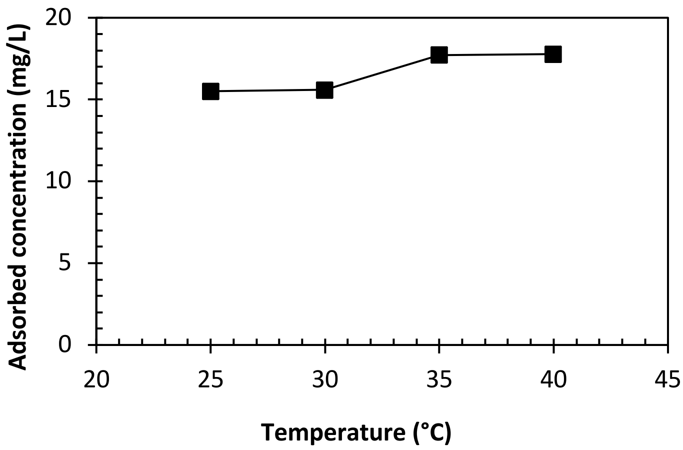


Fig. 8. Effect of temperature on AB113 adsorption by chitin: Ci= 20 mg/L, m/V=2.5 g/L, pH= 6.3, and tc= 90 min

3.7. Adsorption isotherm models

Adsorption isotherms are critical in optimizing the use of adsorbents, and the analysis of the isotherm data by fitting them to different isotherm models is an important step to find the suitable model that can be used for design purposes (Kalam *et al.,* 2021). In this study, the Langmuir, Freundlich, and Temkin equations were used to describe the relationship between the adsorbed AB113 dye uptake onto

dried Chitin and its equilibrium concentration in solution. This study was carried out by varying initial ion concentration from 20 to 300 mg/L at different temperatures and natural pH 6.3.

3.7.1. Langmuir adsorption isotherm

The Langmuir adsorption isotherm (Kalam *et al.,* 2021, Aziam *et al.,* 2017, Elsherif *et al.,* 2022, Nguyen *et al.,* 2021, Ahmadou *et al.,* 2023) assumes that solid surface has a finite number of identical sites which shows homogeneous surfaces. The Langmuir equation and its linearized form may be represented as:

$$Q_e = Q_L \frac{K_L C_e}{1 + K_L C_e} \qquad (\text{non} - \text{linear form}) \qquad (\text{Eq. 3})$$

$$\frac{1}{Q_e} = \frac{1}{Q_L} + \frac{1}{q_L K_L C_e} \qquad (\text{linear form}) \qquad (\text{Eq. 4})$$

Where Q_e (mg/g) is the amount of dye adsorbed per unit weight of the dried Chitin adsorbent at equilibrium, C_e (mg/L) is the equilibrium concentration of dye in the solution, Q_L (mg/g) is the Langmuir maximum adsorption capacity and K_L (L/mg) is the Langmuir constant related to free energy of adsorption. The isotherm constants q_L and K_L were calculated from the slope and intercept of plot between $1/Q_e$ and $1/C_e$, as shown in Fig. 9.
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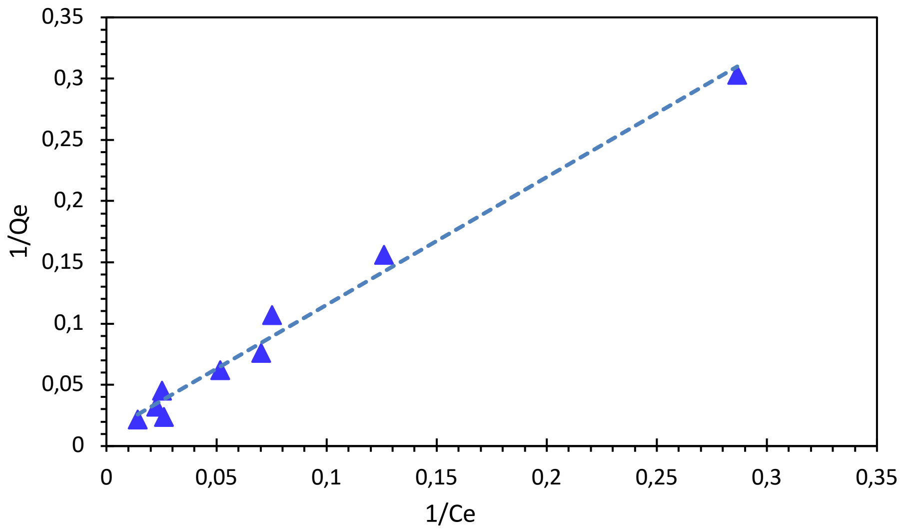

Fig. 9. Langmuir adsorption isotherm of AB113 onto Chitin (m=0.2 g, PH=6.3, T=25℃, t_C=90min)

3.7.2. Freundlich adsorption isotherm

The Freundlich equation shows best fittings to adsorption data for natural heterogeneous adsorbents. The Freundlich adsorption isotherm equation and its linear form can be written as follows (Kalam et al.,2021, Aziam et al., 2017, Elsherif et al., 2022, Nguyen et al.,2021, Ahmadou et al., 2023):

$$Q_e = K_F {C_e}^{1/n} \qquad (\text{non} - \text{linear form}) \qquad (\text{Eq. 5})$$

$$\ln Q_e = \ln K_F + \frac{1}{n} \ln C_e \qquad (\text{linear form}) \qquad (\text{Eq. 6}$$

where, Q_e (mg/g) is the amount of AB113 adsorbed per unit weight of adsorbent; C_e (mg/L) is the equilibrium concentration of solute in the bulk solution; K_F ($mg.g^{-1}$) is the Freundlich constant, which is a comparative measure of the adsorption capacity of the adsorbent, and n is an empirical constant

related to heterogeneity of the adsorbent surface. The parameter n also indicates the nature of the adsorption process. The value of n lies between 0 and 1 for a favorable adsorption, while $n > 1$ represents an unfavorable adsorption, and $n = 1$ represents the linear adsorption, while the adsorption operation is irreversible if $n = 0$. The isotherm constants n and K_F were calculated from the slope and intercept of the plot lnQ_e verses lnC_e (Fig. 10). The values for Freundlich constants and correlation coefficients (R^2) for both temperatures are also presented in Table 1.

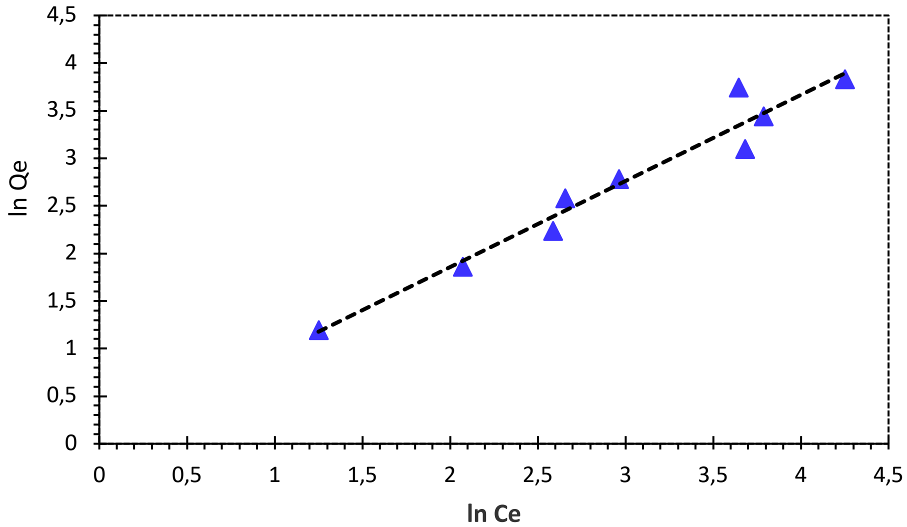

Fig 10. Freundlich adsorption isotherm of AB113 onto Chitin (m=0.2 g, pH=6.3, T=25℃, t_C=90min)

3.7.3. Temkin Isotherm

The Temkin adsorption isotherm model based on the heat of adsorption of the ions, which is due to the adsorbate and adsorbent interactions taken (Kalam et al.,2021, Aziam et al., 2017). The Temkin isotherm equation is given as:

$$\mathbf{Q_e = K_F C_e^{1/n}} \quad \textbf{(non-linear form)} \quad \textbf{(Eq. 7)}$$

$$\mathbf{\ln Q_e = \ln K_F + \frac{1}{n} \ln C_e} \quad \textbf{(linear form)} \quad \textbf{(Eq. 8)}$$

Where T is absolute temperature in Kelvin and R the universal gas constant (8.314 J.mol^{-1}K^{-1}). b_T (J/mol) is Temkin isotherm constant related to the heat of adsorption. K_T (l/mg) is the equilibrium binding constant corresponding to the maximum binding energy.

The Temkin isotherm plot for dye-dried Chitin system are presented in Fig.11 and the isotherm parameters are given in Table 1. Table 1 shows the values of the Langmuir, Freundlich and Temkin constants, extrapolated from the equations of these three models. By fitting the experimental points to the three models, and based on the values of the R^2 coefficient, it appears that the Langmuir model (Fig. 9) better expresses the type of adsorption (R^2= 0.986). Thus, the dye molecules could be adsorbed in monolayers, without any dye-dye interactions.

The numerical value of n in the Freundlich model is between 1 and 10 (i.e. 1/n less than 1), which indicates that the dye AB113 is favorably adsorbed by dried Chitin bio-adsorbent. The low values in this study indicate a weak interaction between adsorbate and adsorbent, supporting an ion-exchange mechanism for the present study. From linear regression of the data points, the R^2 value is rather low indicating that the adsorption of AB113 did not follow the Temkin isotherm closely. In general, Langmuir model had a good agreement with the experimental data for anionic acid blue 113 dye

adsorption. These experiments confirm the efficiency of the dried Chitin to remove acid dyes from aqueous solutions.

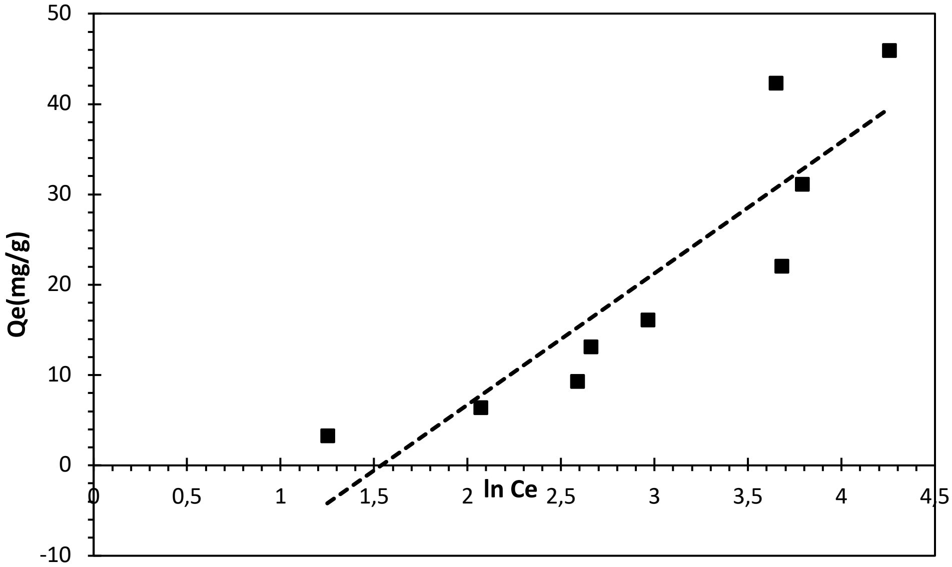

Fig 11. Temkin adsorption isotherm of AB113 onto Chitin (m=0.2 g, PH=6.3, T=25°C, t_C=90min)

Table 1. Parameters of three isotherms for AB113 dye adsorption onto dried Chitin (m=0.2 g, PH=4.3, T=25°C, t_C=90min)

Isotherms	***Langmuir***			***Freundlich***			***Temkin***		
Parameters	K_L $(L.mg^{-1})$	Q_{Lcal} $(mg.g^{-1})$	R^2	K_F $(mg.g^{-1})$	*n*	R^2	K_T $(L.mg^{-1})$	b_T $(J.mol^{-1})$	R^2
	0.010	90.90	0.986	1.047	1.105	0.953	0.214	170.35	0.799

Conclusion

This study investigated the effect of environmental parameters on the removal of the industrial dye from aqueous solutions using the batch equilibration technique. The adsorption was found to be strongly dependent on contact time, initial dye concentration and pH of solution. The pH_{ZPC} of chitin was found to be 6.7. The optimum pH solution of the AB113 adsorption was found to be about 6, indicating that the adsorption of anionic AB113 dye onto chitin at pH value less than pH_{ZPC} = 6.7 is favorable. These results showed also that AB113 uptake is lower at higher pH values (pH > pH_{ZPC}).
The Langmuir, Freundlish and Temkin, adsorption models were used for the mathematical description of the adsorption equilibrium of AB113 dye by Chitin. The experimental data fitted well to the Langmuir adsorption isotherm. The results of present investigation show that the chitin could be used as low cost and eco-friendly material for the removal of industrial dyes such as Acid Blue 113.

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Disclosure statement: *Conflict of Interest:* The authors declare that there are no conflicts of interest. *Compliance with Ethical Standards.*

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