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Adsorption kinetics, equilibrium and thermodynamics of a textile dye V5BN by a natural nanocomplex material: Clinoptilolite

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Highlights

- Efficient removal (~96%) of an industrial dye from simulated wastewater.
- Demonstrated the use of natural clinoptilolite in dye removal in 90 min.
- Adsorption was heterogenous, spontaneous and enthalpy driven.

Abstract

Dyes are considered as a major pollutant released in industrial (leather, textile, and paper) effluents. In this study, the ability of Clinoptilolite in adsorbing an industrial dye (Violet 5BN) was assessed. Clinoptilolite was characterized by Scanning Electron Microscopy, Energy Dispersive analysis using X-ray and Brunauer, Emmett and Teller analysis. Batch studies at varying adsorbent dosage, pH, temperature, and time revealed that 96% of the dye was adsorbed with an adsorbent mass of 1.5 g at 30 °C, pH 5 and reaction time of 90 min. Both Langmuir and Freundlich isotherms were found to be fit, which proves the process to be heterogeneous. The experimental and calculated values of adsorption capacity were almost similar, with correlation coefficients greater than 0.9, thus implying pseudo-second order and intraparticle diffusion as the favorable models. Negative values of ΔG° indicate strong binding energy between the adsorbent and adsorbate, while negative ΔS° values prove less randomness of the process and higher adsorbate concentration on the adsorbent surface due to ion-exchange interaction. The exothermic nature of adsorption is evident from the negative ΔH° recorded. Thermodynamic studies showed the system was a spontaneous and enthalpy driven process, with chemisorption as the predominant mode of adsorption at 30 °C and physisorption at elevated temperatures. The study demonstrates the significance of natural clinoptilolite in environmental protection, as an adsorbent for remediation of dyes.

Previous Next

Keywords

Clinoptilolite; Isotherms; Intraparticle diffusion; Remediation of dyes

1. Introduction

Dyes are compounds, primarily used to impart color. Synthetic dyes are developed at low costs, to provide a vast range of colors and are hence preferred over natural dyes for industrial applications. The major industries using dyes are textile, leather, paper & pulp, paint, printing, and cosmetic industries. Dyes are broadly classified as anionic, cationic or non-ionic [1] and the choice of dye depends on the color and its application. However, the dyes used in industries are considered as major pollutants since they are not fully utilized and the leftovers or spent dyes are removed through the sewage system as an effluent. High thermal and photo stability of dyes enable their persistence in the environment for longer periods of time, if left untreated [2]. This causes health ailments to all forms of life [3] and

hinders the sustainability of aquatic life. The effluent from textile industries is particularly toxic due to the presence of naphthol, vat dyes, nitrates, acetic acid, chromium-based compounds, heavy metals, and other dyeing auxiliaries. The presence of these compounds in the effluent causes turbidity that could prevent photosynthesis, eventually affecting marine life, in addition to clogging the soil pores [4]. Consequently, exclusion or reduction of dyesfrom wastewater becomes mandatory, prior to discharge into the environment.Physicochemical methods such as coagulation [5], ion-exchange [6], ozonation [7], Fenton's oxidation [8], photo catalytic degradation by irradiation [9], UV/H_2O_2 treatment [10] and the use of membranes [11] are majorly employed for the removal of dyes from synthetic wastewater and industrial effluents. Biological methods for dye removal include the use of microbial fuel cells [12], bacteria [13], fungi [14], algae [15] and enzymes [16]. However, most of these methods generate secondary pollutants to the environment.

Adsorption is a simple physical process which provides high efficiency at low costs. Adsorbents such as chitosan [4], clay [17], cotton [18], coconut coir [19], activated carbon [20,21], nano composites [22], wheat straw [23], sugarcane baggase [24], plant waste [25] and nanohydrogels [26] have been studied extensively for the removal of dyes. A natural form of zeolites, i.e., Clinoptilolites ($Na_6[(AlO_2)_6(SiO_2)_{30}].24H_2O$ or $(Na_2K_2CaMg)_3[(AlO_2)_6(SiO_2)_{30}].24H_2O$) are porous, hydrated alumina silicate materials, with three-dimensional crystal structures, occurring in volcanic ashes. They have resistance to extreme temperatures, high absorption level, ion exchange capacity, catalytic activity, and dehydration efficiency. Owing to such properties, clinoptilolite is used as an electrode [27], in drug delivery [28], aquaculture, animal feed [29], as a catalyst [30], chemical sieve, food additive, absorbent of gasses and in agriculture [31]. In addition, clinoptilolite has been studied for its potency in adsorption of ions [32], heavy metals [33], phenols [34], phenolic compounds [35], ammonia [36], dyes [37], salinity [38], pharmaceutical waste [39], chemical and biological oxygen demand [40]. Due to the abovementioned characteristics of clinoptilolite, it was chosen as the adsorbent in this study.

The ability of natural clinoptilolite in adsorbing an industrial dye (Violet 5BN) was assessed through characterization of the clinoptilolite, followed by determining the adsorption kinetics, isotherms and thermodynamic parameters under varying parameters of adsorbent dosage, pH, temperature, and time.

2. Materials and methods

2.1. Characterization of adsorbent

Clinoptilolite was obtained from the Tree of Life, Chennai, India. The morphology and elemental composition of clinoptilolite were studied using Scanning Electron Microscopy (SEM) and Energy Dispersive analysis using X-ray (EDX). The surface area, total pore volume, total pore area and size distribution of the natural zeolite were determined by Brunauer, Emmett and Teller (BET) analysis.

2.2. Adsorbate

An industrial dye, Violet 5BN (V5BN) (CAS Registry No.: 52080-58-7/67989-22-4) was procured from a chemical supplier, Thirukumaran agencies, Chromepet, Chennai, India. The water-soluble dye, with molecular formula $C_{24}H_{27}N_3$, belongs to triarylmethane class of dyes. The auxochromes are substituted amine groups, i.e., methylated amine groups. The structure of the dye is presented in Fig. 1.

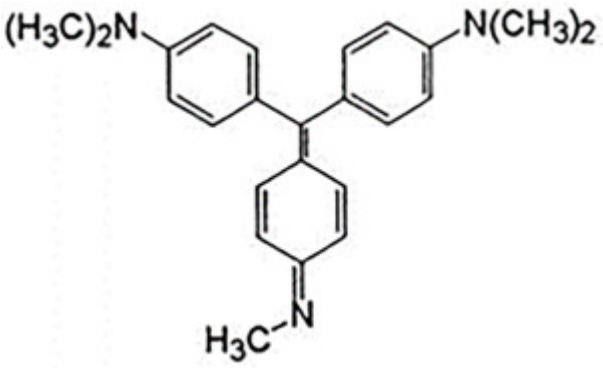

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Fig. 1. Structure of Violet 5BN.

2.3. Sorption studies

Adsorption of V5BN onto clinoptilolite was studied at varying parameters of adsorbent dosage, pH, temperature, and time. Briefly, synthetic solutions (100 mL) containing 50 ppm of the dye at varying pH of 5, 7 and 9 were subjected to batch experiments, at different concentrations of clinoptilolite (0.5%, 1% and 1.5%) and temperatures (30 °C, 37 °C and 45 °C). The batch adsorption study was maintained at 150 rpm in a temperature-controlled shaker. The removal efficiency was evaluated at varying time intervals of 30, 45, 60, 75 and 90 min, by measuring the optical density at 545 nm in UV–Vis spectrophotometer. The percent of dye removed was calculated from the formula:

$$\%\ Removal = \frac{(A_c - A_s)}{A_c} X100$$

where A_c: Absorbance of the control; A_s: Absorbance of the sample

2.4. Adsorption isotherms

The adsorption isotherms using Langmuir and Freundlich models were studied according to [28]. Langmuir adsorption isotherm is expressed by the linear equation:

$$\frac{C_e}{q_e} = \frac{1}{q_m} + \frac{1}{q_m K_L}$$

where C_e is the equilibrium concentration (mg/L), q_e is the amount of dye adsorbed at equilibrium (mg/g), q_m is a constant related to adsorption capacity and K_L is the constant related to the affinity of the binding sites and the energy of adsorption (mg/g). The dimensionless constant R_L depicts the favorability of the adsorption process and is expressed as

$$R_L = \frac{1}{1 + K_L C}$$

where K_L is Langmuir constant and C is the initial concentration of the dye (mg/L).

The equilibrium adsorption according to the Freundlich model is expressed in logarithmic form as

$$\ln q_e = \ln K_f + \frac{1}{n} \ln C_e$$

where q_e is the amount of dye adsorbed at equilibrium time (mg/g), C_e is the equilibrium concentration of the dye solution (mg/L), K_f (L/mg) and n are Freundlich isotherm constants indicating the capacity and intensity or strength of the adsorption process, respectively.

2.5. Adsorption kinetics

Pseudo-second order and intraparticle diffusion models were used to study the kinetics of the adsorption of V5BN by clinoptilolite. The pseudo second order kinetics is given by the equation:

$$\frac{t}{Q_t} = \frac{1}{k Q_e^2} + \frac{t}{Q_e}$$

where Q_e and Q_t represents the quantity of the dye adsorbed per unit mass of the adsorbent at equilibrium state and time t respectively [41].

The intra-particle diffusion model is given by

$$q_t = K_{di} t^{1/2} + C_i$$

where q_t (mg/g) is the capacity of adsorption at any time t, K_{di} (mg/g $min^{1/2}$) indicates the intraparticle diffusion rate constant and C_i (mg/g) represents the thickness of the boundary layer [42].

2.6. Adsorption thermodynamics

The thermodynamic parameters such as change in Gibbs free energy (ΔG^0), enthalpy (ΔH^0) and entropy (ΔS^0), during the adsorption process were studied according to [43,44]. The relation between these parameters is given by the equation

$$\Delta G^0 = -RT\, lnK_L$$

$$lnK_L = \frac{\Delta S^0}{R} - \frac{\Delta H^0}{RT}$$

where K_L is the Langmuir constant, R is the universal gas constant (8.314J/mol K) and T is the temperature (K).

To further understand the mechanism of sorption, i.e., physisorption or chemisorption, the sticking probability (S^*) was calculated [45].

$$S^* = (1-\theta)\, e^{-E_a/RT}$$

$$\theta = [1 - C_e/C_o]$$

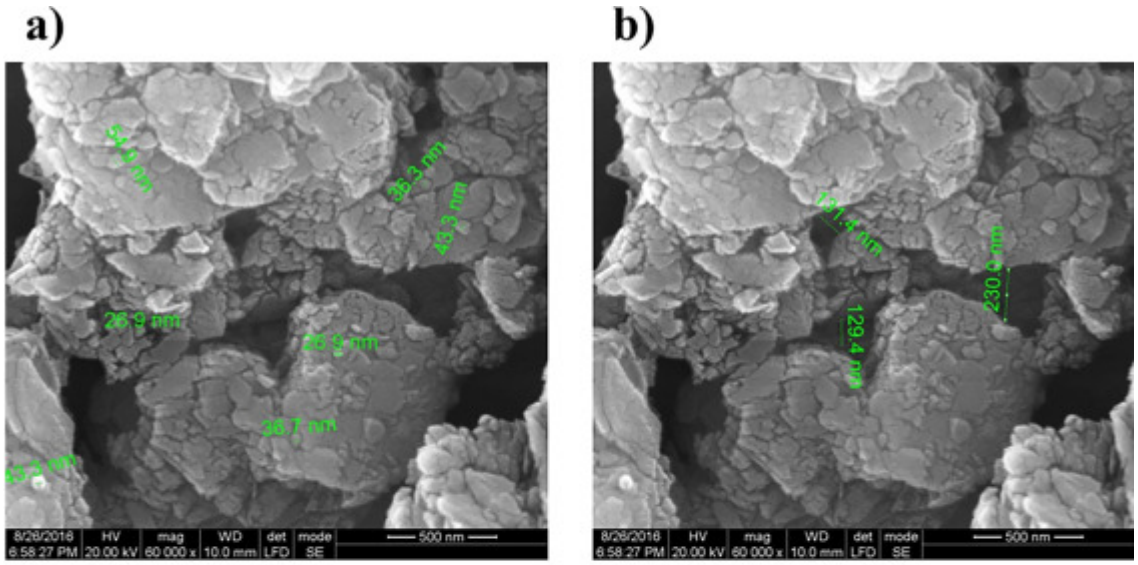
where S^* is a function of the adsorbate-adsorbent system; E_a is the activation energy; θ denotes the surface coverage; R is the gas constant and T is the temperature (K).

3. Results and discussion

3.1. Characterization of clinoptilolite

The surface morphology of clinoptilolite studied through SEM revealed the particle size as small as 13nm (Fig.2a). Numerous pores were observed with a size range of 125nm to 410nm (Fig.2b). EDX analysis confirmed the presence of magnesium, aluminum, silicon, potassium, calcium, and iron, in addition to carbon and oxygen (Fig.2c-d). The presence of SiO_2, Al_2O_3, K_2O, Na_2O, CaO and trace amounts of MgO were previously reported in natural

clinoptilolite [46]. Thus, SEM-EDX analysis confirmed the porous nature of clinoptilolite with aluminosilicate composition.



Element	Weight (%)	At (%)
CK	20.68	32.32
OK	32.34	37.95
MgK	00.60	00.46
AlK	07.65	05.33
SiK	30.42	20.34
KK	02.87	01.38
CaK	02.97	01.39
FeK	02.47	00.83

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Fig. 2. a-b) SEM image and c-d) SEM-EDX analysis of clinoptilolite.

The surface area observed by BET was 80.30 m²/g. The pore volume as observed by the t-Plot was 0.036 cm³/g and the average pore width was recorded to be 49.76Å. From the N_2 adsorption isotherm (Fig.3), a hysteresis curve was observed between relative pressures of 0.4 to 1. According to the IUPAC classification of isotherm curves, clinoptilolite is observed to follow type IVa. This type of adsorption is known to occur due to extension of pore width beyond its critical width and is dependent on the adsorption system and temperature. The hysteresis loop falls under type H4, among the 5 types. In H4 loops, high uptake at low relative pressure is associated with the filling of the pores of the material. Further, such

types of isotherm curves denote monolayer-multilayer adsorption on mesoporous materials such as zeolite, carbon, oxide gels, etc. [47].

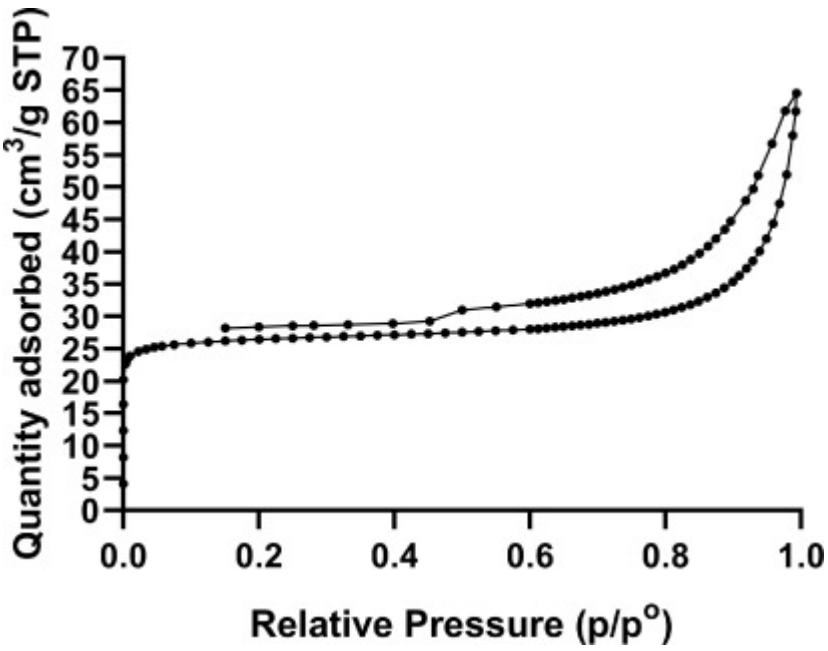

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Fig. 3. N_2 adsorption isotherm of clinoptilolite.

3.2. Sorption studies

Adsorption of a molecule or ions occurs due to interaction of dipole or charged species of the adsorbate and the adsorbent [48]. Zeolites are considered as suitable adsorbents due to the possession of ions such as Al^{+3} and Si^{+4}, which enable ion exchange with the adsorbate [49]. In this study, the adsorption of V5BN by clinoptilolite varied at different parameters, in the range of 52.79 to 96.96%. The percent removal rapidly increased with time, with the maximum at 90 min, after which there was no further difference. Hence, 90 min was considered as the time at which equilibrium was attained. The rapid increase in adsorption was followed by a slow, gradual increase. This is possibly due to the availability of a large number of free surface sites for adsorption as soon as the adsorbent interacts with the adsorbate [43]. Following this initial interaction, the dye molecules in the solute were either repelled or further adsorbed, which varied on other parameters.

The maximum percent of the dye adsorption was 96.96% at 30 °C, 96.57% at 37 °C and 91.06% at 45 °C (Fig.4). At the initial stages of adsorption, there was not much difference between the batch reactions maintained at 30 °C and 37 °C. With increase in temperature to 45 °C, there was a drastic reduction in adsorption of dye molecules. In general, higher

temperatures are known to cause swelling in the internal structure of zeolite [50], which could have caused lower adsorption rate initially. With increase in time of exposure at 45 °C, the mobility of the dye molecules increased, enabling interaction with the active sites of clinoptilolite and enhancing the adsorption efficiency to 91.06% by 90 min.

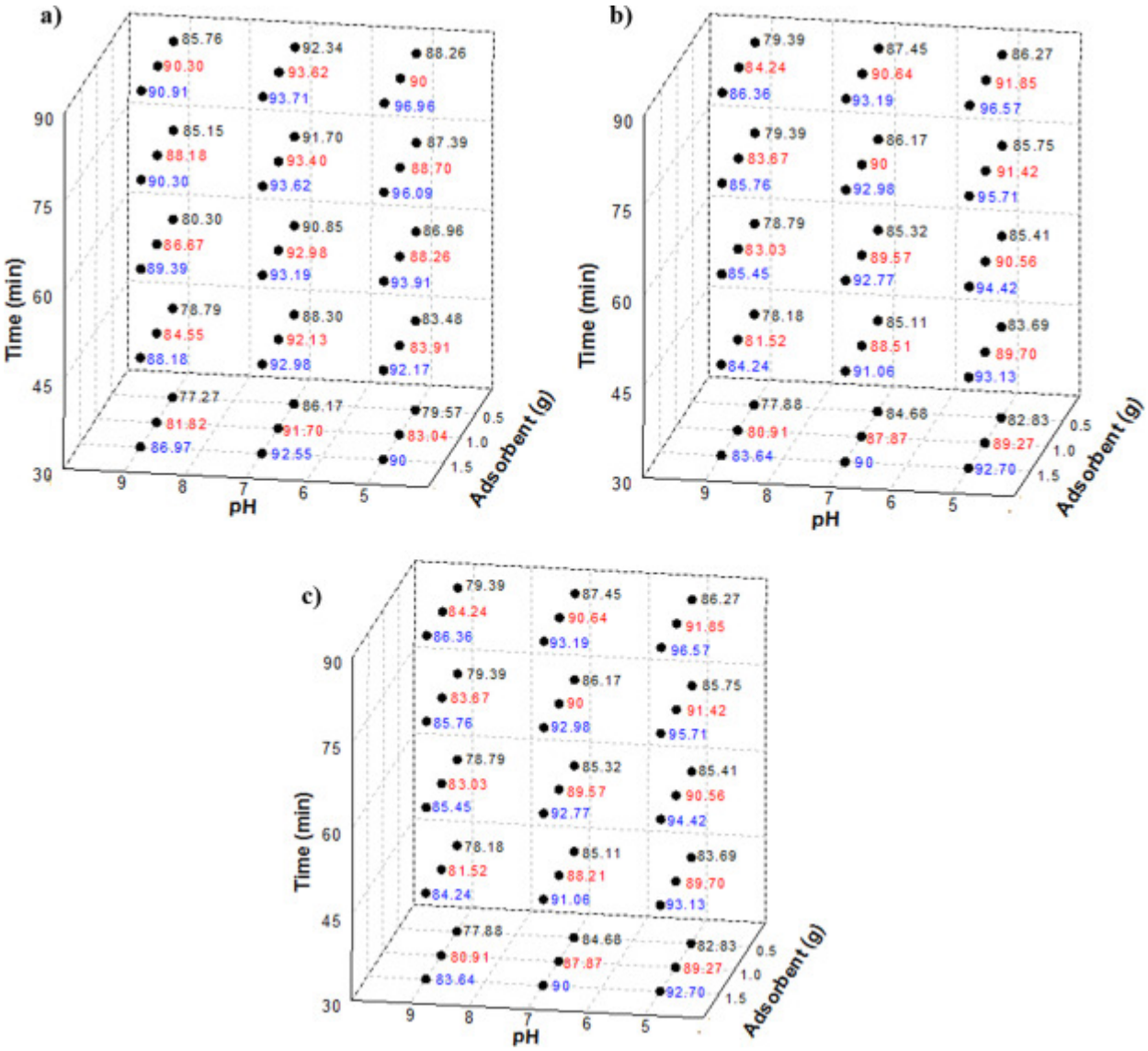

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Fig. 4. Percent removal of V5BN by adsorption using clinoptilolite at a) 30 °C b) 37 °C c) 45 °C.

Ionic strength is known to affect the charge distribution and surface binding site of the sorbent [51]. In the present study, the highest removal of V5BN was recorded at pH of 5 (96.96%). At this pH, the minimum percent removal was 52%. At pH 7, the range of percent removal was 62.98 to 93.71, while it was 73.64 to 90.91% at pH 9. By increasing the pH, there was a 3% decrease in the maximum percent removal. Basic dyes release intense

molecular cation and reduced ions, on dissolution. Since clinoptilolite possesses negative charge, electrostatic forces favor the adsorption of cationic dyes [52].

Previously, a maximum of 94% removal of methylene blue dye was attained by photocatalysis of synthetic zeolite doped with a semiconductor [53]. However, photocatalysis of dyes requires higher reaction time and incurs cost due to synthesis/incorporation of catalysts [54,55]. In the present study, the dosage of the adsorbent, i.e., clinoptilolite directly correlated with the percent removal, primarily owing to high surface area and large number of sites that are available for adsorption at higher concentrations of the adsorbent [56]. Moreover, natural clinoptilolite had the ability to remove about 96% of the dye V5BN at temperature close to ambient (30°C), which is easy to achieve when scaled up for industrial use, thereby reducing the expenses incurred as well.

3.3. Adsorption isotherms

Adsorption isotherms indicate the distribution of adsorbate between the liquid and solid phases at the equilibrium, in addition to suggesting the suitable model for designing of an adsorption process. The adsorption capacity of clinoptilolite declined with increasing temperature, with the maximum values being 7.96, 7.41 and 5.73 mg/g at 30, 37 and 45 °C respectively, in Langmuir isotherm. The experimental and calculated adsorption capacity were almost close, with correlation coefficients >0.98. The K_L values increased with increase in the adsorbent dose, at all temperatures and pH studied. This proves high affinity of the dye molecules with the adsorbent. The separation factor, R_L was between 0 and 1, thus indicating the favorability of the adsorption process.

Similar to Langmuir isotherm, the Freundlich isotherm was also found to be favorable. This is evident from the values of 1/n, wherein values between 0 and 1 are known to exhibit favorable process. This implies the heterogeneity of the adsorbent sites at all the temperatures studied. With increase in the adsorbent dosage, the values of n increased. Generally, in adsorption processes with $n>1$, the adsorption occurs on the high energy sites initially, followed by low energy sites [57]. In the present study, both the models - Langmuir and Freundlich, were found to be appropriate, thus suggesting the adsorption process to be heterogeneous. The values obtained from Langmuir and Freundlich isotherms are presented in Table S1.

3.4. Adsorption kinetics

Kinetics relates the rate of removal of adsorbate from solution and provides an insight on the mechanism of adsorption. The adsorption of V5BN using clinoptilolite was investigated

through pseudo-first order, pseudo-second order and intraparticle diffusion models. Pseudo-first order model did not fit with the data. The correlation coefficients of pseudo-second order model was greater than 0.97 and the experimental values of q_e were almost similar to the calculated values. This implies that the adsorption process followed pseudo-second order model [58]. The increase in k value indicates that the rate of adsorption increased with increase in the dosage of clinoptilolite, due to the large number of active sites. Fig.5a depicts the pseudo second order model at the experimental conditions of 30°C and pH 5.

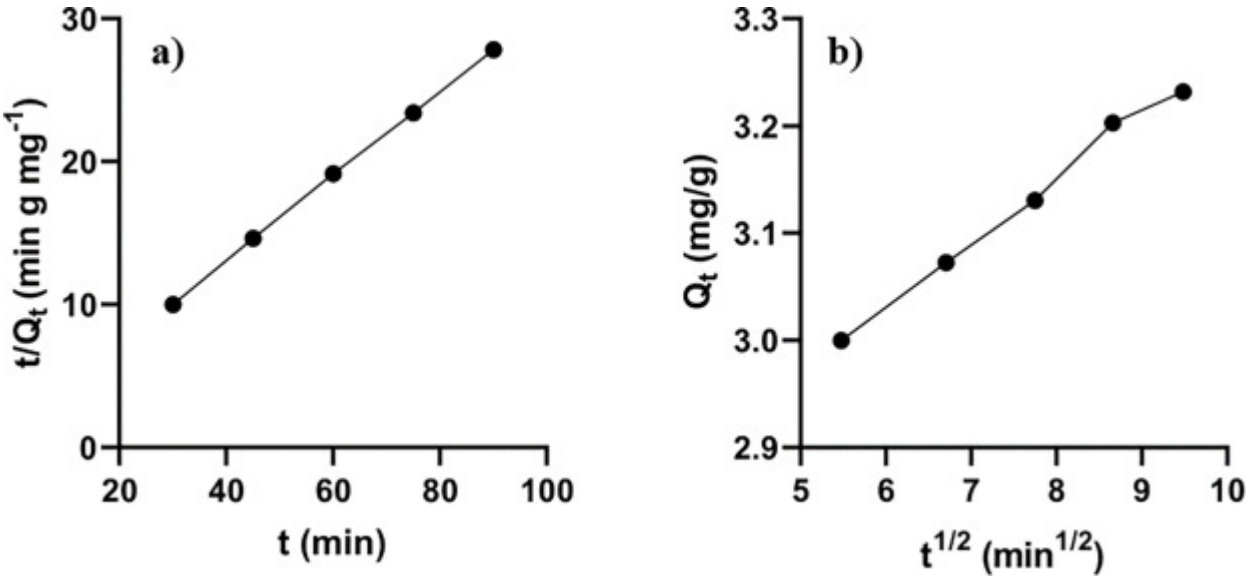

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Fig. 5. a) Pseudo second order model b) Intraparticle diffusion model at the experimental conditions: 1.5g adsorbent; 30°C and pH 5.

The transport of the adsorbate onto an adsorbent is governed by film diffusion, pore diffusion, surface diffusion and adsorption on pore surface, or a combination of them, which is evidenced through intraparticle diffusion model [59]. As seen in Fig.5b, there is a linear increase in adsorption. Under differing experimental parameters, linear phase of adsorption capacity was observed, probably attributed to external diffusion, which gradually reached the equilibrium as it approached the maximum adsorption. This is similar to the adsorption of dyes by modified zeolite [60]. The intraparticle diffusion rate constant K_{di} was found to decrease with increase in the adsorbent dosage. The thickness of the boundary layer decreased in a similar way. In general, the greater the value of C, greater is the boundary effect [61]. The decrease in the C values could be due to the large number of active sites on the adsorbent, to which the dye molecules were bound, thus reducing the boundary effect.

The kinetic constants from pseudo-second order and intraparticle diffusion models are presented in Table S2.

3.5. Thermodynamic studies

In an adsorption process, the thermodynamic parameters that have a significant role are standard free energy (ΔG°), enthalpy (ΔH°) and entropy (ΔS°) and are presented in Table S3. In the present study, negative values of ΔG° were recorded, indicating the spontaneous nature of the adsorption process and external energy is not required [45]. The high temperature was not favorable for the adsorption process since the values of ΔG° decreased with rise in temperature. This correlates with the reduction in percent removal at higher temperatures. In addition, the negative values of ΔG° indicate stronger binding energy between the adsorbent and adsorbate, than between the adsorbate and solvent [62]. Negative ΔS° values prove less randomness of the adsorption process and indicate the increase of adsorbate concentration on the adsorbent surface, which is known to occur due to ion-exchange interaction [63].

Enthalpy values in the range of −2.1 to −20.9 kJ/mol represent physical adsorption, while chemical adsorption is represented with the magnitude −80 to −200 kJ/mol [64]. [65] reported that natural zeolite interacts with dye molecules through hydrogen bonding and van der waals forces. In adsorption of V5BN, ΔH° ranging from −39 to −123 kJ/mol was recorded, which represents the involvement of both physical and chemical interactions. Further, the negative values of ΔH° depict the exothermic nature of the process.

The values of the sticking probability in the adsorption of V5BN by clinoptilolite were between 0 and 1, for all the experimental data. S^* is a temperature dependent function of the system, which denotes the entry of adsorbate into the intraparticle space after encountering the adsorbent surface [66]. The activation energy was in the range, 8.08 to 76.13 kJ/mol (Table S4). In general, values of E_a in the range 5–40 kJ/mol signify physisorption, while 40–800 kJ/mol indicate chemisorption [67]. Interestingly, the magnitude of activation energy was higher at 30 °C, confirming chemisorption to be the predominant mechanism. With increase in the temperature of the system, the values of E_a decreased. Hence, at higher temperatures, the process was physisorption.

4. Conclusion

The adsorption of an industrial dye, V5BN, by an aluminosilicate material, i.e., clinoptilolite was proven in the present study. The effect of different parameters showed that up to 96% of the dye was adsorbed by clinoptilolite at adsorbent dose of 1.5 g, temperature of 30 °C, pH

5 and reaction time of 90 min. Langmuir and Freundlich isotherms described the process to be heterogeneous. The adsorption followed pseudo second order and intraparticle diffusion models. In addition, the negative values of ΔG°, ΔS° and ΔH° indicate the system to be a spontaneous, less random and enthalpy driven process, with chemisorption as the predominant mode of adsorption process at 30 °C and physisorption at elevated temperatures. The study presents the promising ability of natural clinoptilolite for the adsorption of industrial dyes. Future studies on adsorption of dyes and other pollutants in wastewater by clinoptilolite, reusability and industrial scalability of clinoptilolite are warranted.

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Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Appendix. Supplementary materials

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Special issue articles Recommended articles

Data availability

The data has been detailed in the manuscript and supplementary material

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