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Kinetics, Isotherm and Thermodynamic studies on the Adsorption of Anionic Dye Using SnO_2 Nanoparticles

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Abstract

Tin (II) Oxide was synthesized and used for the removal of Alkali Blue (AB). The effect of variables such as contact time, dosage, concentration, pH and temperature for the adsorption process were investigated using Batch Adsorption Method. The synthesized nanoparticles were characterized using XRD and average crystallite size of 7.27 nm was obtained. FTIR were carried out to determine the functional groups present at the surface of the particle with functional groups such as –OH, C=C, C-H, C≡C detected. SEM analysis of the SnO_2 was carried out. The changes in the morphology observed is due to the attachment of the dye molecules at the surface of the nanoparticles. The percentage removal and optimum contact time of AB was obtained as 77.4% at 20 min. Kinetics studies shows that adsorption process fits better to pseudo second order due to its R^2 value being closer to unity and the experimental value of qe 3.9193 being closer to the calculated value 3.8715. Freundlich Isotherm indicating multilayer adsorption shows a better fit in the Isotherm studies. Lower value of Mean square energy (E) 0.500 kJ/mol for the adsorption being < 8 kJ/mol indicate a successful physical adsorption. Negative values of ΔH, ΔG and ΔS revealed that the process was exothermic, feasible and there is decrease in randomness. Value of ΔH - 10.173 kJ/mol confirmed the adsorption process as physical in nature. The results indicated that Tin (II) Oxide nanoparticles can be used as a low cost adsorbent for the removal of dyes such as AB from aqueous solutions.

Keywords: Adsorption, SnO_2, Kinetic, Thermodynamics, Isotherm.

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1. Introduction

In developing countries, industrial wastewater management is one of the major issues under discussion. Untreated textile effluents are discharged in the drain which deteriorate the water bodies (Khan and Malik, 2014). Almost 10 to 25% of the textile dyes are lost during the processing as effluents. Wastewater effluents are mostly organic in nature with dissolved oils and coloring agents. Moreover, fabric soaked with grease and oil adds to solid wastes that are hazardous and toxic to environment

(Sivaram *et al.,* 2019; El Hammari *et al.,* 2022). The release of toxic wastewater from industries is becoming a constant threat to the well-being of natural water resources and populations. Because of poor wastewater management policies of developing countries, 20 to 40% waterborne diseases are caused by industrial wastewater (Azizullah *et al.,* 2011; Halder and Islam, 2015). Seventy thousand tons of dyestuff is manufactured on annual basis, and the majority of these synthetic dyes are water soluble. These dyes have very complex structures, which make them less biodegradable that is why effective treatment methods are of interest currently (Shi *et al.,* 2007; Kumar and Saravanan, 2017; Beulah and Muthukumaran, 2020). A wide range of conventional methods have been investigated for dye removal extensively, but they have their own limitations, and these are not profitable at economical level. Principally, adsorption can be employed at large scale for the purification of wastewater at relatively large flow rates. Moreover, this method produces high-quality effluent that does not form harmful substances such as free radicals and ozone which are produced in photodegradation process via Ultraviolet (UV) radiation. The process of adsorption is fast, universal, and inexpensive. Various nano conventional sorbents are employed for removing chemical pollutants like chitin, peat, sulfonated coal, wheat straw, organomon t-morillonite, apple pomace, fly ash, dyestuff, and coir pith (Gupta *et al.,* 2005). There are specific interactions that occurs between the adsorbed contaminants and adsorbent materials. These interactions may be due to the simple mass transfer from liquid to solid phase (G *et al.,* 2005; Akartasse *et al.,* 2022; Zaaboul *et al.,* 2024; Latifi *et al.,* 2025). Three components are used in these complicated interactions i.e., adsorbate, adsorbent, and wastewater. Wastewater comprises synthetic solution and effluents.

In this investigational study, SnO_2 nanoparticles was employed to adsorb Alkali Blue dye. The dye removal process was used to optimize contact time, initial concentration of dye, dosage, pH and effect of temperature. Kinetics, thermodynamics, isotherms, regeneration, and reusability of the adsorbent material was evaluated for optimum uptake of the dye from the aqueous medium.

2.0 Materials and methods

2.1 Synthesis of Tin Oxide Nanoparticle

Tin oxide (SnO_2) nanoparticles were synthesized by co-precipitation method using precursor's stannous chloride and methanol. 3.5 g of stannous chloride was dissolved in 100ml of methanol and kept under magnetic stirring for 30 minutes. 10ml of Ammonia (NH_3) was rapidly injected into the container and was continuously stirred for additional 30 minutes. Following rapid injection, the reaction system color gradually changes slowly from transparent to white. The precipitate was filtered, washed with methanol and dried at 60 ºC for 4 hours by using hot air oven. The prepared nanoparticles were annealed at 200 ºC for 2 hours in hot air oven for characterization (Balakrishnan and Murugesan, 2021).

2.2 Preparation of the Dye Stock Solution and Working Solutions

1.00 g of Alkali blue (AB) dye was accurately weighed using analytical weighing balance and dissolved in 1.0 L of distilled water to prepare 1000 mg/L of the aqueous dye solutions (stock solution). The working solutions, 10, 20, 30, 40, 50, 100, 150, 200 and 250 mg/L and the standard calibration of solutions of the dye were all prepared by serial dilution. Hydrochloric Acid (HCl) and Sodium Hydroxide (NaOH) were used to adjust the pH of AB dye solution during the experiments.

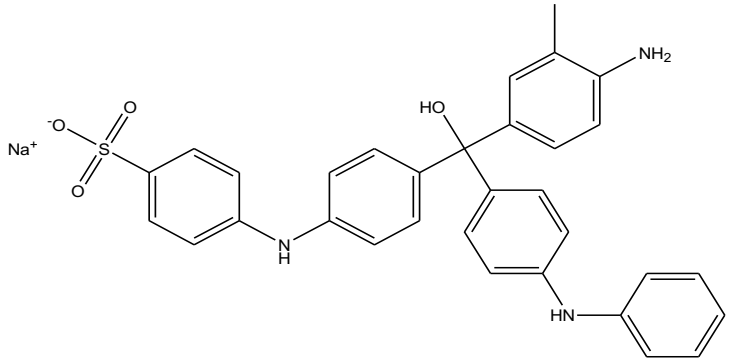

Figure 1: Structure of Alkali Blue Dye

2.3 Batch Adsorption Experiment

The influence of variables including, amount of adsorbent, contact time, temperature, pH and Initial concentration on the removal of AB was investigated in batch mode at room temperature (30 ± 3 °C). 100 cm^3 of dyes in a 120 cm^3 bottle was agitated at 200 rpm along with a fixed mass of the nanoparticles. The final concentration of the dye was determined spectrophotometrically using UV-Visible spectrophotometer (Hitachi 2800) at a predetermined corresponding λ_{max} 582.9 nm.

The percent removal of dye was calculated using equation (1):

$$\%R = \frac{C_O - C_t}{C_O} X\ 100 \qquad (1)$$

Co and Ct (mg/L) are the initial dye concentration and the dye concentration at any time, respectively.

2.4 FT-IR Analysis of Tin Oxide Nanoparticle Before and After Adsorption

FT-IR measurements of the SnO_2 nanoparticles was carried out to identify the possible functional groups that may be responsible for the adsorption of Alkali Blue (AB) dye using FTIR Cary 630 from Agilent technologies and the spectra was recorded in the wavelength interval 4000 to 600 cm^{-1}.

2.5 Scanning Electron Microscopy (SEM)

Scanning electron microscopy (SEM) gives the surface morphology of SnO_2 nanoparticle at a desired position and therefore displayed topographic/morphological picture with better resolution and depth of focus Eddy *et al.,* (2014).

2.6 Brunauer-Emmett-Teller (BET) Analysis

Brunauer-Emmett-Teller (BET) theory aims to explain the physical adsorption of gas molecules on a solid surface and serves as the basis for an important analytical method for the measurement of the specific surface area of the prepared SnO_2 nanoparticle (Marwani *et al.,* 2014).

2.7 X-Ray Diffraction Analysis

X-ray diffraction measurement was carried out to determine the crystal phase composition and size of the nanocomposite. The average crystallite size (D) was calculated from XRD pattern according to the Scherer equation:

$$D = \frac{K\lambda}{\beta COS\phi} \quad (3)$$

Where, k is constant (about 0.9), λ is the wavelength (0.15405 nm), β is the full width at half maximum (FWHM) of the diffraction line and θ is the diffraction angle (Habib *et al.,* 2018)

2.8 Desorption and Reusability

Desorption studies was carried out to regenerate the used adsorbent using Acid (HCl), Base (NaOH), Salt (NaCl) and Distilled Water. Reusability of the adsorbent was tested for up to five cycles.

3.0 Results and Discussion

3.1 Characterisation

The synthesized nanoparticle was characterized using Fourier Transform Infrared (FTIR) Spectroscopy, Brunauer-Emmett-Teller (BET) Analysis, X-Ray Diffraction Analysis, Scanning Electron Microscopy (SEM).

Fourier Transform Infrared (FTIR) Spectroscopy

FTIR Spectroscopy is often employed in the detection of different functional groups on the surface of the synthesized nanomaterials. Functional groups determination is very important in understanding of adsorption phenomena. **Figure 2** present the FTIR Spectrum of SnO_2, before and after adsorption of AB dye. The absorption peak at 3508cm^{-1} represents the presence of OH stretching vibrations, the C – H stretching was observed at 2883cm^{-1} (Ma *et al*., 2017). C≡C stretching vibrations was observed at 2108 and 2136 cm^{-1} and the absorption band observed at 1985 cm^{-1} reflects the skeletal stretching of allene group. The peaks around 600 – 700 cm^{-1} can be attributed to the Sn – O band (Lv *et al.,* 2006).

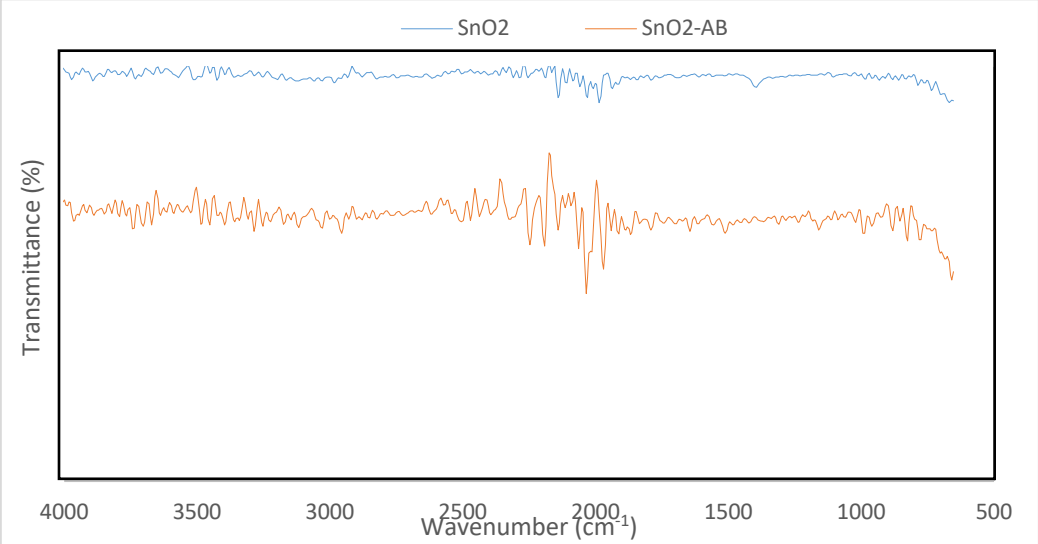

Figure 2: FTIR Spectra of SnO_2 Nanoparticle before and after Adsorption of AB

Brunauer-Emmett-Teller (BET) Analysis

Brunauer-Emmett-Teller (BET) theory aims to explain the physical adsorption of gas molecules on a solid surface and serves as the basis for an important analysis method for the measurement of the specific surface area of prepared nanomaterials (Marwani *et al.,* 2014). The surface area, pore volume, micro-pore surface area and pore diameter of SnO_2-Np are presented in Table 1. SnO_2-Np was found to have 265.625 m^2/g surface area, 0.130 cc/g pore volume, 249.093 m^2/g micro-pore volume and 2.132 nm pore diameter respectively.

Table 1: Brunauer-Emmett-Teller (BET) Analysis

Adsorbent	Surface Area (m^2/g)	Pore Volume (cc/g)	Micropore Surface Area (m^2/g)	Pore Diameter (nm)
SnO_2	265.625	0.130	249.093	2.132

X-Ray Diffraction Analysis

Figure 3 represents the XRD patterns of SnO_2-Np. Crystallinity and crystal phases of the synthesized materials were investigated. In the XRD pattern, many peaks are found at the 2θ angles of 16.61^0, 27.9^0, 37.81^0 which corresponds with Braggs planes of (110), (101), and (200). The average crystallite size was calculated to be 7.27 nm using Debye Scherer Equation.

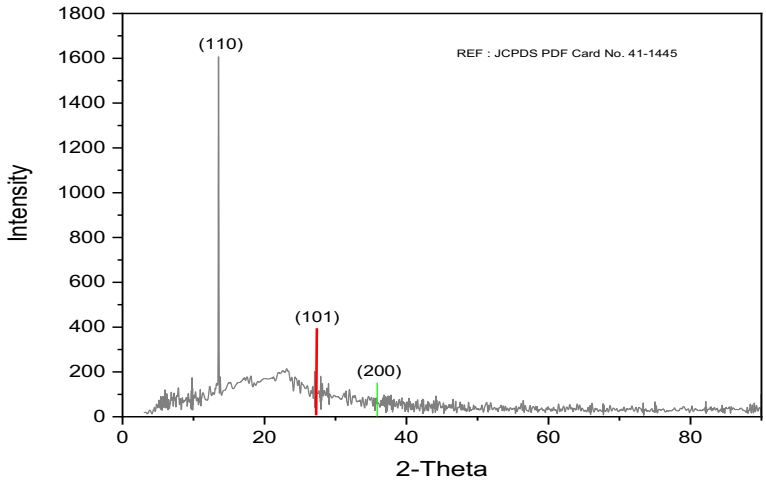

Figure 3: XRD Patterns of SnO_2-Np

Scanning Electron Microscopy (SEM)

Scanning Electron Microscopy (SEM) involves examining the surface morphology of the adsorbent material before and after adsorption process. It allows us to determine changes in surface morphology, such as the deposition of dye molecules, roughness at the surface which can provide insights into the adsorption mechanism and efficiency of the adsorbent material (Liu *et al.,* 2021). SEM micrograph of SnO_2 nanomaterial show that the nanoparticles are granular in size and have irregular spherical morphology as shown in Figure 4. Appreciable changes were observed in the micrographs after the adsorbent have been loaded (adsorption). The changes are indication that there is interaction between the dye molecule and the adsorbent surface.

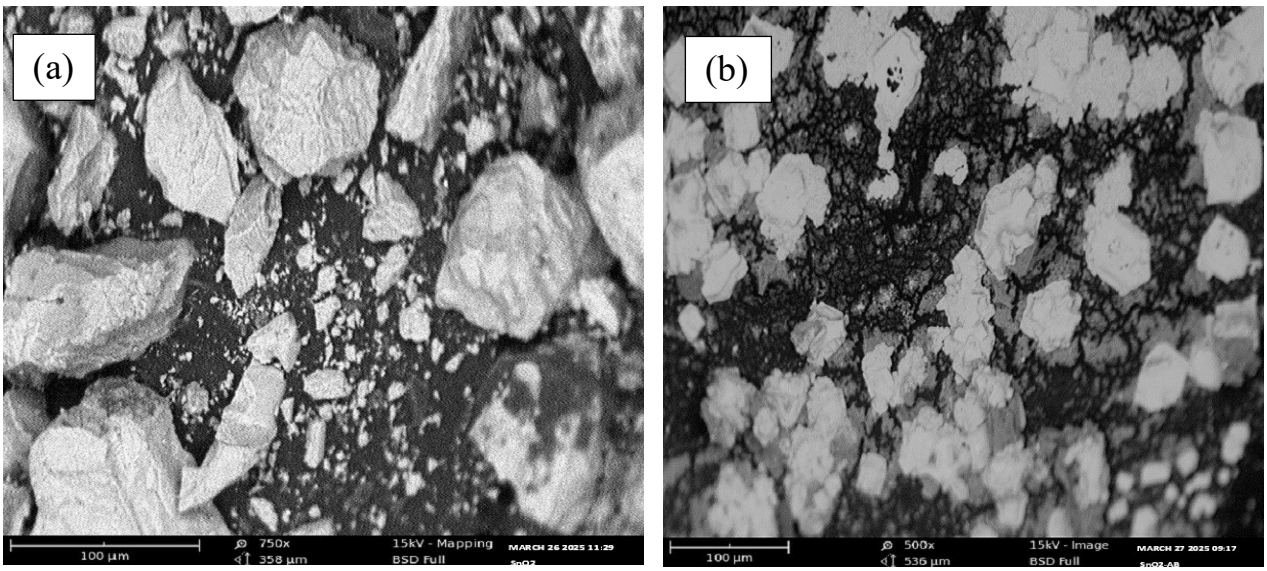

Figure 4: SEM Micrographs before a) SnO_2-Np and after adsorption b) SnO_2-Np – AB

3.2 Batch Adsorption Studies

Effect of experimental Parameters such as Effect of Contact time, adsorbent dosage, Initial dye concentration, pH and Temperature were carried out.

Effect of Contact Time

One of the most foremost factors to fabricate an efficient adsorbent for wastewater treatment is the contact time factor (Kumari *et al.,* 2017). Figure 5 shows the results of influence of contact time on the adsorption of AB dye molecules using SnO_2 was studied through various time intervals (1–60 min). The equilibrium time obtained for AB onto SnO_2 is 20minutes where 77.4% of the dye was removed. This is due to high interaction between adsorbent and the dye, and also the availability of more active sites on the nanoparticles for the dye adsorption (Ibrahim and Ibrahim, 2018a). The adsorption active sites were then reduced as the adsorption time increases (Moufi *et al.,* 2016), the decrease might be due to exhaustion of active sites and repulsive force between the adsorbent and adsorbate.

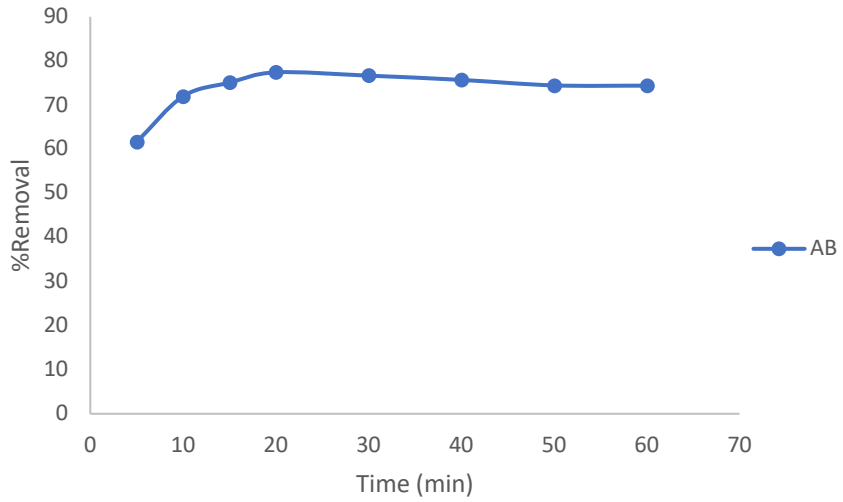

Fig. 5: Effect of Contact Time on the Removal of AB Using SnO_2

Effect of Adsorbent Dosage

The determination of the effect of adsorbent dose is very important because it estimates the capacity of adsorbent to remove the amount of dye. Initially the amount of dye adsorption increases because of empty active sites on the adsorbent surface (Ehrampoush *et al.,* 2011). Figure 6 shows the result of effect of adsorbent dosage on the adsorption of AB onto SnO_2 which was studied through various dosage varied from (0.2–1.0 g). In the adsorption of AB onto SnO_2 there were sharp increase in the adsorption capacity from 0.2 – 0.6 g, where 78% of the dye was removed. From 0.8g there was decrease in the percentage removal. The decrease may be due to decrease in the surface area available resulting from overlapping or aggregation of adsorption sites as similarly observed by Nethaji *et al.* (2013).

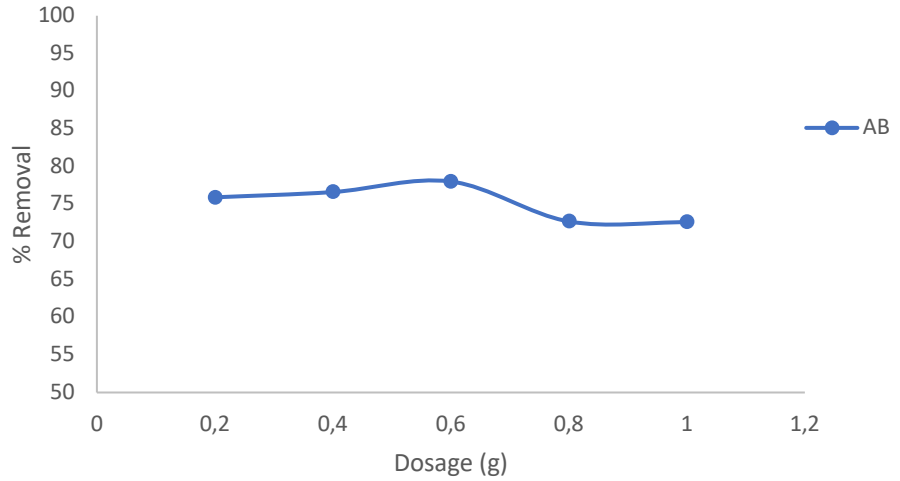

Figure 6: Effect of Dosage on the Removal of AB Using SnO_2

Effect of Initial Dye Concentration

Figure 7 shows the results of effect of Initial dye Concentration on the adsorption of AB using SnO_2 in which various concentrations of 10, 20, 30, 40, 50, 100, 150, 200, 250 mg/L was studied. The study showed that the removal efficiency increase with increase in initial concentration to 92.68% at 150 mg/L. Increase in the concentration at higher concentration may be due to the availability of active sites for saturation at higher concentration.

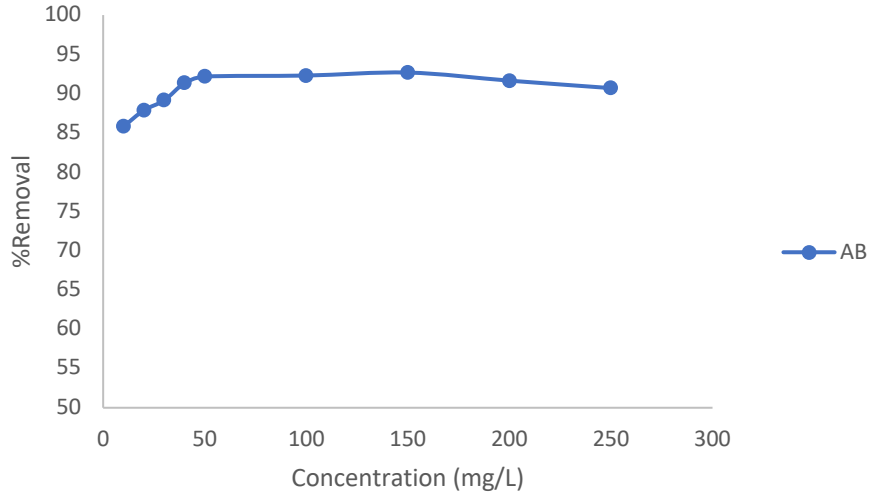

Figure 7: Effect of Concentration on the Removal of AB Using SnO_2

Effect of pH

Figure 8 shows the results of effect of pH on the adsorption of AB using SnO_2. pH of the dye were varied from 2- 12. The removal efficiency decreases with increase in pH from 95.8% at pH 2 to 80.6% at pH 12. AB is an anionic dye that exist in the form of negatively charged ions when in aqueous

solution. The rate of adsorption onto the surface of the adsorbent is controlled by the surface charge on the adsorbent which is influenced by the pH of the solution (Alqarni, 2022).

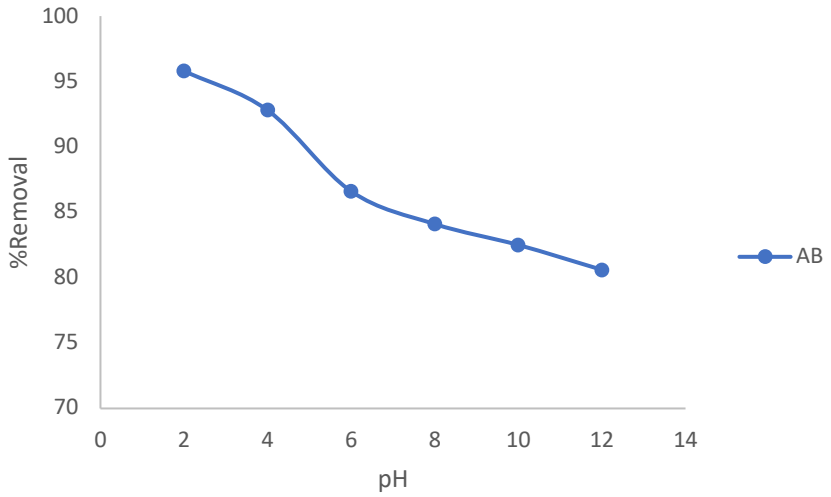

Figure 8: Effect of pH on the Removal of AB Using SnO_2

Effect of Temperature

The effect of temperature was studied by varying the temperature from 303 to 333 K. Figure 9 shows the results of effect of Temperature on the adsorption of AB dye molecules using SnO_2. The results shows that there is slight decrease in the removal efficiency with increase in temperature. The decrease in removal efficiency with increase in temperature is attributed to desorption of weakly bonded or physisorbed adsorbates molecules due to increased solubility in liquid phase. The physisorbed molecules are far from the surface (low binding energy) and require lower energy for desorption. With increase in temperature, the attractive forces between adsorbent surface and dye molecules are weakened and the adsorption decreases (Ibrahim and Ibrahim, 2018b). .

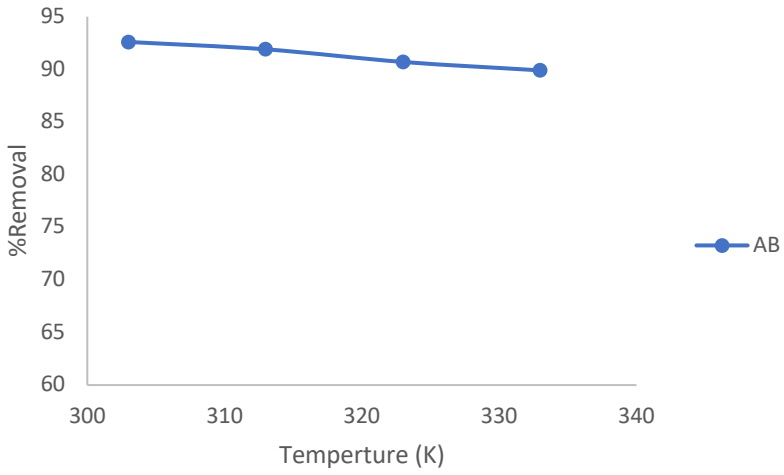

Figure 9: Effect of Temperature on the Removal of AB Using SnO_2

Kinetics Study of the Adsorption Process

Kinetic studies were employed in order to investigate the mechanism of the adsorption process and its potential rate controlling steps. In order to understand the kinetic behaviour of the adsorption process, kinetic models such as pseudo first order, pseudo second order, Elovich kinetic and intraparticle diffusion models were evaluated to fit the results of the experimental data and are summarized in **Table 2**. From the data analysis, the q_e experimental and q_e calculated values from pseudo first order are very far from each order for all the adsorption process. Also correlation coefficient (R^2) for all the adsorption process was very low compared to other kinetic models in the table 2. Also it was observed that the experimental results were best fitted to pseudo-second order kinetic model due to it having correlation coefficient (R^2) closer to unity and the q_e experimental and q_e calculated values were close.

Table 2: Kinetics parameters for the adsorption of AB onto SnO_2-NPs

Models	Kinetic Parameters	AB
Pseudo First Order	$q_{e,exp.}$(mg/g)	3.9193
	$q_{e,cal.}$(mg/g)	0.5921
	k_1(min^{-1})	0.0352
	R^2	0.3104
Pseudo Second Order	$q_{e,exp.}$(mg/g)	3.9193
	$q_{e,cal.}$(mg/g)	3.8715
	k_2(g/mg.min)	0.3011
	R^2	0.9977
Elovich	α(mg/g.min)	1547.9
	β(mg/g)	3.1776
	R^2	0.5968
Intraparticle Diffusion	C (mg/g)	3.0280
	k_{diff}(mg/g.$min^{-1/2}$)	0.1228
	R^2	0.4688

Thermodynamic Study of the Adsorption Process

Thermodynamic considerations of an adsorption process are necessary to conclude whether the process is spontaneous or not. The negative values of ΔG shows that the adsorption process is feasible and spontaneous. The change in free energy for physisorption is usually between –20 and 0 $kJmol^{-1}$, whereas chemisorption are in the range of –80 to 400 $kJmol^{-1}$ (Zarrouk *et al.* 2012). ΔG values obtained in this research was within the range of –20 and 0 $kJmol^{-1}$, implying that the adsorption process is through physisorption mechanism. Similar results showing low values of ΔG was reported by Salmani *et al.* (2017). The negative values of ΔH for adsorption confirmed that the adsorption is exothermic in nature (heat is released from the adsorption process). The value of ΔS is negative which implies a decreased in randomness/disorder at the solid/liquid interface during the adsorption process (Saha and Chowdhury, 2011).

Table 3: Thermodynamic parameters for adsorption onto SnO_2-NPs

Dyes	ΔG (kJ/mol) ΔG = ΔH – TΔS				ΔH (kJ/mol)	ΔS ($Jmol^{-1}K^{-1}$)
	303	313	323	333		
AB	- 6.409	- 6.284	- 6.160	- 6.036	- 10.173	- 12.424

Isotherm Study of the Adsorption Process

Adsorption isotherms describe how adsorbates interact with adsorbents and so are critical in optimizing the use of the adsorbents. In this research the equilibrium data were modeled using Langmuir, Freundlich, Temkin, D-R, Elovich, Jovanovic, Harkin-Jura and Redlich and Peterson isotherms. Based on the linear regression coefficient (R^2) obtained for each model, it was found that Freundlich model provided the best fit for the adsorption process. The adsorption process is said to be favourable when the value of n_F satisfies the condition $1 < n_F < 10$, otherwise it is unfavourable (Ibrahim and Sani, 2014). The values of n_f for the adsorption process is within the range $1 < n_F < 10$, indicating a favourable physical adsorption. The Temkin heat of adsorption B_T values for the adsorption process being less than 80 kJ/mol indicates a favourable physical adsorption for the dye onto the adsorbent as described by Inglezakis and Zorpas (2012). The magnitude of E is used from D – R isotherm model for estimating the type of adsorption mechanism. If the magnitude of E is between 8 and 16 kJ/mol, it is indicated that the adsorption process is chemical adsorption, while for value of E < 8 kJ/mol; the adsorption process is physical in nature (El-Araby *et. al.,* 2017). The value of mean square energy E for the adsorption being < 8 kJ/mol further indicate a successful physical adsorption.

4.2.3 Desorption Studies

Desorption studies help to explain adsorbate and adsorbent recovery, and the adsorption mechanism. Since the regeneration of the adsorbent makes the treatment process economical, desorption studies were performed to regenerate the spent adsorbent (Senturk and Alzein, 2020). To explore the possibility of regeneration of spent adsorbent, desorption studies were evaluated for their desorption efficiency. Various regenerates such as HCl, NaCl, NaOH and Distilled Water (H_2O). From the results presented in Figure 10, it shows that NaOH offered better desorption efficiency for AB. Distilled water produced the least desorption efficiency. In basic medium, the concentration of hydroxide ions was effective to desorb AB. The findings was in same with previous report on Egg By-Products as a Tool to Remove Direct Blue 78 Dye from Wastewater: Kinetic, Equilibrium Modeling, Thermodynamics and Desorption Properties (Murcia-Salvador *et al.,* 2020).

Table 4: Isotherms parameters for the adsorption of AB onto SnO_2-NPs

Adsorption isotherm	Parameters	AB
Langmuir	Q_M(mg/g)	84.75
	K_L(L mg^{-1})	0.016
	R_L	0.200
	R^2	0.2004
Temkin	A_T(L/mg)	0.502
	B_T(J/mol)	14.392
	R^2	0.9547
Freundlich	n_f	1.2043
	1/n	0.830
	k_f(mg/g)	1.160
	R^2	0.9666
D-R	X_M(mg/g)	20.49
	β (mol^2/J^2)	2×10^{-6}
	E(kJ/mol)	0.500
	R^2	0.8153
Elovich	qm(mg/g)	62.89
	K_E (L/mg)	4.330
	R^2	0.2574
Jovanivic	qm(mg/g)	3.111
	K_J	0.134
	R^2	0.7722
Harkin Jura	B	1.039
	A	3.867
	R^2	0.4413
Redlich Peterson	B	0.204
	A (L/g)	1.160
	R^2	0.4544

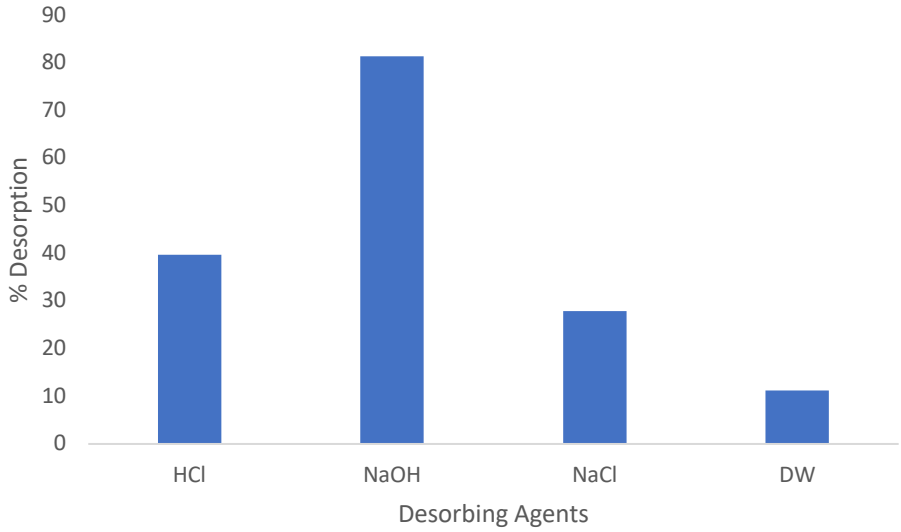

Figure 10: Selection of Desorbing Medium Using SnO_2

4.2.4 Reusability Studies

The adsorption efficiency of AB decreases from the first cycle to the fifth cycle as seen in Figure 11. This can be due to the saturation of the surface of the adsorbents (Baytar *et al.,* 2020). The successful reuse of SnO_2 for up to five cycles shows that the adsorbents can be used for multiple times for the removal of AB dye from wastewater. Similar trend of results was reported by Kyzas *et al.* (2022) who studied Removal of Benzene and Toluene from Synthetic Wastewater by Adsorption onto Magnetic Zeolitic Imidazole Framework Nanocomposites in six successive cycles.

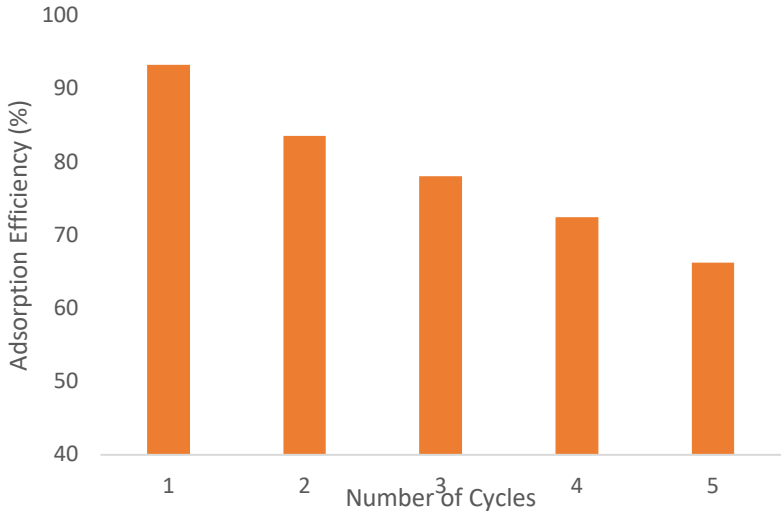

Figure 11: Adsorption Efficiency for Reuse of SnO_2 after Five Cycles

Conclusion

In this research, SnO_2 nanoparticle has been synthesized and used as an effective adsorbent for the removal of AB dye from aqueous solutions. The effects of variables, such as initial dye concentration, contact time, dosage, temperature and pH were studied. Isotherm modeling revealed that the Freundlich model described the adsorption of AB. The adsorption process fit successfully to pseudo-second-order kinetic model. The values of mean free energy indicate that the adsorption process to be physical in nature. Desorption studies shows it can be regenerated successfully using NaOH. Reusability studies shows that the adsorbent can be reused for more than five cycles. In view of these results, it can be concluded that synthesized SnO_2 nanoparticle can be utilized as a low-cost and effective adsorbent in the removal of AB from aqueous solutions.

Conflict of Interest: The authors declare that the research was conducted in the absence of any commercial or financial relationships that could be construed as a potential conflict of interest.

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