Anchor University Journal of Science and Technology (AUJST)
A publication of the Faculty of Science Applied and Health Science, Anchor University Lagos

URL: journal.aul.edu.ng

In AJOL: https://www.ajol.info/index.php/aujst

Vol. 6 No 1, July 2025, Pp. 62-71

ISSN: 2736-0059 (Print); 2736-0067 (Online)

Synthesis, Characterization of Zirconium Dioxide Nanoparticles and Its Adsorption Kinetics

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Submitted 30 October, 2024
Accepted 01 May, 2025

Competing Interests.
The authors declare no competing interests.

ABSTRACT

.This study presents the synthesis of zirconium dioxide (ZrO_2) nanoparticles using a green synthesis method with aqueous neem leaf extract as the reducing agent. The synthesized nanoparticles were characterized using UV-Visible spectroscopy which gave an absorbance at 350 nm, X-ray diffraction (XRD) which shows the crystallinity class of the ZrO_2 NPs to be tetragonal, and scanning electron microscopy (SEM) showed spherical like shapes, homogenous dispersion in nature and agglomeration. The application of these nanoparticles in wastewater treatment was explored by analyzing adsorption effects under varying conditions such as temperature which indicated a maximum dye removal of 97% at 30^{o}C , a high pH favoured higher removal of dye at 98.9% , adsorbent dose increased from (0.15 g = 87% to 1.05 g = 97%), as the agitation speed increased the removal percentage increased from 87 to 94%, initial concentration showed an initial increase, and contact time peaked at (30 min = 98% to 105 min = 90%). The adsorption process was further evaluated using isotherm models which showed a Freundlich value n of 1.4963 and kinetic model result showed a pseudo-second order kinetic model with a high correlation coefficient (R^2 = 0.9993). The thermodynamic parameter revealed that the adsorption process is spontaneous, feasible and endothermic. This study demonstrates the potential of ZrO_2 nanoparticles as an effective adsorbent for wastewater treatment.

Keywords. synthesis, characterization, zirconium dioxide nanoparticles, adsorption kinetics

1. Introduction

Nanochemistry is a branch of nanoscience which deals with the chemical application of nanomaterial in nanotechnology. It also involves the study of the synthesis and characterization of material on a nanoscale science Brown, (2011). Nanochemistry is relatively a new branch of chemistry concerned with the unique properties associated with assemblies of atoms or molecules of nanoscale (~1-100 nm). It is also the science of tools, technologies and methodologies for novel chemical synthesis example employing synthetic chemistry to make nanoscale building blocks of desired shape, size composition and surface structure and possibly the potential to control the actual self-assembly of the building blocks to various desirable sizes.

The field of nanotechnology is one of the most active areas of research in modern materials science. New applications of nanoparticles (NPs) and nanomaterials are emerging rapidly. Green synthesis provides advancement over chemical and physical method as it is cost effective, environment friendly, easily scaled up for large-scale synthesis and further there is no need to use high pressure, energy, temperature and toxic chemicals. Synthesis of NPs using biological entities has great interest due to their unusual optical, chemical, photo electrochemical and electronic properties (Mohanpuria et al., 2008). Nanotechnology has emerged as a promising field for environmental application, particularly in waste water treatment.

Zirconium dioxide (ZrO_2) nanoparticles (NPs) have shown potential due to their high surface area and stability and have gained significant attention in recent years due to their unique properties and potential applications in various fields, including catalysis, electronics, and environmental remediation (Tran et al., 2022).

The increasing contamination of water bodies by industrial dyes poses significant

https://dx.doi.org/10.4314/aujst.v6i1.6

environmental and health risks. Methylene blue, a common dye used in textile and paper industries, is a major pollutant in wastewater (Oladoye et al., 2022). Traditional methods of dye removal are often inefficient and environmentally hazardous. The use of nanoparticles, particularly those synthesized through green methods, offers a promising alternative for effective and sustainable wastewater treatment (Goutam et al., 2020).

This study focuses on the green synthesis and characterization of ZrO_2 NPs using neem leaf extract which is environmentally friendly, cost effective and non-pollutant for the adsorption of methylene blue in wastewater treatment.

MATERIALS AND METHOD

Materials

Test tubes, distiller, volumetric flask, pipette, wash bottles, pyrex glass bottles, filter paper and beakers. Neem (*Azadirachta indica*) leaves, Zirconium chloride ($ZrCl_4$, 99%), methylene blue (MB, 98.5%) and deionized (DI) water was used throughout the experiments, Oven (Gallenkamp, MODEL A050626), centrifuge (Hermle, Z 300, Germany), heating magnetic stirrer CB162, thermal balance (PERKIN ELMER-TGA 300/15), Scanning Electron Microscopy (SEM model Q150R, Phenom ProX,by phenomWorld Einhoven), X-ray Diffractometer (XRD, Philips Analytical, PW-3040), UV-Vis spectrophotometer (Genesys 10s), Perkin Elmer Frontier Fourier Transform Infrared Spectroscopy (FTIR).

Method

Collection, Pretreatment and Preparation of Neem (*Azadirachta indica*)

The leaves of Neem (*Azadirachta indica*) were collected from Federal College of Forestry Jos North Local Government Area Plateau State, Nigeria. The plant leaves were validated by a staff of Department of Botany, University of Jos, Plateau State, Nigeria with a voucher number FHJ 796. The leaves were washed with tap water to remove dirt and dust. Furthermore, the plant leaves were washed with distilled water for more cleaning and were air dried indoors at room temperature (Biswas *et al.,* 2002). Leaves were ground using clean mortar and pestle to fine powder. Ten (10) grams of the powder was dissolved in 100 cm^3 of distilled water and heated for 1h at 80°c. The extract was filtered through Whatman's filter paper No. 1, the filtrate was collected in a 250 cm^3 volumetric flask and then stored at 4°C for further use, modified from (Vimala et al., 2014).

Green synthesis of Zirconium dioxide Nanoparticles

Exactly 2 g of zirconium chloride was weighed and dissolved in 100 cm^3 aqueous leaves extract solution of Neem (*Azadirachta indica*) under constant stirring. After complete dissolution of the mixture, the solution was kept under vigorous stirring at 80°C for 2 hours, subsequently allow to cool at room temperature and a precipitate was formed and the supernatant was discarded. The obtained precipitate solution was centrifuged at 4,000 rpm for 15 min after thorough washing with distilled water and dried at 80°C for 4 hours in an oven (Jalill et al., 2017).

Characterization of Zirconium dioxide Nanoparticles

The green synthesis of Zirconium dioxide NPs was characterized using UV-visible spectrophotometer, Fourier-transform Infrared Spectroscopy (FTIR) and Scanning Electron Microscopy (SEM).

Absorbance measurement was carried out at a wavelength range of 310-700 nm using UV-visible spectrophotometer at a scan interval of 5 nm in order to obtain the UV-visible spectrum of the nanoparticles (Priyanshu, 2023). SEM was used to examine the surface morphology and size of the synthesized ZrO_2 nanoparticles. X-ray Diffraction (XRD) patterns of the ZrO_2 NPs were analyzed to observe the crystal phase, crystalline size and purity of ZrO_2 NPs.

Batch Adsorption Studies: Preparation of Stock Solutions of Methylene blue Dye

Stock solution was prepared in deionized water from the Methylene blue ($C_{16}H_{18}N_3ClS$, MW: 373.90) and an aqueous stock solution (1000 mg/L) was prepared by dissolving 1.0 g of the dyes in 1000 cm^3 of deionized water.

One hundred cubic centimeter (100cm^3) of the stock solution was transferred into 1000cm^3 volumetric flask using a pipette and 900 cm^3 of deionized water was added to the mark. Same procedure was carried out for 200 cm^3 and 800 cm^3 deionized water was added to the mark.

Serial dilution 100 mg/L, 200 mg/L, 300 mg/L, 400 mg/L 500 mg/L 600 mg/L 700 mg/L of dyes were prepared from the standard solution (Kumar et al., 2014).

Batch Adsorption Experiments: A batch adsorption study was carried out using 120 cm^3 sample bottles. The effects of different

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parameters such as initial adsorbate concentration, adsorbent dose, pH, temperature, contact time and agitation speed were studied. The Erlenmeyer flasks (120 cm^3) were pretreated with the respective adsorbate for 24 hours to avoid adsorption of the adsorbate on the container walls. Following similar methods for the adsorption parameters used by Ozer et al. (2007) and ,Kumar et al (2014) the methods used are listed below.

Effect of Temperature: One hundred and fifty milligrams (150mg) of the adsorbate were added to the MB solution (100 cm^3) at the initial concentration of 700mg/L and it was centrifuged for 120min at range of temperatures from 30^0 C to 90^0 C. The adsorbed amount of MB in percentage was recorded.

Initial Concentration: The effect of initial dye concentration was carried out by preparing adsorbent–adsorbate solution with all other parameters under study at a constant and different initial dye concentration (700 mg/L, 600 mg/L, 500 mg/L, 400 mg/L, 300 mg/L, 200 mg/L and 100 mg/L) and centrifuged until equilibrium was achieved.

pH: The effect of pH solution on the adsorption process was studied by preparing adsorbent–adsorbate solution with fixed adsorbent dose, dye concentration, temperature, contact time and temperature but with different pH from 1-13 by adding NaOH (0.1 M) or HCl (0.1M) solutions and then centrifuge until equilibrium.

Agitation Speed: The standard solutions of the MB were mixed with the adsorbent and agitated at different agitation speeds (500, 1000, 1500, 2000, 2500, 3000 and 3500 rpm) on a centrifuge. The resulting suspension of each of the dyes was filtered using a Whatman No.1 filter paper and the filtrate was analyzed for the corresponding dye concentration. The MB concentrations were determined using a wavelength at 660 nm after getting the UV spectra of the solution with a spectrophotometer. The calibration curve was obtained from the spectra of the standard solutions (100 – 700 mg/L) concentrations. Removal efficiency was finally calculated by using the relationship. Adsorption Equations:

$$\%\ Adsorption = (Co - Ce / Co)\ X\ 100 \quad (1)$$

$$qe = V\,(Co - Ce) / W \quad (2)$$

Where Co (mg/L) is the initial dye concentration, Ce is the concentration at equilibrium of pre-determined time t, V (L) is the volume of dye solution used and W (g) is the weight of the adsorbent.

Effect of Dosage: The adsorbent–adsorbate solution was prepared with different quantity of adsorbents (0.15, 0.3, 0.45, 0.6, 0.75, 0.9 and 1.05 g) added to fix initial dye concentration then centrifuge together until equilibrium time.

Effect of Contact Time: The adsorbent–adsorbate solution was studied as a function of varied centrifuge /contact time (15, 30, 45, 60, 75, 90, and 105min) at temperature of 30^0 C pH of 7, adsorbent dose of 0.15 g and concentration of 700 mg/L of MB.

RESULTS AND DISCUSSION

In this study, the green synthesis of Zirconium dioxide nanoparticle was synthesized using *Azadirachta indica* leaf extract. The color change from light green to dark violet indicates synthesis of ZrO_2 NPs.

Characterization of ZrO_2

The UV-visible absorption spectrum of ZrO_2 NPs (Figure 1) shows the characteristic surface plasmon resonance (SPR) with strong absorbance at 350 nm peak maxima, which can be attributed to the formation of ZrO_2 (Arshad et al., 2022).

X-ray Diffraction (XRD) patterns of the ZrO_2 NPs were analyzed to observe the crystal phase, crystalline size and purity of ZrO_2 NPs. The crystalline phase peaks were observed at 2θ = 17.4°, 25.2°, 27.9°, 29.6°, 39.5° and 4.2° corresponding to (110), (101), (111), (102), (112), and (211) (Figure 2). planes respectively. These belong to tetragonal-ZrO_2 having lattice parameters of (a = 3.695 Å, b = 5.185 Å, c = 5.312 Å, d = 5.212 Å, e = 5.147 Å, and f = 5.147 Å). This outcome agrees with Tharani, (2016) who also synthesized ZrO_2 NPs. Particle size = 59.37±0.01 calculated using Schearer

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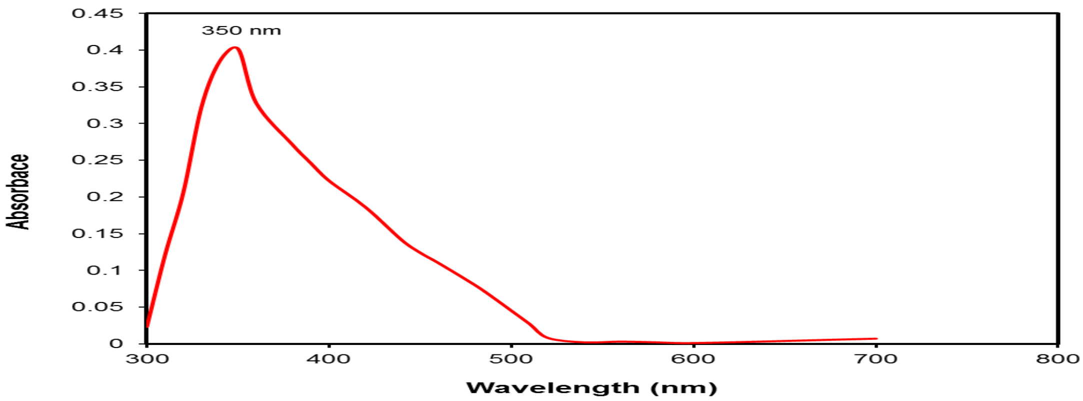

Figure 1: **UV-Visible Spectrogram of the Synthesized ZrO_2**

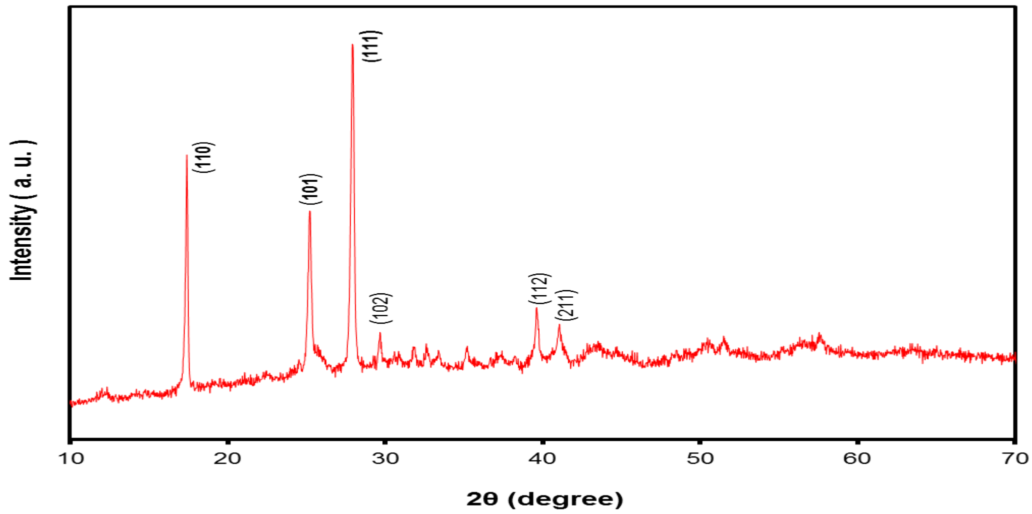

Figure 2: XRD pattern of synthesized ZrO_2

equation (InstaNANO,2024).

The morphology, image and physical structures of ZrO_2 NPs was identified using SEM of the nanoparticles with various magnifications of 1500, 1000, 500 and 300 from top clockwise. The images shows that the ZrO_2 NPs are spherical-like shapes, homogenous dispersion in nature and agglomerations were formed (Buzuayehu, 2012). This dispersed state increases the surface area of the ZrO_2 NPs hence conferring it with excellent adsorbent properties (Figure 3).

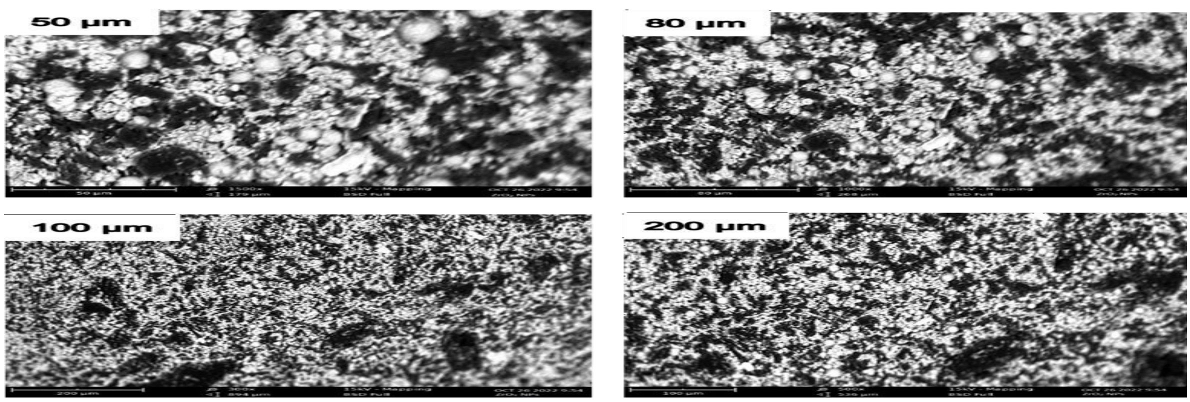

Figure 3: SEM Micrograph

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Batch Adsorption Parameter

Effect of Temperature

It was observed that the adsorption capacity decreased from 97 to 82.4 % with increase in temperature from 30 to 90°C. This indicates that the ZrO_2 NPs optimally adsorb just below 30 degrees Celsius. Which makes it ideal for ambient room temperature maximum adsorption of the dye in waste water.

Effect of Adsorbate Concentration

It was observed that the percentage of dye removal (from 100%) decreases with an increase in the initial dye concentration at 100 mg/l. At 700mg/l the percentage dye removal was at 96, which may be due to the saturation of adsorption sites on the adsorbent surface (Radovic et al., 1997). At a low concentration there will be unoccupied active sites on the adsorbent surface, and when the initial dye concentration increases, the active sites required for adsorption of the dye molecules will not be optimally available due to the occupation of these sites by the higher concentrations as shown on the graph on Figure 5 (b).

Effect of pH Solution

Generally, at low pH of the solution, the percentage of dye removal will decrease for cationic species of the dye adsorption, as seen in the result of 4 (c) where percentage removal dropped to below 80%. At high pH solution, the cationic species in the solution interface decreases, but increases at the adsorbent surface which appears negatively charged. As a result, the cationic species dye adsorption increases and anionic dye adsorption shows a decrease. Figure 4 (c) shows an increase with pH from 1-13, the percentage removal showed an increase from 90.8 % to 98. 9% as the pH increased especially above pH of 7.

Effect of Agitation Speed

The effect of the agitation speed on the MB dye adsorption were determined. Figure 4 (d) shows that increase in agitation speed (500-3000 rpm) resulted in a corresponding increase in the percentage removal (87 % to 94%), this agrees with similar results obtained by (Hadjmohammadi et al.,2011).

Effect of Dosage

The effect of adsorbent dosage is shown in Figure 4 (e). It was observed that there was an increase in sorption capacity and percentage removal respectively as the adsorbent dose increases from (0.15g = 87% to 1.05 = 97%) and this is as a result of increased binding sites made available by increasing the adsorbent dose (Calvete et al., 2009).

Effect of Contact Time

From Figure 4 (f) the contact time, it is observed that at 30 minutes it exhibited a high sorption capacity before subsequently declining as the contact time increased while the percentage removal also showed a decrease from (15 minutes = 98% to 105 minutes = 90%). This result agrees with that obtained by (Kumar et al., 2014).

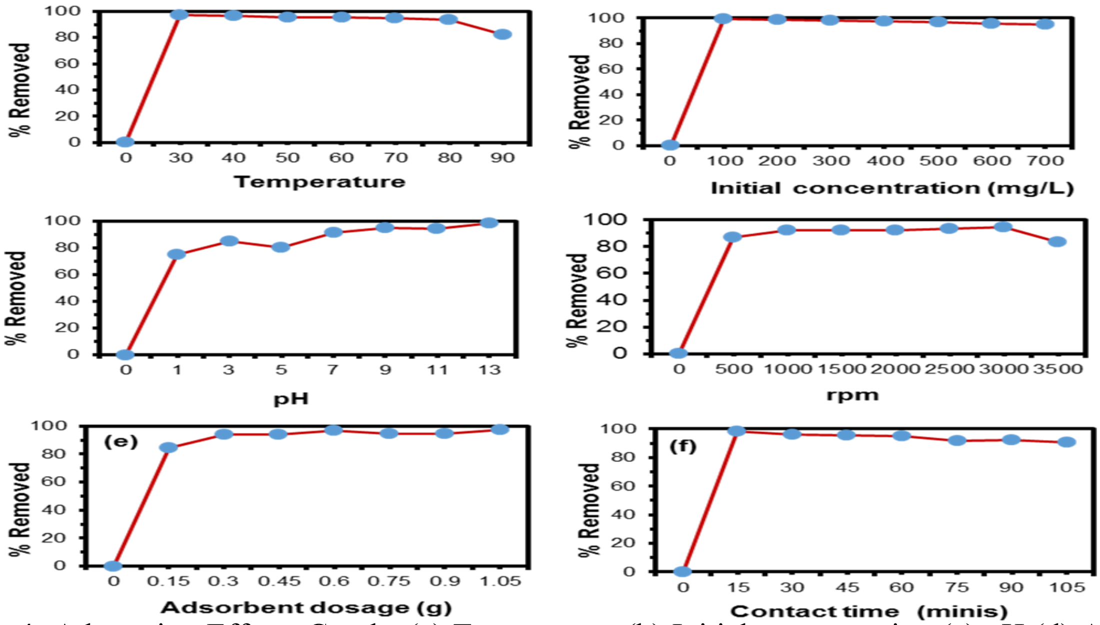

Figure 4: Adsorption Effects Graphs (a) Temperature (b) Initial concentration (c) pH (d) Agitation Speed (e) Adsorbent dosage (f) Contact time

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Adsorption Isotherms Modeling

The graphs on Figure 5 shows the Adsorption Isotherms of (a) Langmuir Isotherm (b) Freundlich isotherm. The isotherms and their parameters are presented in Figure 4.4 and Table 1 respectively. From the values of the correlation coefficient (R^2) obtained, the Freundlich model showed the best relationship in the adsorption process followed by the Langmuir model with R^2 values of 0.9415 and 0.9046 respectively. Also, n value of 1.4963 was obtained from the Freundlich equation. According to Batool et al. (2018), a value of n greater than 1 is an indication of favorable adsorption

Adsorption Kinetics Modeling:

The adsorption kinetics was determined by the method described by measuring adsorptive uptake of dye from aqueous solution at different time intervals from 30 to 105 minutes. Kinetic data were fitted using the Lagergren pseudo-first-order model, a pseudo-second-order model (Blanchard et al., 1984) and an intraparticle diffusion model (Weber and Morris, 1963) to investigate the mechanism of adsorption and potential rate-controlling steps. The pseudo-first-order Lagergren equation is given as:

$$log(q_e - q_t) = \log q_e - \frac{K_1 t}{2.303}$$

(3)

The pseudo-second-order equation is

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{1}{q_e} t$$

where qe (mg/g) is the mass of metal adsorbed at equilibrium, qt (mg/g) the mass of metal adsorbed at time t, k_1 is the pseudo-first-order rate constant of adsorption (min^{-1}) and k_2 is the pseudo-second-order rate constant of adsorption (mg/g.min). The intra-particle diffusion equation can be written as follows:

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

(4)

Where C is the intercept which describes the boundary layer thickness and Kt (mg/g.min1/2) is the rate constant of intra-particle diffusion.

The results obtained from kinetic studies are shown in Figure 6 and Table 2**.** The pseudo-first -order and pseudo-second-order kinetic model plot with a best fitting for correlation coefficient ($R^2 < 0.9258$) and ($R^2 < 0.9993$) respectively. The results, however is suitable for the adsorption process, indicated very good compliance with the pseudo-second-order kinetic model with a high correlation coefficient ($R^2 = 0.9993$). The large intercept obtained from the intraparticle plot implies that surface adsorption plays a great role in the rate-limiting step (Krishna *et al*., 2018).

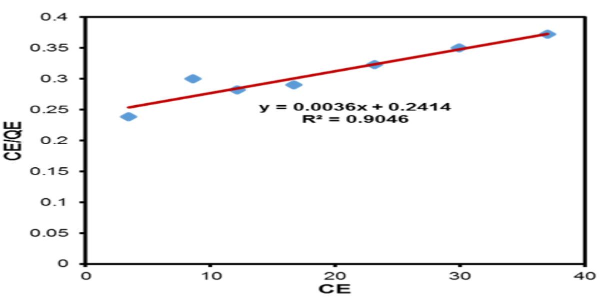
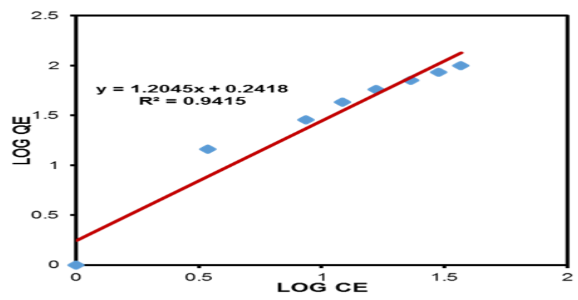


Figure 5: (a) Langmuir Isotherm



(b) Freundlich isotherm

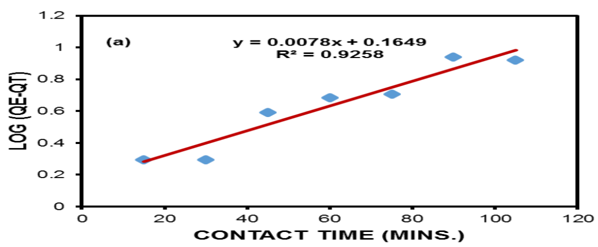
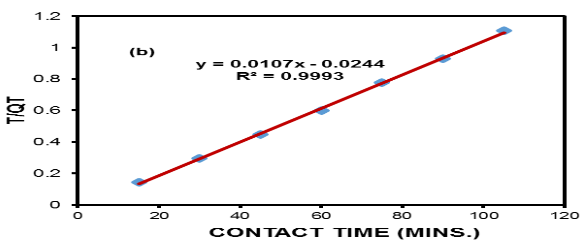


Figure 6: Pseudo (a) First and



(b) Second order Kinetics

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Table 1: Isotherm Constants

Isotherm models	**Constants**	**Methylene blue**
Langmuir	KL (Lmg^{-1})	0.0345
	qmL (mgg^{-1})	1.0231
	R^2L	0.9046
Freundlich	KF (Lmg^{-1})	0.3678
	n (g/L)	1.4963
	R^2F	0.9415

Key: KL = Langmuir constant. $\mathbf{qm_L}$ = maximum adsorption capacity. $\mathbf{R^2{}_L}$ = Separation factor Langmuir. $\mathbf{R^2{}_F}$ = Separation factor Freundlich. **n** = Adsorption capacity. **KF** = Freundlich constant

Table 2: Kinetic Constants

Adsorbent	**DYES Adsorbate**	$\mathbf{q_{e,exp}}$ **(mg/g)**	**Pseudo- first order**			**Pseudo- second order**		
			$\mathbf{q_{e,cal}}$**(mg/g)**	$\mathbf{K_1}$	$\mathbf{R^2}$	$\mathbf{q_{e,cal}}$ **(mg/g)**	$\mathbf{K_2}$	$\mathbf{R^2}$
ZrO_2 NPs	**MB (cationic)**	9.0564	0.6759	0.0006	0.9258	5.7690	0.8565	0.9993

Thermodynamic study

The thermodynamic parameters were evaluated the effect of temperature on the adsorption process using the following mathematical relationship from experimental data obtained at various temperature. The standard free change (ΔG^0), enthalpy (ΔH^0) and enthalpy (ΔS^0). (Jagung, 2011; Saleem and Bhatti, 2011).

$$K_c = \frac{C_{ads}}{C_{sol}}$$
(5)

$$\Delta G^o = -RT\ lnKc$$
(6)

$$lnKc = \frac{\Delta_{S^o}}{R} - \frac{\Delta H^o}{RT}$$
(7)

The values obtained for various thermodynamic parameters are given in Figure 7 and Tables 3. Negative values were obtained for ΔG0 at all temperatures and the values increased with an increase in temperature. According to Singha and Das (2011), negative ΔG0 values indicate spontaneous and feasible adsorption. The positive values of ΔH^o confirmed the endothermic nature of dye sorption by ZrO_2 NPs, the positive value of entropy change (DS°) corresponds to an increase in the degree of freedom of the adsorbate because the number of desorbed water molecules was larger than that of the adsorbed molecules. Therefore, the driving force of MB molecule adsorption on ZrO_2 NPs was because of the entropy effect rather than enthalpy change (Singha and Das 2011).

CONCLUSION

This study shows that the ZrO_2 NPs can be used as an adsorbent for the adsorption of MB from its aqueous solution. The amounts of dyes adsorbed were found to vary with contact time, adsorbent dosage, pH, initial concentration, and

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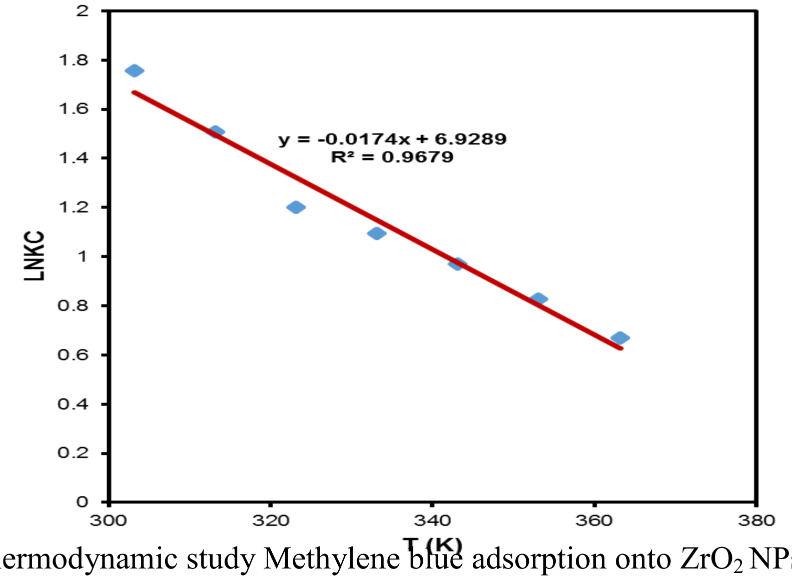

Figure 7. Thermodynamic study Methylene blue adsorption onto ZrO_2 NPs

Table 3. Thermodynamic parameters

agitation speed where adsorption of dyes increased with increasing adsorbent dose but decreases with increasing initial concentration of the dyes. The Langmuir, Freundlich isotherm models were used for the mathematical description of the adsorption equilibrium of the dyes onto the ZrO_2 NPs and both isotherms model described well fitted, which indicated that the adsorption process is favorable. The kinetic models were applied to examine the controlling mechanism of dye adsorption from aqueous solution. The pseudo - first order kinetic models and pseudo-second order kinetic model described the data better. The thermodynamic parameters such as ΔG, ΔS and ΔH was observed, the adsorption of the methylene blue dye by the adsorbent is spontaneous, feasible, endothermic and physical in nature from the negative and positive values of ΔG and ΔH respectively. The positive value of ΔS showed an increase in the degree of freedom of the adsorbed species. The potential applications of ZrO_2 NPs sorbent for adsorptive removal of methylene blue dye confirmed excellent percentage removal values from aqueous solutions.

ACKNOWLEDGMENT

We wish to thank the Department of Chemistry and the Nigerian Defence Academy for their support throughout this research work.

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