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Synthesis, Characterization and Adsorption of methylene blue dye using titanium dioxide nanoparticles

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Abstract
The application of nanomaterial in the reclamation of wastewater has been a trending aspect of research in the recent time. This research work was carried out to investigate the adsorption of methylene blue dye using TiO_2 nanoparticles. The TiO_2 nanoparticles used in this work was synthesized by sol-gel method using titanium isopropoxide. After the synthesis, the titanium dioxide powder that was obtained and was calcined at 500°C. The titanium dioxide nanoparticles obtained was characterized using SEM, FTIR, and XRD. The FTIR result showed that -OH group, $-CH_2$ group, C═O group and Ti-O-Ti stretching band played major role in the efficacy of TiO_2 nanoparticles. SEM morphology indicates the porous nature of the nanoadsorbent while the XRD confirm the crystalline nature of the nanomaterial. Influence of process variables were investigated and the results indicate that the three factors studied affected the adsorption process. Adsorption isotherm parameters gave good fit to the four models used in this work with R^2 values above 0.900. The kinetic studies did not fit well with Pseudo-first-order model but fitted the other three models.

Keywords: Nanomaterial, methylene blue dye (MBD), Titanium dioxide (TiO_2), nanoparticles, nanoadsorbent, adsorption, removal.

1. Introduction

In developing countries like Nigeria, water contamination has been the most challenging situation because of its negative impact on human health. There is always an indiscriminate discharge of industrial and domestic wastewater, oil spillage, improper waste disposal, agricultural run-off etc. The side effect of this indiscriminate discharge is so worrisome because diseases like typhoid, cholera, diarrhea, dysentery, skin irritation, cancer, liver dysfunction etc. are contracted through the use of contaminated water (Ota et al, 2025).

Industrial and municipal wastewater have continually been discharged into the environment without being properly treated and this calls for much attention (Lin et al, 2022). Industrial effluents usually have low pH, high temperature, high metals and metalloids, high cationic substances which are very detrimental to the ecosystem (Gezahegn et al, 2025).

Methylene blue dye is among the most frequently used synthetic dyes in textile industries (Rosly et al, 2022). Methylene blue has numerous uses which are dying of clothes, hair colorant, paint coloring, paper coloring etc. (Fayoud et al, 2016). The industries that use it generate a lot of wastewaters. It is regarded as a toxic substance since it can cause eye irritation, gastrointestinal irritation, diarrhea, cyanosis, dyspnea etc. (Akpomie et al, 2017; Igwegbe et al, 2019). As a result of this, it is very pertinent to treat wastewater generated from these industries before discharging them into the environment or better still recycle and reuse them after proper treatment.

Many adsorbents have been used by many researchers for the adsorption of dyes, heavy metals and other contaminants from wastewater. Some of these adsorbents are; activated carbon, biochar, nanoclay, carbon nanotube, zeolite, kaolinite, fly ash, metal and metal oxide etc. (Akpomie and Dawodu, 2016; Aniagor and Menkiti, 2024; Norsuhailizah et al, 2020).

The use of nanoadsorbents for wastewater treatment have captured the attention of researchers because of their unique physical and chemical properties such as size, shape, high surface area, magnetic properties, surface morphology, high solubility, high adsorptive capacity, high catalytic activities etc as compared to conventional adsorbents (Haijiao Kulkarni et al, 2016; Lu et al, 2016; Okey-Onyesolu et al, 2025).

Titanium dioxide which is also known as titania exists in four different polymorphs namely rutile, anatase, brookite and titanium dioxide B. The crystal structures differ from one another (Tijani et al, 2017). Different methods have been utilized in the synthesis of TiO_2 nanoparticles, nanotube and nanocomposite. Some of these methods are sol-gel method, hydrothermal method, aqueous solution based chemical method, chemical vapour deposition, direct oxidation method, sonochemical method, microwave method, co-precipitation method, solvothermal method, electro deposition (electroplating) (Irshad et al, 2021; Jaison et al, 2015, Selvi et al, 2022; Tijani et al, 2017).

Njabulo et al, 2024 reported the antibacterial efficacy of both the TiO2 andTiO2/Cu nanoparticles prepared through the sol−gel method. In the course of their studies they found out that the antibacterial efficiency was determined to be 90 and 80% against B. subtilis and E. coli, respectively. Selvi et al, 2022 synthesized TiO_2nanoparticles using green synthesis method. The TiO_2 nanoparticles they prepared was only oven dried without annealing. Sazid et al, 2025 developed TiO_2 nanoparticles with sol-gel method and calcination temperature of 550ºC and 950ºC. They investigated on the photocatalytic degradation of methylene blue dye. Urbashi et al (2022) fabricated TiO2/SiO2 nanoparticles for methylene blue removal and photocatalytic degradation under natural sunlight and low-power UV light. Okafor et al, 2025 investigated the Synthesis and Structural Characterization of Polyaniline–Tin Oxide Nanocomposite for Device Application.

This research work which is aimed at the synthesis, characterization and application of titanium dioxide nanoparticles for the adsorption of methylene blue dye. Sol gel method was used to synthesize the titanium dioxide nanoparticles. The synthesized nanoadsorbent was characterized using Fourier Transform Infrared (FTIR) analysis, Scanning Electron Microscopy (SEM) analysis, X-Ray Diffraction (XRD) analysis. From the literature, it was observed that a good number of works have been done on the synthesis of titanium dioxide using sol-gel method. But no work has been done using calcination temperature of 500ºC. Most of the studies on MBD removal were done using photocatalysis degradation method but this work investigated the use of batch adsorption method and the range of factors used in this investigation have not been used in other research works for the uptake of methylene blue dye. Most of the isotherm studies were limited to Langmuir and Freundlich models but this investigation was extended to Tempkin and DRK models. The kinetic studies were also extended to intraparticle diffusion and Elovich models.

The influence of process parameters on the removal efficiency of methylene blue dye using titanium dioxide nanoparticles was investigated. The influence of contact time on the adsorption efficiency was investigated at time range of 0.5mins to 8mins. Influence of concentration was studied at the range of 100mg/l to 300mg/l and dosage was determine at the range of 0.01g to 0.12g. The results obtained were used to carry out the isotherm and kinetic studies.

This investigation will be of benefit to industries that generate colored wastewater especially the textile industries. It will also help the researchers to know the areas that need further research. Most importantly, it will save cost, time and facilities in wastewater treatment processes

2.0 Materials and methods

2.1 Materials:

Titanium (iv) isopropoxide, Sodium Hydroxide, Distilled water, Tetraoxosulphate (vi) acid, Hydrochloric acid, Ethanol.

2.1.1 Synthesis of TiO_2 nanoparticles using Sol-gel method

Sol-gel method was used to obtain synthesized TiO_2 nanoparticles. Titanium (iv) isopropoxide was dissolved in ethanol in the ratio of 1: 2 of TTIP to ethanol and later water was added in TTIP to water ratio of 1:3, with continuous stirring at a speed of 1000rpm for 1hour. 0.2M solution of NaOH was added to adjust the pH to 7. It was stirred vigorously for 30mins to form sols. Thereafter, the sols were allowed to age for about 12hours in order to increase the thickness of the sols and form gel. After aging, the gel was dried under 200ºC for 1hour to evaporate the water and remove impurities through evaporation. The dried gel obtained was then calcined at 500ºC for 2hours. After which the substance obtained is Titanium dioxide nanoparticles.

2.1.2 Preparation of the dye stock solution

The dye and other chemicals used in this research were of analytical grade and were bought from Quality Chemicals Nig. Ltd, Onitsha. All the chemicals were used without further purification. To prepare the dye stock solution, 1g of methylene blue dye was weighed with electronic balance and poured into 1000ml volumetric flask and then filled with distilled water to the calibration mark to obtain 1000mg/l of the dye stock solution. The working solutions which are 100mg/l, 150mg/l, 200mg/l, 250mg/l etc. were prepared from the stock solution using the dilution formula.

2.2 Batch adsorption studies

Batch adsorption experiments were carried out at 30ºC in batch mode. The batch experiments were performed in different plastic containers of 100ml capacity using an average speed electric shaker of 500rpm. During each experiment, 0.1g of the adsorbent was added to 40ml of methylene blue dye solution in 100ml container and covered. The stirring was kept constant for each run throughout the experiment ensuring equal mixing. The container containing the sample was withdrawn from the electric shaker at the predetermined time interval, filtered through whatmann filter paper and analyzed. Influence of dosage, agitation time and concentration on the adsorption process were determined. Influence of contact time on the process was carried out at time intervals of 0.5, 1, 2, 3, 4, 5 and 8 minutes, dosage at 0.01, 0.03, 0.06, 0.08, 0.1 and 0.12grams and initial concentration at 100mg/l, 150mg/l, 200mg/l, 250mg/l and 300mg/l. The experimental adsorption capacity q_e (mg/g) and % Removal were calculated using equation 1 and 2 respectively (Abonyi et al, 2023):

$$q\mathrm{e} = \left(\frac{Co-Ce}{m}\right)V \qquad (1)$$

$$\%\mathrm{Removal} = \left(\frac{Co-Ce}{Co}\right) \times 100 \qquad (2)$$

Where C_o and C_e (mg L^{-1}) are the initial methylene blue dye concentration and the equilibrium concentration respectively; V (litre) is the volume of the methylene blue dye solution used and m (g) is the mass of dry titanium dioxide nanoparticles. The results obtained from the batch experiments were used for isotherm and kinetic modelling. Table 1 represents the model equations used in this investigation.

Table 1: Isotherm and Kinetic models equaions used in this work:

Model Name	**Linear equation**	**Reference**	**Eqn. No.**
	Isotherm models		
Langmuir	$\frac{Ce}{qe} = \frac{1}{qLKL} + \frac{Ce}{qL}$	Olasehinde et al, 2020	3
Freundlich	$\mathrm{Log\ qe} = \mathrm{Log\ KF} + \frac{1}{n}\log Ce$	,,	4
Tempkin	$\mathrm{qe} = \mathrm{Bln}A_T + \mathrm{BlnCe}$	,,	5
Dubinin-Radushkevich	$\ln q_e = \ln q_m - \beta(\varepsilon^2)$	Akpomie and Dawodu, 2014	6
	Kinetics Models		
Pseudo-first-order	$\mathrm{Log\ (qe - qt)} = \mathrm{Log\ qe} - \left(\frac{K1t}{2.303}\right)$	Fayoud et al, 2016	7
Pseudo-second-order	$\frac{t}{\mathrm{q}t} = \frac{1}{K2qe^2} + \frac{t}{q_e}$	,,	8
Intraparticle diffusion	$q_t = K_{dif}.t^{1/2} + C$	Akpomie and Dawodu, 2014	9
Elovich	$q_t = \left[\frac{1}{\beta}\right]\ln(\alpha\beta) + \left[\frac{1}{\beta}\right]lnt$	,,	10

3.0 Results and Discussion

3.1 Instrumental Characterization

3.1.1 Fourier Transform Infrared (FTIR) Analysis

FTIR spectrum for titanium dioxide nanoparticles as an adsorbent for the adsorption of methylene blue dye is depicted in figure 1. From the figure, it can be seen that there are observable peaks at 3900cm^{-1} - 3300cm^{-1}, 2400cm^{-1} to 2000cm^{-1}, 1800cm^{-1} to 1600cm^{-1}, 1500cm^{-1} to 1300cm^{-1} , 1200cm^{-1}to 1000cm^{-1}, 850cm^{-1}to 500cm^{-1}. It can be seen that the intensity of the peaks within 3650cm^{-1} to 3200cm^{-1} is very high, this can be attributed to the presence of hydroxyl (-OH) group stretching vibration (Khade et al, 2015; Niu et al, 2021). The band between 2300cm^{-1} to 2000cm^{-1} indicates C≡N and C≡C stretching vibration (Igwegbe et al, 2019). The stretching vibration around 1500cm^{-1} to 1300cm^{-1} can be attributed to the presence of C=C aromatic bond (Ani et al, 2019). The peak within 1350cm^{-1} to 1100cm^{-1}, can be attributed to CH_3 and C-O-C stretching vibration (Aravind et al, 2021; Elhady et al, 2024; Mustapha et al, 2020). Another band within 900cm^{-1}and 700cm^{-1} can be attributed to Ti-O-Ti stretching bond (Irshad et al, 2021; Yu et al, 2020).

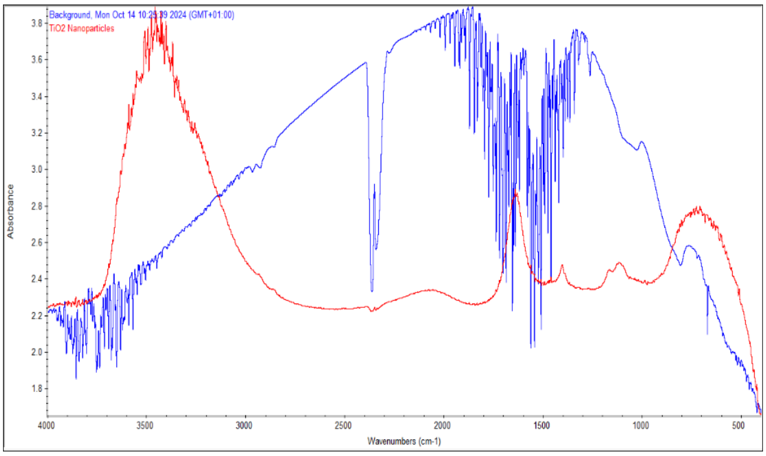
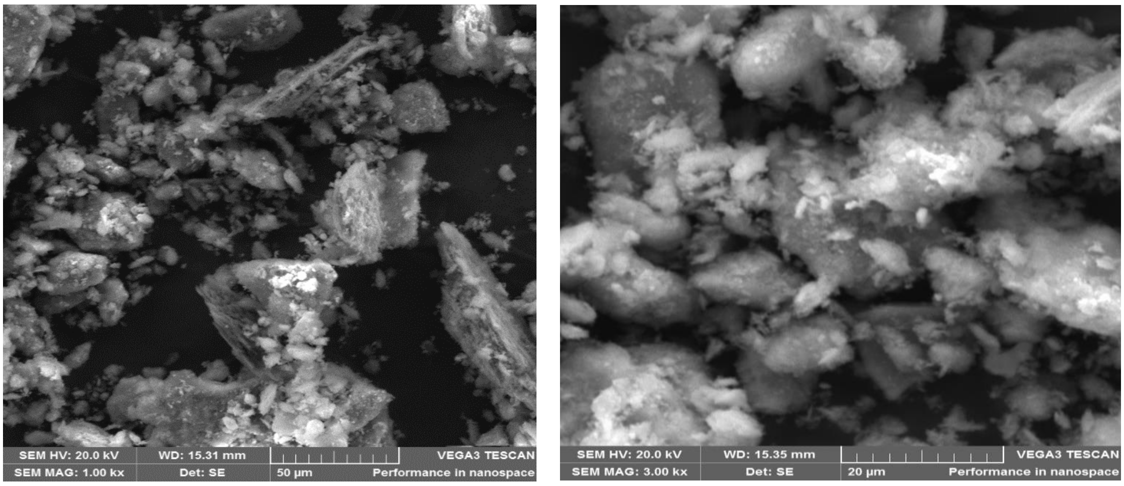


Figure 1: FTIR spectrum for Titanium dioxide nanoparticles

3.1.2 Scanning Electron Microscope Analysis (SEM)

SEM micrographs of the synthesized TiO_2 nanoparticles at different magnification of 1.00kx and 3.00kx is depicted in figures 2 a and b respectively. From the SEM morphology, it can be observed that the adsorbent exhibited porous and irregular structure which provided adequate active sites where the MBD were attached (Tijani et al, 2019). The SEM morphology of Titanium dioxide nanoparticles show high porosity of the adsorbent and presence of carbon which might be as a result of high calcination temperature. The coarse nature and irregular shape of the nanoadsorbent is revealed in the SEM images.



3.1.3 XRD Analysis

The XRD spectrum of TiO_2 nanoparticles is presented in figure 3. The spectrum reveals very few strong and sharp positions. Iron titanium Oxide is visible at 2ϴ of 40.42°, 48.86°, 52.52°, 62.21° with reference code of 01-075-0402 and scale factor of 0.365. Titanium oxide is visible at 2ϴ of 62.21° and 67.27° with reference code of 01-073-1117 and scale factor of 0.183. XRD sharp peaks of 48.86 (200), 52.52 (105), 62.21 (204), 67.27 (116) correspond to the tetragonal body-centred cubic structure of the nanoadsorbent (Irshad et al, 2021; Khade et al, 2015; Sugapriya et al, 2015). The sharp peaks at 40.42 (111), 52.32 (110) corresponding to the face centred cubic (FCC) of the crystalline titanium dioxide nanoparticles (Mustapha et al, 2020). Mustapha et al, 2020 also reported the presence of phase mixture (rutile and anatase) in the titanium dioxide nanoparticles calcined at 500°C. The XRD peaks are affected by calcination temperature. Since the titanium dioxide is calcined at temperature below 600°C, it indicates that it exists in rutile and anatase phases. Similar report was given by Jabbar et al, 2025 in the XRD result of the TiO_2 nanoparticles they synthesized with $TiCl_4$ using sol-gel method at calcination temperature of 450°C and 900°C. The peak positions exhibited by titanium dioxide nanoparticle indicate the crystalline structure of the nanoadsorbent (Irshad et al, 2021). Nevertheless, the adsorbent is not 100% pure as little impurity was observed.

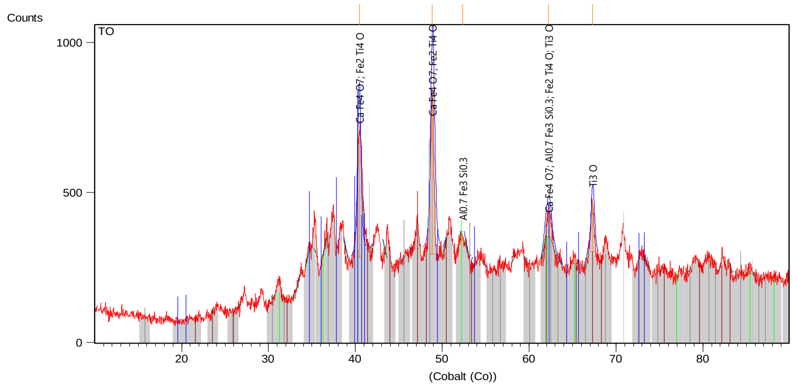


Figure 3: XRD for Titanium dioxide nanoparticles.

3.2 Influence of Process variables

3.2.1 Influence of Dosage

Influence of adsorbent dose is of paramount importance in the determination of the percentage removal (%R) of the adsorbent for the adsorbate (Aningo et al, 2017). It is also an important factor that affects the percentage adsorption of the methylene blue dye onto Titanium dioxide nanoparticles. The influence of adsorbent dose for the adsorption of methylene blue dye using titanium nanoparticles are presented in figure 7. It can be observed that as the adsorbent dose increased from 0.01g to 0.10g the percentage adsorption of TiO_2 nanoparticles for the adsorption of methylene blue dye increased from 20% to72%. Akpomie et al, 2017 reported a similar result in their work "Attenuation of methylene blue from aqua-media on acid activated montmorillonite of Nigerian Origin". The increase in the percentage adsorption can be accredited to corresponding increase in surface area since there will be more active binding sites on the surface of the adsorbent with increase in adsorbent dose (Akpomie et al, 2017).

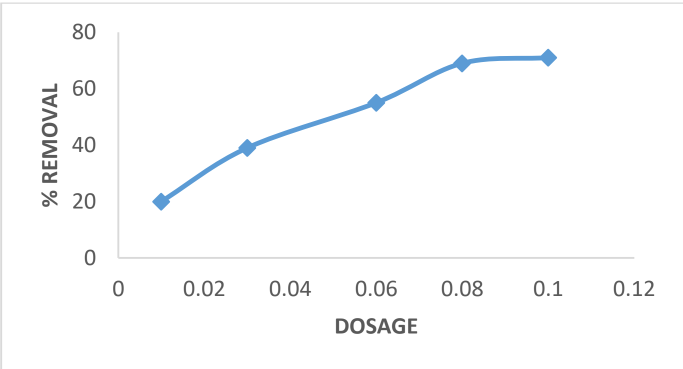


Figure 5: Percentage Removal versus Agitation time

3.2.3 Influence of initial concentration

Influence of initial concentration is another very important parameter to investigate during adsorption studies (Akpomie et al, 2017). In this work, five different concentrations were used to study the effect of initial methylene blue dye concentration on the adsorption of the dye using titanium dioxide nanoparticles. Figure 10 shows the percentage adsorption versus initial concentration for the adsorption of methylene blue dye using titanium dioxide nanoparticles. As the initial concentration is increasing the percentage adsorption is decreasing while the adsorption capacity was increasing. This is due to the fact that at higher concentration, the ratio of the initial number of MB dye molecules attached to the surface of the adsorbents was very low compared to the number of available molecules, which result in low percentage adsorption. At lower dye concentration, surface molecules get completely adsorbed on the dye molecules and result in higher percentage adsorption (Lincy et al, 2020). In order words, at higher
concentration there are more of the particles of the methylene blue dye in the solution that need to be placed on the surface of the adsorbent (Chime et el, 2016).

The decrease in percentage adsorption with an increase in the initial concentration of the methylene blue dye was due to the fact that at lower concentration the actives sites of the adsorbents were still available and can be occupied by the dye molecules but as the concentration is being increased from 150mg/l to 300mg/l the active sites of the adsorbents became more saturated thereby causing less adsorption of the adsorbate. According to Rosly et al 2022, the decrease in the rate of methylene blue dye adsorption may be as a result of the battling of the dye molecules for the active sites of the adsorbents which led to the reduction of the adsorption capacity with increase in initial dye concentration.

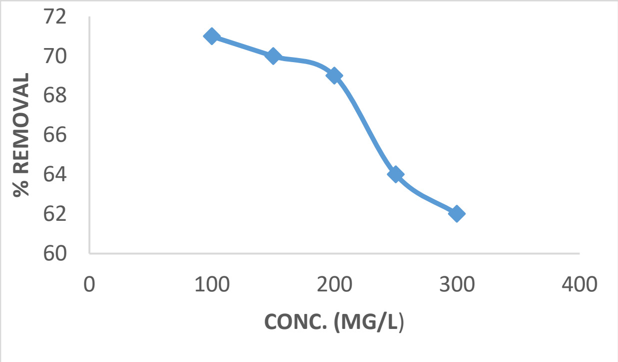


Figure 6: Percentage Removal versus Initial Concentration

3.3 Adsorption isotherm models

In every adsorption process, isotherm models are of paramount importance to study the relationship between the amount of adsorbate and its equilibrium concentration in solution (Igwegbe et al, 2019). In this study, the four isotherm models used are Langmuir, Freundlich, Tempkin and Dubinin-Radushkevich isotherm models. These models are the most frequently used adsorption isotherm models.

Table 2: Adsorption Isotherm Models for MBD onto titanium dioxide nanoparticle at 30°C

Langmuir isotherm Model			Freundlich Isotherm Model			Tempkin Isotherm Model			Dubinin-Raduchkevich Isotherm model		
q_L (mg/g)	**K_L (L/mg)**	**R^2**	**K_F (mg/g) $(mg/l)^{1/n}$**	**N**	**R^2**	**B (mg/g)**	**A(L/g)**	**R^2**	**q_m(mg /g)**	**β (mol^2/kj^2)**	**R^2**
0.4129	3.713	0.955	0.410	1.89	0.925	25997	1.00	0.968	0.3290	$3.0x10^{-8}$	0.952

3.3.1 Langmuir Isotherm Models

Langmuir isotherm parameters q_L(mg/g) and K_L (L/mg) are the monolayer adsorption capacity of the adsorbents and the adsorption constant respectively (Akpomie and Dawodu, 2016). The two parameters were determined from the slope and intercept of the plot of Ce/qe versus Ce. From table 3, it can be observed that the q_L and K_L values for the adsorption of MBD using titanium dioxide nanoparticles are 0.413 and 3.713 respectively. The correlation coefficient (R^2) values which is 0.9554 implies that the adsorption of methylene blue dye using titanium dioxide nanoparticles gave a good fit to the Langmuir adsorption isotherm model. The dimensionless separation factor (RL) explains whether the isotherm models are favorable (0<RL<1), unfavourable (RL > 1), linear (RL =1) or irreversible (RL=0) (Igwegbe et al, 2019). For the adsorption of methylene blue dye using titanium dioxide nanoparticles, the R_L values are within the range of 0.001 to 0.009. Langmuir isotherm model is favorable for the adsorption process since 0 < 0.009 <1 (Fayoud et al, 2016). In order words, it signifies that the adsorbent used in this investigation is viable for the uptake of methylene blue dye.

3.3.2 Freundlich Isotherm model:

Freundlich isotherm model has to do with multilayer adsorption and adsorption on heterogeneous surface. It also defines the exponential distribution of active sites of the surface (Fahdil et al, 2018).

Freundlich isotherm parameters K_F and n were determined from the slope and intercept of a plot of log qe against log Ce. K_F is the adsorption capacity while n is the adsorption intensity.

Table 3 shows the parameters for the adsorption isotherm models for the adsorption of methylene blue dye onto titanium dioxide nanoparticles. From the table, 0.410 and 1.89 are the K_F and n values respectively. Since the values of n fall between 1 and 10, it implies that the adsorption is a favorable adsorption (Fayoud et al, 2016). The regression coefficient value (R^2) for titanium dioxide nanoparticles is 0.925, which is well fitted to Freundlich isotherm model indicating a multilayer heterogeneous adsorption process.

3.3.3 Tempkin Isotherm Model:

Tempkin isotherm model parameters were calculated from a plot of qe versus ln Ce. B (mg/g) (isotherm constant) was calculated from the slope and it's in relationship with heat of adsorption. A (L/g) is related to the maximum binding energy (Olasehinde et al, 2020). Heat of adsorption B(mg/g) was gotten to be 25997 while the equilibrium binding constant A (L/g) was gotten to be 1. The regression coefficient which is used as the basis to determine a good fit to the model is given as 0.968 for the adsorption process being discussed here. From the values, it can be observed that the adsorption of MBD onto titanium dioxide nanoparticles fitted well to Tempkin isotherm model.

3.3.4 Dubinin-RadushKevich isotherm model

Unlike Freundlich isotherm model, Dubinin-Radsushkevich isotherm model does not assume a heterogeneous process (Akpomie et al, 2017). From the model equation, a plot of ln qe versus ε^2 gave straight line graph from which the constants qm (mg/g) and β (mol^2/j^2) were calculated from the intercept and slope respectively. q_m (mg/g) is the theoretical saturation capacity while β (mol^2/j^2) is the coefficient related to mean free energy of adsorption (Akpomie et al, 2021). Table 3 presents the adsorption isotherm variables for Dubinin –Radushkevich isotherm model. From the table it can be observed that the values of qm (mg/g) is 0.3290 and the value of β is $3.0 x 10^{-8}$. The correlation coefficients (R^2) value is 0.952 which implies that the process is well fitted to the model. The mean adsorption energy E (Kj/mol) is given as (Elkhaleefa et al, 2021):

$$E = \frac{1}{(2\beta)^{0.5}} = \frac{1}{\sqrt{2\beta}} \qquad 11$$

This was calculated from this isotherm model. The immensity of the mean free energy determines whether the adsorption process is in line with physisorption, ion exchange or chemisorption (Olasehinde et al, 2020). Elkhaleefa et al, 2021 reported that "mean free energy" for physisorption (<40kj/mol) and that of chemisorption is (> 40kj/mol). The mean free energy for adsorption of MBD onto titanium dioxide nanoparticles is 40.82, this value of mean free energy indicate that the adsorption process is chemisorption.

3.4 Adsorption Kinetic Studies

In every adsorption process, kinetic studies are very important. This is because it provides us with the information needed to determine the adsorption mechanism and the design of the process (Akpomie et al, 2021). In this research work, four kinetic models were used to determine the kinetic parameters needed for the analysis and interpretation of the data. They are Pseudo-first-order, Pseudo-second-order, intraparticle and Elovich models.

Table 3. Kinetic model for MBD onto titanium dioxide nanoparticles at 30ºC

qe_{exp} (mg/g)	Pseudo-first-order Model	Pseudo-second-order Model	Intraparticle diffusion model	Elovich model
0.13	qe_{cal} = 0.0242 K_1 = 0.4719 R^2 = 0.2903	qe_{cal} = 0.1693 K_2 = 3.8417 R^2 = 0.9799	K_{diff} = 0.0503 C = 0.0156 R^2 = 0.980	β = 27.855 α = 1.777 R^2 = 0.976

3.4.1 Pseudo-first-order model

Pseudo-first-order presumes that the rate at which the adsorption sites are occupied is in commensurate with the number of vacant sites (Akpomie et al, 2021).

The kinetic parameters for the adsorption of methylene blue using titanium dioxide nanoparticles at 30ºC are depicted in table 4. From the table, it can be observed that the K_1 values is 0.4519. The experimental qe_{exp} value is 0.13 while the calculated (qe_{cal}) value is 0.0242, they are not concordant values. The correlation coefficient (R^2) value is 0.2903. These values did not fit well with the Pseudo-first-order since it is far below 1 (Elkhaleefa et al, 2021).

3.4.2 Pseudo-second-order model:

Pseudo-second-order model presume that the rate at which the adsorption sites are occupied during adsorption process is equivalent to the square of the number of sites that were unoccupied (Akpomie and Dawodu, 2016).

Pseudo-second-order variables qe and K_2 were determined from the slope and intercept of a plot of t/qt versus t. The values of the parameters are depicted in table 4.

The correlation coefficient (R^2) value is close to 1 which implies a good fit for the adsorption of methylene blue dye onto titanium dioxide nanoparticles for this model. There is an agreement between the qe_{exp} and qe_{cal} since they are in a very close range. Since the correlation coefficient (R^2) of Pseudo-second-order model is greater that of Pseudo-first-order model, this indicates that the adsorption process is chemisorption in nature (Igwegbe et al, 2016).

3.4.3 Intraparticle diffusion model

Intraparticle diffusion model is one of the kinetic models used to analyze this work. It provides us with the data that helps us to study the mechanism that is involved in the adsorption process (Fayoud et al, 2016).

Intraparticle diffusion model parameters were determined from a plot of qt versus $t^{1/2}$. K_{dif} and C were determined using the slope and intercept of the graph respectively. K_{dif} ($mg/gmin^{1/2}$) is the intraparticle diffusion rate constant and C is the thickness of the boundary layer constant. From table 4, it can be observed that the correlation coefficients (R^2) value is 0.9894. This implies that there is presence of intraparticle diffusion in this adsorption process.

Intraparticle diffusion is the sole rate controlling step if the plots are straight line graphs that passes through the origin (C = 0) (Olasehinde et al, 2020). Since the plot in this work did not pass through the origin it entails that the model is not the sole rate controlling step. The K_{dif} value is 0.0503, since this value is very low, it implies that the assumption has been confirmed. The value of C (intercept) is 0.0156 for the adsorption processes and this signifies the presence of a boundary layer effect and also implies a surface phenomenon like mass transfer or liquid film diffusion in the adsorption process (Akpomie et al, 2017).

3.4.4 Elovich kinetic model

To determine the level of fitness of the experimental data to the Elovich kinetic model, a plot of qt versus ln t gave straight line graph. Elovich model parameters are presented in table 4. The constant β (g/mg) which correspond with the surface coverage and activation energy of the system was calculated from the slope of the graph and the value is 27.855. The initial sorption rate (α (mg/gmin) of the system which was determined from the intercept of the graph is 1.775 (Onoh et al, 2020).

The correlation coefficient (R^2) value is 0.9761. This indicates a good fit of the adsorption processes to Elovich kinetic model.

4.0 Conclusion

This investigation focuses on the synthesis, characterization and adsorption of methylene blue dye using titanium dioxide nanoparticles. The titanium dioxide nanoparticles used was synthesized with titanium (iv) isopropoxide using sol-gel method. The titanium dioxide powder produced during the synthesis was calcined at 500^oC to produce crystalline anatase titanium dioxide nanoparticles as confirmed by the XRD analysis. Characterization of the nanoadsorbent was carried out using SEM, FTIR and XRD. The FTIR result shows that the functional groups present in the prepared nanoparticles contributed to the high adsorptive capacity of the adsorbent. SEM image proved the coarse and high pore spaces in the adsorbent. Influence of process parameters such as dosage, time and initial dye concentration were investigated using batch adsorption process. The results obtained from the effect of these factors proved that the adsorption of MBD using TiO_2 nanoparticles is dependent on these factors. The adsorption process fitted well to the four isotherm models used in this work. Kinetic experiments did not follow the Pseudo-first-order model but followed the other three models used in this investigation.

5.0 Recommendation

I recommend that further research work should be carried out to study the effect of calcination and adsorption temperatures, solution pH and particles sizes. This adsorbent is recommended for textile industries and other industries that make use of dye but the factors should be increased for optimum adsorption process. Further research work should be conducted on the optimization of the process parameters for the adsorption f methylene blue dye using titanium dioxide nanoparticles synthesized using sol-gel method.

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Nomenclature

Ce= Equilibrium concentration, mg/l;

Co = Initial concentration, mg/l;

Ct = Concentration at time t, mg/l;

A= Temkpin constant, l/g;

q_e = Adsorption capacity at equilibrium, mg/g;

qe_{exp} = Experimental adsorption capacity, mg/g;

qe_{cal} = Calculated adsorption capacity, mg/g;

qm = Maximum adsorption capacity, mg/g;

K_1 = Pseudo-first-order constant;

K_2 = Pseudo-second-order constant;

K_F = Freundlich constant, (mg/g)

$(mg/l)^{1/n}$;

K_L = Langmuir constant, l/mg;

R_L = Dimensionless separation factor.