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Comparative Study for Adsorptive Removal of Coralene Blue BGFS Dye from Aqueous Solution by MgO and Fe_2O_3 as an Adsorbent

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Abstract
Textile industries represent biggest impact on the environment due to high water consumption and waste water discharge as government control water pollution by setting strength regulation for waste water discharge, removal of color from waste water becomes more and more essential and attractive. Adsorption technology is very efficient in treatment of textile effluent. In this paper comparison of adsorption phenomena of textile dye Anthraquinone blue onto two different adsorbents MgO nano powder and Fe_2O_3 amorphous powder has been studied for removal of said dye from aqueous solutions. The adsorption of Anthraquinone blue on adsorbents occurs by studying the effects of adsorbent amount, dye concentration, contact time and pH of solution. All results found that MgO nano powder and Fe_2O_3 provide a fairly high dye adsorption capacity, which combined with their fulfilment of pollution control board's standards, lack of pollution, lower environmental hazard and low-cost makes them promising for future applications. The present work also provides information on optimum value of different operating parameter for dye removal by two adsorbent.

Keywords: Textile industries, dye removal, MgO nano powder, Fe_2O_3 amorphous powder, Adsorption, Anthraquinone blue dye.

I. Introduction:

Dyes are synthetic organic compounds capable of colouring fabrics, typically derived from coal tar and petroleum based products. Dye consists of two main groups of compounds, chromophores (responsible for colour of the dye) and auxochromes (responsible for intensity of the color). According to the AATCC (American Association of Textile Chemists and Colorists), currently more than 10,000 various types of dyes are synthesized and available in the world. [1–3]. Dyes are classified according to the chemical structure and type of application. Based on chromophores, 20–30 different groups of dyes can be classified, with azo, anthraquinone, phthalocyanine and triarylmethane etc. Azo (around 70%) and anthraquinone (around 15%) compose the largest classes of dyes. Many industries, such as dyestuffs, textile, paper and plastics, use dyes to colour their products; as a result, these industries produce coloured wastewater as an unavoidable by-product [4, 6, 12, 14, 24, 25, 26, 29].

Among various industries, the textile industry ranks first in the usage of dyes for colouration of the fibers. The textile sector alone consumes about 60% of total dye production for coloration of various fabrics and out of it, it is estimated that around 10–15% of dyes are wasted into the environment upon completion of their use in the dyeing unit which generates a strongly coloured wastewater, typically with a concentration in the range of 10–200 ppm or mg/L [24,26]. Colour in the effluent is one of the most noticeable indicators of water pollution and the discharge of highly coloured synthetic dye effluents is aesthetically very unpleasing and can damages the receiving water body by hindering the penetration of light. Moreover dyes are stable, recalcitrant, colorant, and even potentially carcinogenic and toxic [9, 10], their release into the environment creates serious environmental, aesthetical and health problems. Thus, industrial dye-laden effluents are an increasingly major concern and need to be effectively treated before being discharged into the environment in order to prevent these potential hazards [4, 12, 14, 16, 21].

Different methods are available for the removal of dyes from wastewater. These include physiochemical treatment, biological treatment, combined chemical and biochemical processes, chemical oxidation, adsorption, coagulation, filtration and membrane treatments; each of these has their own specific advantages and disadvantages. With the reference of available abundant literature review adsorption is a well-known separation process and is widely used to remove certain classes of chemical pollutants from water, especially those which are practically unaffected by conventional biological wastewater treatments. It has been found to be superior to other techniques in terms of initial cost,

flexibility and simplicity of design, ease of operation and insensitivity to toxic pollutants. In addition, this technique can handle fairly large flow rates, producing a high-quality effluent that does not result in the formation of harmful substances. Different natural and synthetic adsorbents have been evaluated for the removal of dyes from textile effluent. Among these materials, activated carbon is one of the most widely studied and used adsorbents for environmental pollution control. The main disadvantage of activated carbon is its high production and treatment costs [4, 6, 12, 14, 30]. Thus, many researchers throughout the world have focused their efforts on optimizing adsorption and developing novel alternative adsorbents with high adsorptive capacity and low cost. In this regard, much attention has recently been paid to powder technology. Nano-materials have large specific surface areas, and thus a large fraction of atoms are available for chemical reaction [7, 15, 20].

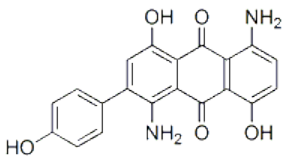
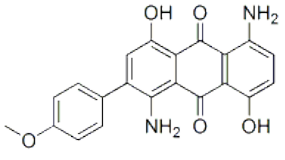
The present study focuses on the application of MgO nanoparticles and Fe_2O_3 amorphous powder as an adsorbent for the removal of Anthraquinone blue dye from aqueous solution. The effect of different variables, including dosage of adsorbent, concentration of dye, pH of the liquid and contact time, on removal of the model dyes was evaluated [3,7, 13, 15, 18, 19,20 ,22, 23, 25 ,27, 31].

II. Materials and method

2.1- Chemicals

All the chemicals and reagents used were of analytical grade. Magnesium chloride hexahydrate ($MgCl_2 \cdot 6H_2O$) and Potassium hydroxide pellets (KOH were procured from Finar Chemicals Pvt. Ltd. (India). Anthraquinone blue dye was obtained from Colourtex, Surat (India) and was used as received without further purification. The available details of these dye is listed in Table 1. Double distilled water was used to prepare all the solutions. The stock solution was prepared by dissolving 1 g of the dye in 1 L of double distilled water to obtain working solutions of varying concentrations for further experiments. The pH was adjusted using 0.1 N H_2SO_4 and NaOH.

Table 1: Dye detail

Title	Dye	
Commercial name of dye	Coralene blue BGFS	
Chemical name of dye	Mix. Of 1,5-diamino-4,8-dihydroxy(p-hydroxy-phenyl)anthraquinone & 1,5-diamino-4,8-dihydroxy(p-methoxy-phenyl)anthraquinone	
Chemical formula	$C_{20}H_{14}N_2O_5$&$C_{21}H_{16}N_2O_5$	
Class	Anthraquinone	
C.I number	C.I disperse blue 73	
Molecular weight	362 & 376	
Molecular structure		

2.2- Adsorbents

(a). Magnesium oxide (MgO) nanoparticles MgO nanocrystallites were synthesised via sol gel method as reported in literature [20] The synthesised MgO was used as an adsorbent for removal of dye.

(b). Ferrous Oxide (Fe_2O_3) amorphous powder:

Powdered Fe_2O_3, a brown powder, were recently used in several applications like adsorption, magnetic storage media, solar energy transformation, electronics, Ferro fluids and catalysis. It is used as an effective adsorbent in the dye waste water treatment. For these experiments Ferrous Oxide amorphous powder of 99% pure was purchased from Merck chemicals Pvt. Ltd, Mumbai (India). The technique

was found to be very useful and cost effective for better removal of dye.

2.3- Adsorption experiments

The adsorption experiments were carried out as batch tests in magnetic stirrer. In a batch test, 100 mL dye solution of desired concentration was prepared in 250 mL glass flask by suitable dilution of the stock solution and its desired pH was adjusted. Then known amount of MgO nanoparticles was added and the resulting suspension was kept under constant stirring for predefined time. After stirring, the suspension was centrifuged and the supernatant was analyzed for the dye removal capacity. Same procedure was followed for Fe_2O_3 adsorbent.

2.4- Analysis

Suspension was centrifuged at 3500 rpm in centrifuger (Centrific, Model 228, Fisher Scientific). Samples of solutions before and after treatment were analyzed for the dyes using a UV 1100-Spectrophotometer (vortex) at their maximum absorption wavelengths (figure 1). The dye concentrations were calculated from the standard calibration curve obtained from standard dye solutions. The pH of the liquid samples was measured using a pH meter (Sense Ion 378, Hack).

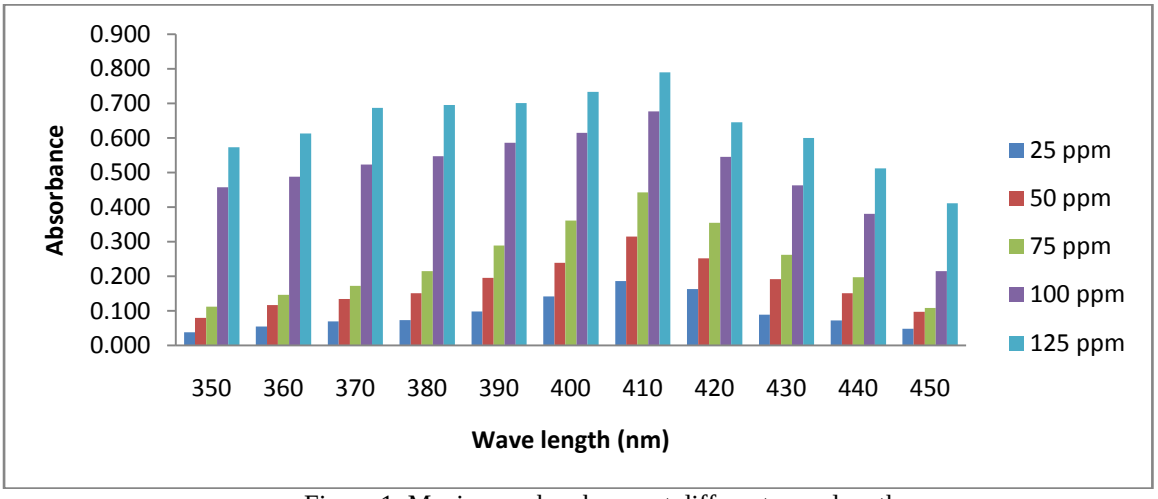


Figure 1: Maximum absorbance at different wavelength

III. Result and discussion

3.1. Comparison of dye removal capacity of two adsorbent

Efficiency of the adsorbents was investigated for removal of Anthraquinone blue from liquid solutions. The experiments were performed under different experimental conditions. Results are presented in the following section.

3.1.1- Effect of solution pH

Solution pH is an important parameter that affects adsorption of dye molecules. The effect of the initial pH of the solution on the anthraquinone blue adsorption onto adsorbents was assessed at different values, ranging from 2 to 12, with a stirring time of 65 min. The initial dye concentrations and dosage of each adsorbent were kept constant at 125 mg/L and 0.2 g per 100 mL dye solution, respectively, for all batch tests in this experiment. Fig. 2 presents the comparison of effect of the different initial solution pH on the dye removal efficiencies of MgO nano particles and Fe_2O_3 powder. As shown in Fig. 2, removal of dye decreased from 88 to 64% when the pH was increased from 2 to 9 for MgO powder. After increasing pH to 12, % dye removal also increases to 75%. Since the maximum removal of Anthraquinone blue dye is achieved at a pH of 2, acidic condition is favourable for MgO nanoparticles. It is also found that by using Fe_2O_3 powder maximum removal of 88% was achieved at pH 2. i.e acidic pH. While increasing pH up to 12, % dye removal decreased up to 25%. So, pH 2 is optimum pH for both adsorbents.

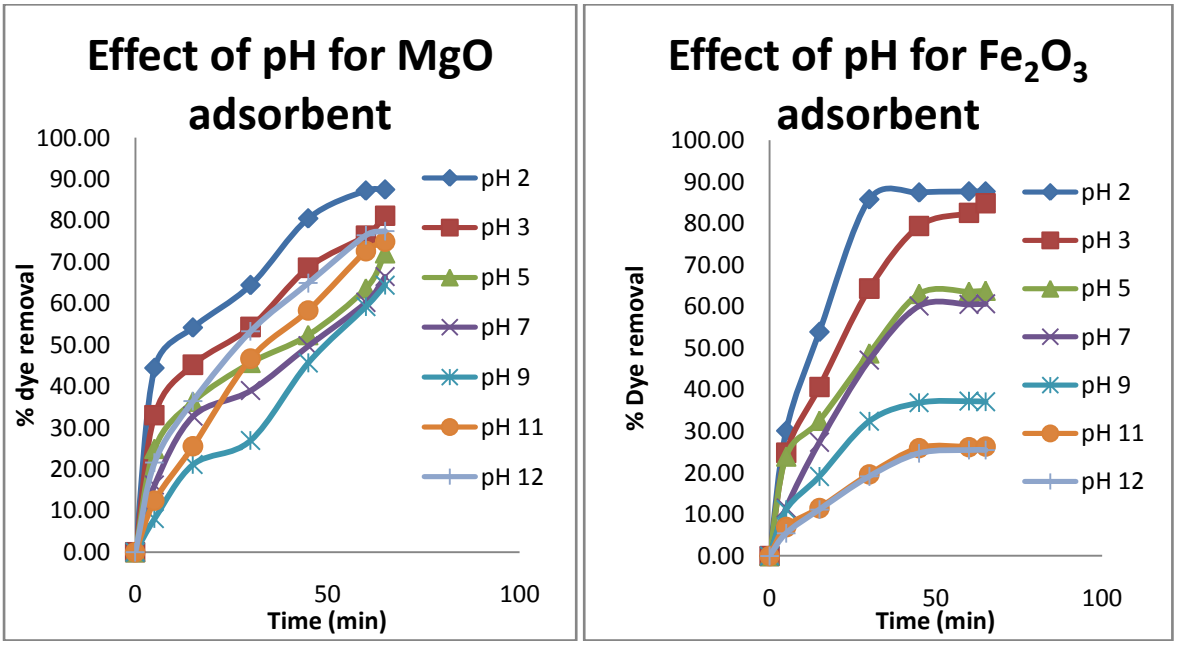
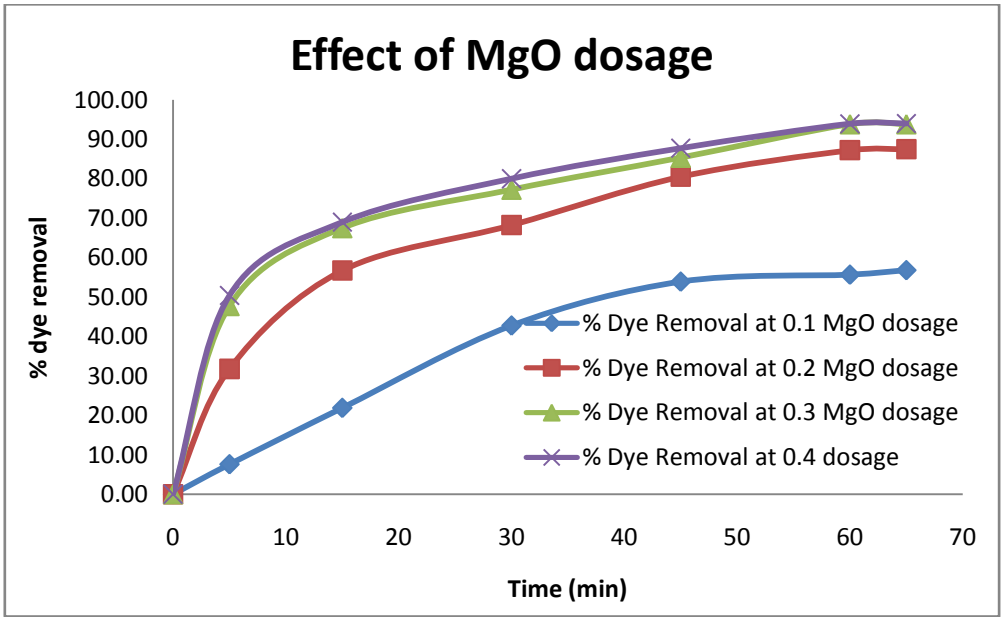


Figure 2: Comparison of effect of initial pH on dye removal capacity of both adsorbent

3.1.2. Effect of adsorbent dosage

The comparison of effect of adsorbent quantity on removal of Anthraquinone blue is represented in figure 3. For MgO powder adsorbent, behaviour was investigated in batch experiments by adding various amount of adsorbent in the range of 0.1–0.4 g powder into the flask containing 100 mL of dye solution. The initial dye concentrations and the pH of the solutions were fixed at 125 mg/L and 2, respectively, for all batch experiments. The suspension was then stirred for 65 min, after which time the solution was coagulated and settled and the supernatant was analyzed for the remaining dye. As indicated in figure 3, 88% of anthraquinone blue was removed at the initial dosage of 0.2 g, respectively. The removal of dye increased with increasing MgO dosage to 0.3 g and reached to over 94% for said dyes at this dosage. After that there is no significance change in % dye removal by increasing in MgO dosage to 0.4 g. This observation can be explained by that further increasing the adsorbent dose did not affect the removal of dye. Hence, the optimum dosage of nano-MgO powder for removing Anthraquinone blue dye was found to be 0.3 g.

While for Fe_2O_3 powder adsorbent, same experiment was carried out for various amount of adsorbent in the range of 0.2–0.35 g powder. The initial dye concentrations and the pH of the solutions were fixed same as 125 mg/L and pH 2. As presented in figure 3, 94% of Anthraquinone blue was removed at the initial dosage of 0.3 g, respectively. After that there is no significance change in % dye removal by increasing in Fe_2O_3 dosageg. Hence, the optimum dosage of Fe_2O_3 powder for removing Anthraquinone blue dye was found to be 0.3 g.



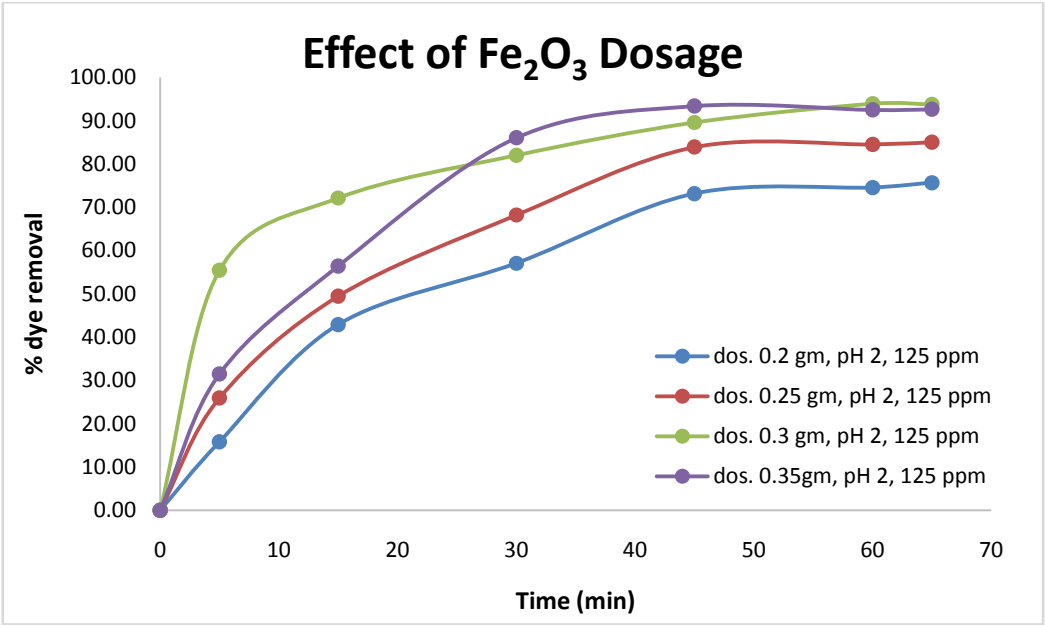


Figure 3: Comparison of effect of adsorbent dosage on dye removal capacity

3.1.3. Effect of contact time

The variation of adsorption capacity of MgO and Fe_2O_3 powder toward dye with time is depicted in figure 3, It is evident from the figure that the adsorption was rapid initially. Almost 50% dye removal is attained in first 5 min for optimum adsorbent dosage. And the dye removal percentage improved slowly with time. The initial rapid adsorption is due to the presence large number of active sites on the surface of adsorbents. Anthraquinone blue dye adsorption equilibrium was attained within 65 min of contact time with nearly 94 to 96% dye removal from the solution for MgO powder and 93 to 94% for Fe_2O_3 powder.

3.1.4. Effect of dye concentration

The initial dye concentration is another important variable that can affect the adsorption process. The effect of initial concentration of Anthraquinone blue dye between 25 and 125 mg/L was studied on their adsorption onto MgO powder under previously determined optimum conditions. The results, in terms of removal efficiency versus initial concentration of dye, are indicated in Fig. 4. According to Fig. 4(a), dye removal slightly decreased from around 96% at a concentration of 25 mg/L to 94% when the concentration was increased to 125 mg/L. Overall, we found that the prepared MgO powder had high adsorption affinities for anthraquinone blue, which are models of

anthraquinone class dyes. The adsorption capacity of MgO at the maximum investigated dye concentration was 25 mg/g for each of the tested dyes.

As per shown figure 4(b), while for Fe_2O_3 adsorbent, maximum % dye removal was achieved at 125 ppm dye concentration. By decreasing dye concentration from 125 to 50 ppm, % dye removal also decreases from 94% to 57%. By further decreasing initial dye concentration up to 25 ppm, % dye removal increasing from 57% to 76%.

3.1.5: Adsorption isotherms study

Isotherm parameters values for the MgO adsorbent are presented in Table 4. all isotherms were favourable but the data fits well with Temkin isotherms yielding high R^2 values, close to 1.

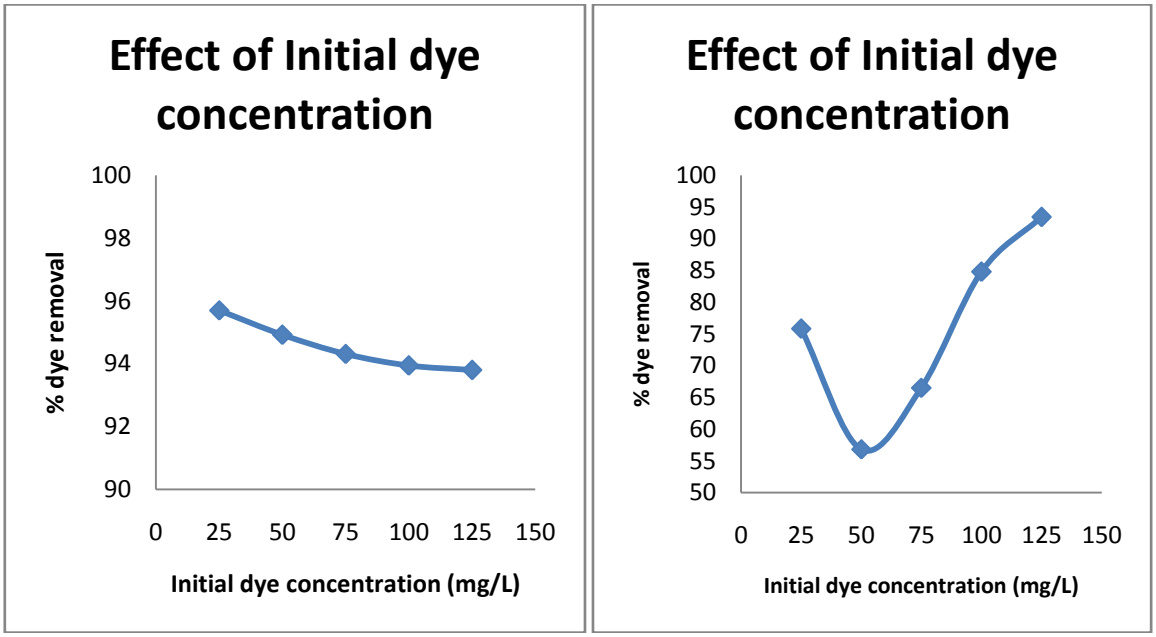


Figure 4. (a)For MgO Adsorbent, (b) For Fe_2O_3 Adsorbent

Table 2: Isotherm parameters values for MgO adsorbent

Adsorbent	**Dye Conc.**	**Langmuir**				**Freundlich**			**Temkin**		
		Qm	**K_L**	**R_L**	**R^2**	**N**	**K_F**	***R^2***	**A**	**b**	**R^2**
MgO	125ppm	26.31	0.294	0.026	0.393	4.184	72.747	***0.823***	3219.663	350.83	0.869
But adsorption fits with Temkin isotherms as its value of R^2 is higher											

Table 3: Isotherm parameters values for Fe_2O_3 adsorbent

Adsorbent	**Dye Conc.**	**Langmuir**				**Freundlich**			**Temkin**		
		Qm	**K_L**	**R_L**	**R^2**	**N**	**K_F**	***R^2***	**A**	**b**	**R^2**
Fe_2O_3	125ppm	25.707	0.165	0.046	0.643	3.706	88.872	***0.936***	6592.692	387.266	0.8875
From the graph, value of R^2 is higher of Freundlich isotherm. So, it is best fit.											

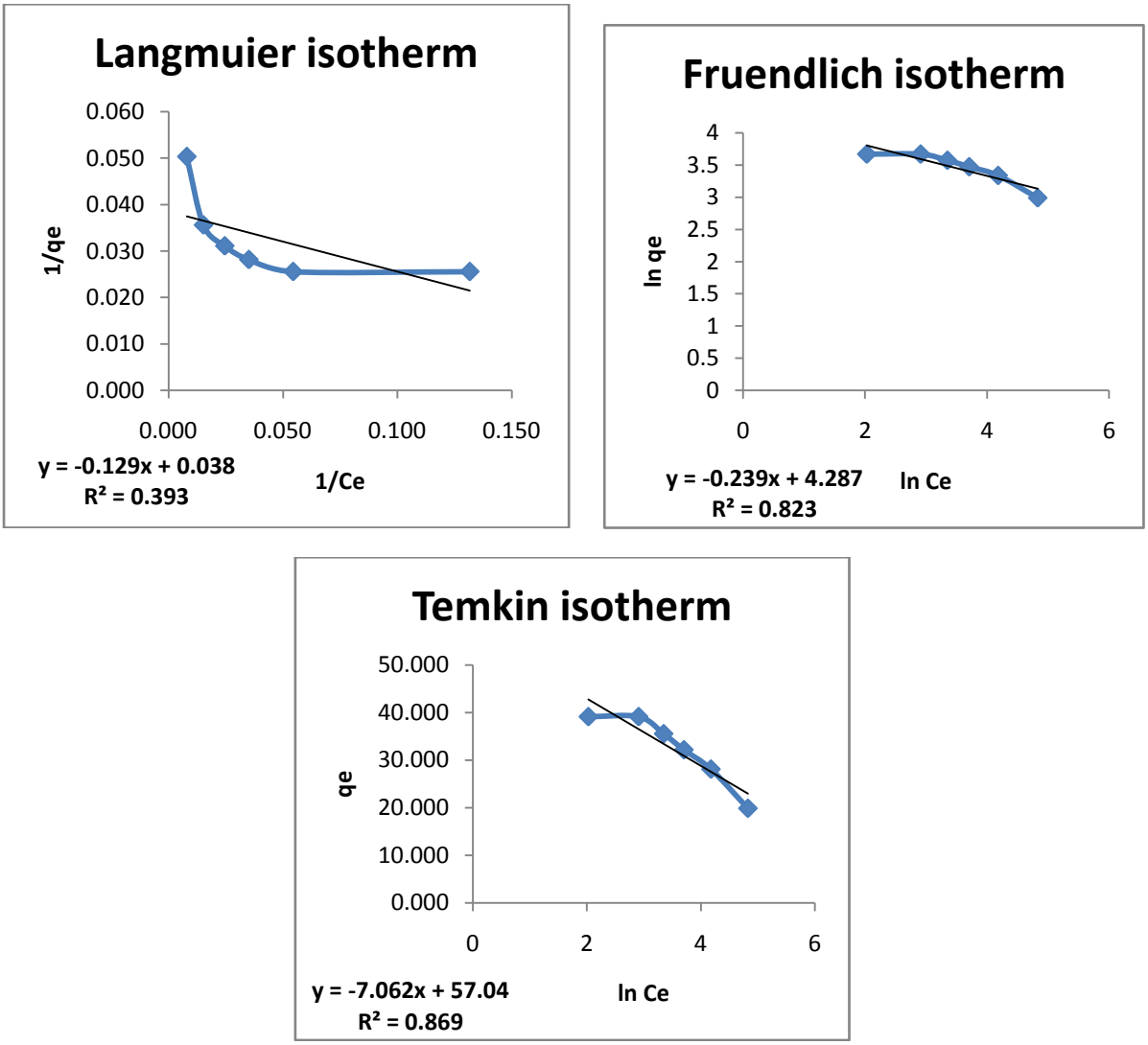
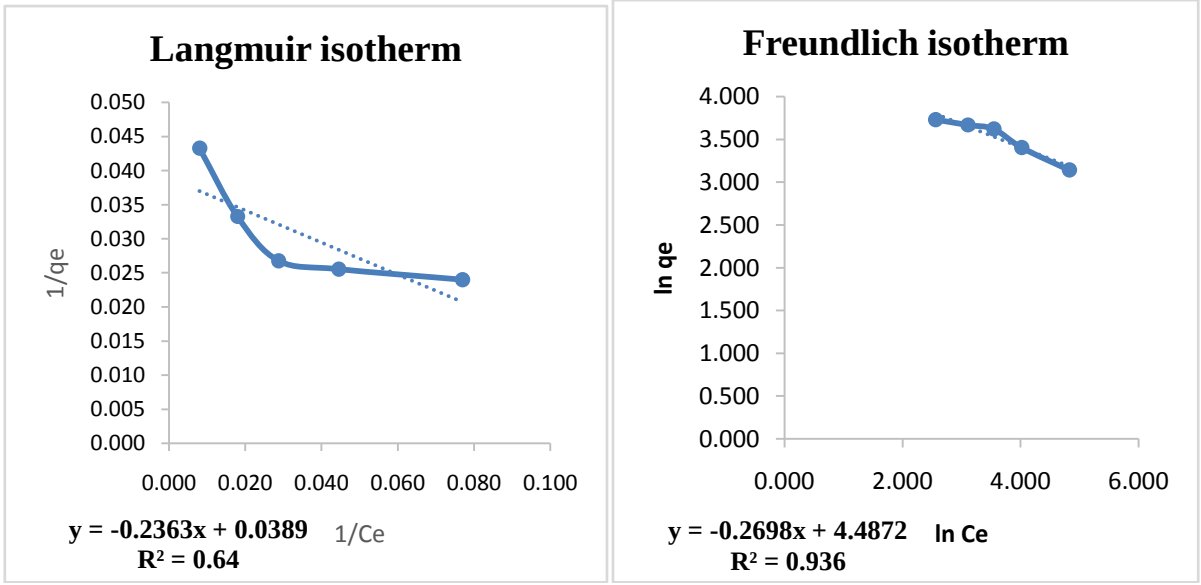


Figure 4: different Isotherms study for MgO adsorbent



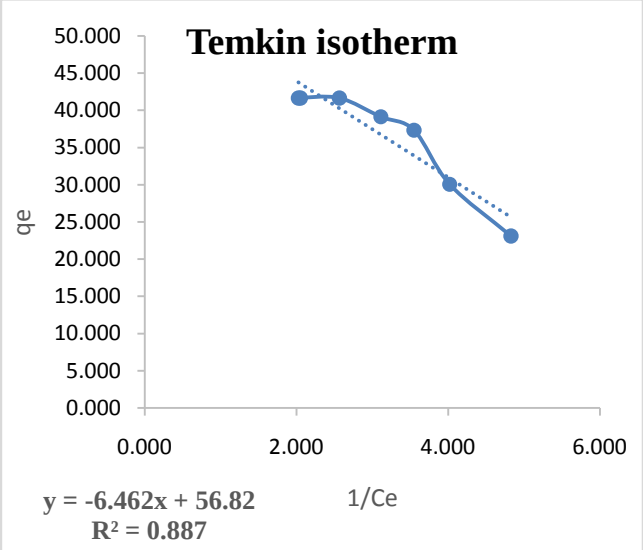


Figure 5: different Isotherms study for Fe_2O_3 adsorbent

3.1.6: Adsorption kinetics study:

The transient behaviour of the dye sorption process was analyzed by using the pseudo-first-order, pseudo-second-order, and intraparticle diffusion models. Application of a single kinetic model to sorption on solid adsorbents may be questionable because of the heterogeneity of adsorbent surfaces and diversity of adsorption phenomena [21].

3.1.6.1-Pseudo-first-order model

The pseudo-first-order kinetic model has been widely used to predict dye adsorption kinetics. The pseudo-first-order rate expression suggested originally by Lagergren based on solid capacity is expressed as follows.

$$dqt/dt = K_1 (qe - qt)$$

After integrating both side for boundary conditions of q=0 at t = 0 and qt = qe at t = te,

$$\ln (qe - qt) = \ln qe - (K_1 . t)/ 2.303$$

Values of qe and K_1 can be obtained from the slope and intercept of the plot ln (qe − qt) versus t.

3.1.6.2. Pseudo-second order model

Pseudo-second order model is expressed by the equation

$$dqt/dt = K_2 (qe - qt)^2$$

after integrating both side for boundary conditions of q=0 at t = 0 and qt = qe at t = te,

$$1/ (qe-qt) = 1/qe + K_2 . t$$

we can rearrange above eq.

$t/qt = 1/(K_2 . qe^2) + t/qe$

Values of qe and K_2 can be obtained from the slope and intercept of the plot t/qt versus t.

3.1.6.3. Intraparticle diffusion studies

It is necessary to identify the steps involved during adsorption in order to interpret the mechanism of adsorption. It is assumed that the adsorption process consists of several steps. Migration of the dye from the bulk of the solution to the sorbent surface, diffusion of the dye through the boundary layer, intraparticle diffusion, and adsorption of the dye on the internal sorbent surface. The intraparticle diffusion rate can be expressed in terms of the square root time. The mathematical dependence of qt versus $t^{0.5}$ is obtained if the sorption process is considered to be influenced by diffusion in the spherical particles and convective diffusion in the solution [21]. The root time dependence, the intraparticle diffusion model is defined as follows:

$$Qt = K_1 . t^{0.5} + C$$

The plot q versus $t^{0.5}$ is given by multiple linear regions representing the external mass transfer followed by intraparticle or pore diffusion

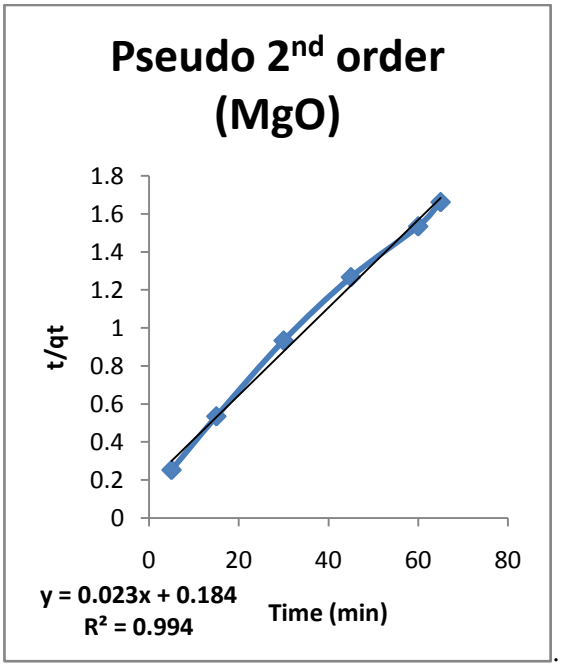
To describe the adsorption behaviour and rate, the data obtained from adsorption kinetic experiments were evaluated using pseudo first-and pseudo-second-order reaction rate models. Second order model was found to fit with the data very well. Plots of experimental results of the anthraquinone blue dye fitted to the selected adsorption models are shown in Fig. 6 and 7. The values of q_{exp}, q_{pre}, K_1, K_2 and the corresponding linear regression coefficient R^2 values are summarized in Table 4 and 5. As showing Table 4 and 5, higher values of R^2 were obtained for pseudo-second-order than for pseudo-first-order adsorption rate models and intraparticle diffusion, indicating that the adsorption rates of anthraquinone blue dye onto the MgO nanoparticles and Fe_2O_3 powder adsorbent can be more appropriately described using the pseudo-second order rate.

Table 4: Adsorption kinetic constants for MgO adsorbent

Adsorbent	Dye Conc.	Pseudo first Order kinetics			Pseudo second Order kinetics			Intraparticle Diffusion	
		K_1	Qe	R^2	K_2	Qe	R^2	K_I	R^2
MgO	125 ppm	0.052	16.51	0.966	0.0028	43.47	0.994	3.213	0.980

Table 5: Adsorption kinetic constants for MgO adsorbent

Adsorbent	Dye Conc.	Pseudo first Order kinetics			Pseudo second Order kinetics			Intraparticle Diffusion	
		K_1	Qe	R^2	K_2	Qe	R^2	K_I	R^2
Fe_2O_3	125 ppm	0.0516	23.71	0.986	0.004	45.249	0.997	3.3897	0.967



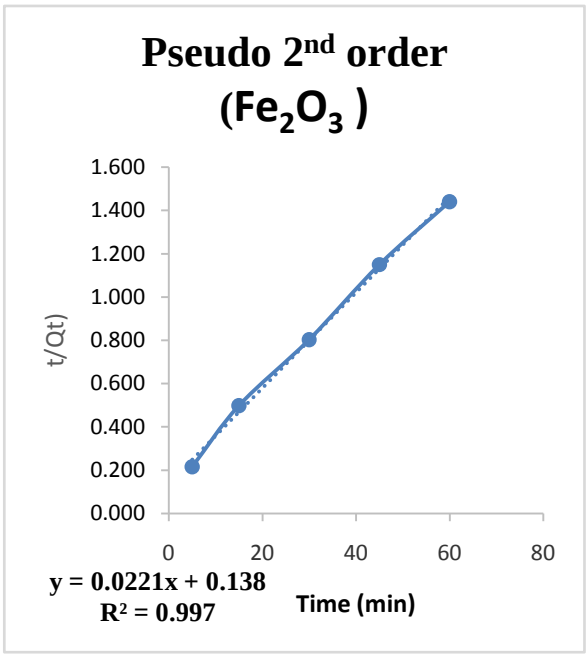


Figure 6: Pseudo second order kinetics for anthraquinone dye removal

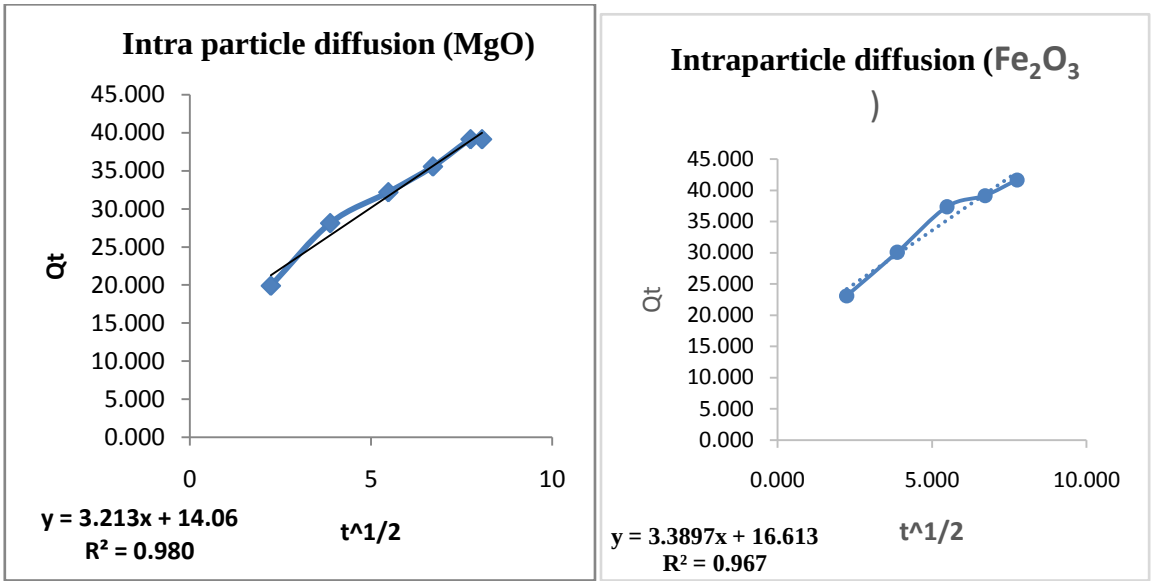


Figure 16: Intraparticle diffusion kinetics for anthraquinone dye removal

3.7- Result

From the above experimental observations, it can be noted:

1. For both adsorbent MgO and Fe_2O_3 powder, give best result at acidic pH range i.e. pH 2.
2. 0.3 g adsorbent dosage is optimum dosage for both adsorbent for 100 ml dye sollution. Both MgO and Fe_2O_3 adsorbents give 94% dye removal for 125 ppm dye concentration at 0.3 gm dosage.
3. For MgO powder adsorbent, time for reaching at equilibirium decrease with decreasing dye concentration. While for Fe_2O_3 adsorbent equillibium was achieved at 30 min for all concentration.
4. Adsorption equilibrium was attained 65 min of contact time with nearly 94 to 96% dye removal from the solution for MgO powder and 93 to 94% for Fe_2O_3 powder.
5. Almost 50% dye removal is attained in first 5 min for both adsorbent at optimum dosage.
6. For MgO adsorbent dye removal slightly decreased from around 96% at a concentration of 25 ppm to 94% when the concentration was increased to 125 ppm. While for Fe_2O_3 adsorbent, maximum % dye removal was achieved at 125 ppm dye concentration. By decreasing dye concentration from 125 to 50 ppm, % dye removal also decreases from 94% to 57%. And further decreasing initial dye concentration up to 25 ppm, % dye removal increasing from 57% to 76% .
7. The isotherm evaluations revealed that the Freundlich model attained better fits to the experimental equilibrium data for Fe_2O_3 adsorbent. While Temkin model attain better fits for MgO adsorbent.
8. Adsorption kinetic data followed a pseudo-second-order rate for both adsorbents.

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