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Removal of naphthol green B dye from polluted waters using hydrogen peroxide treated red mud

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ABSTRACT

The enhanced adsorption abilities of Hydrogen peroxide treated red mud are investigated for the removal of an anionic dye, Naphthol green B dye from polluted water. Various physicochemical parameters like pH, sorbent concentration, agitation time, temperature and initial concentration of the dye are optimized for the maximum removal of the dye from simulated waters. The nature of adsorption is analyzed using Langmuir, Freundlich, Temkin and Dubinin-Radushkevich isotherms and found that the adsorption is well described by Langmuir adsorption model with R^2 *: 0.9728;* $R_{L:}$ *0.0634 suggesting the monolayer formation onto the surface of the adsorbent and further, Temkin and Dubinin-Radushkevich isotherms reflect the 'physisorption' nature. On the analysis of kinetics of adsorption by Pseudo-first-order, pseudo-second-order, Bangham's pore diffusion and Elovich equations, it is revealed that adsorption follows pseudo-first-order kinetics. The thermodynamic studies reveal that adsorption of the dye onto the surface of the adsorbent, is an exothermic process. The developed procedures are successfully applied to the samples collected from the effluents of textile industries.*

Key words: Naphthol green B dye, water treatment, activated red mud, adsorption, applications.

INTRODUCTION

In the recent past, increasing research interest is being envisaged in finding methodologies for the removal of pollutants from water/waste waters based on un-conventional methods because the traditional methods are costly and complicated and moreover, they have one or the other disadvantage. Our research group is pursuing these aspects of un-conventional methods and successfully has developed methods based on the bio-sorbents derived from plant materials for the extraction of Chromium (VI) [1-4], Zinc [5, 6], Aluminum (III) [7-10], Fluoride [11-17], Nitrite [18, 19], Nitrates [20], Ammonia [21-23], Phosphate [24, 25] and some Dyes [26-31] and some heavy metal ions evoking the mechanism of oxidation ponds [32].

Red mud, a waste product of Aluminum industries is interesting the researchers as it possesses good sorption characteristics and it is being investigated as adsorbent for the removal of pollutants from waters. Some successful investigations for the removal of As (III, V) (33,34), Pb (II) (35,36), Cr(VI) (37) , Co(II) (38), Zn (II) (39,40), Phosphate (41-43), Nitrate (44) and Dyes (45-47) from waste waters using red mud as adsorbent have been reported in the literature.

In the present work, Hydrogen peroxide treated red mud is used as adsorbent in the removal of highly pollutive anionic dye, Napthol green B dye. This dye is the Sodium salt of Naphthol Green Y and is soluble in water. It is used in various dying industries for staining nylon, wool, papers etc. This dye is not bio-degradable and its presence in water is highly undesirable. Its presence in the water bodies imparts color to the waters and thereby affects the photosynthesis and other metabolisms resulting the stress on biota and aquatic life. [48-50]. Hence, the removal of this dye from polluted waters assumes significance

In the present investigation, the removal of Naphthol green B dye is studied using Hydrogen peroxide treated Red mud as adsorbent. The sorption characteristics of the adsorbent are optimized for the maximum removal of the dye.

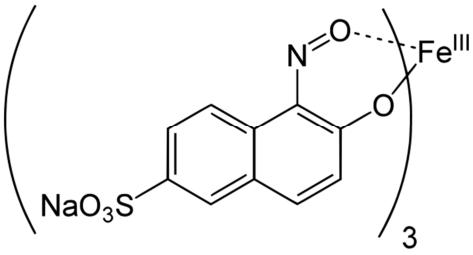


Figure 1: Napthol gree B dye structure

MATERIALS AND METHODS

2.1. Adsorbent:

Table No:1 Chemical properties of red mud	
PARAMETER	**RESULT (%)**
Alumina as Al_2O_3	15.47
Iron as Fe_2O_3	58.78
Silica as SiO_2	6.58
Titanium as TiO_2	4.39
Soda as Na_2O	3.63
Calcium as CaO	1.49
Phosphorus as P_2O_5	0.159
Vanadium as V_2O_5	0.110
LOI (105-1000^{0}C)	7.22

Red mud collected from Utkal Alumina, Rayagada, Orissa was washed with distilled water repeatedly until the filtrate shown neutral pH, dried at 105^{o}C for two hours and meshed to 75 µ. Then to a suitable amount of red mud, 6% H_2O_2 was added in 1:2 ratio (w/w), stirred well and digested for 24 hrs at room temperature (30^{o}C). Then the sample was filtered, washed with distilled water until the filtrate shown neutral pH and dried at 105^{o}C for two hours. Thus obtained sample was used in this work. The composition of the active red mud was analyzed adopting the standard procedures available in literature before treatment and results were presented in Table 1.

2.2 Reagents and Chemicals:

A.R. grade chemicals purchased from Merck. India Pvt. Ltd and Sd. Fine Chemicals were used and the solutions were prepared with double distilled water. Stock solution of Naphthol green B dye of concentration 1000 ppm was prepared and the solution was suitably diluted as per the need.

2.3: Method: Batch methods of extraction were adopted [51-53]. Definite quantities of treated red mud were added to 100 ml of Naphthol green B dye solution (100 ppm) taken into 250 ml conical flask, pH was adjusted to a desired value and resulting solution was subjected to equilibration in horizontal shaker at 300 rpm. After certain contact time, the solution was filtered through Whatman No.1 filter paper and the residual dye in the filtrate was analyzed using Spectrophotometric method.

Estimation of the Dye: The dye has λmax at 714 nm and obeys Beers-Lambert's law even at low concentrations. The O.D. values were measured at the said λ_{max} using UV-Visible Spectrophotometer (SL-159, Elico) against blank. The O.D values for un-known solutions were referred to standard graphs (drawn between O.D and known concentrations) to find the residual concentration of the dye.

The dye uptake capacity was calculated using Amount adsorbed qe = $\frac{(C_0 - C_e)}{m} \times V$, where q_e = amount of dye adsorbed, m = mass of adsorbent (g), V = volume of the solution (L), C_0 = initial Concentration of dye (mg/ L), C_e = equilibrium dye concentration (mg/ L) and q_e = amount of dye quantity adsorbed at equilibrium (mg/g). The percent removal of the dye from the solution was calculated by the following equation: % removal = $\frac{(C_0 - C_i)}{C_0} \times 100$, where C_0 (mg/L) and C_i (mg/L) are the initial and final dye concentrations respectively.

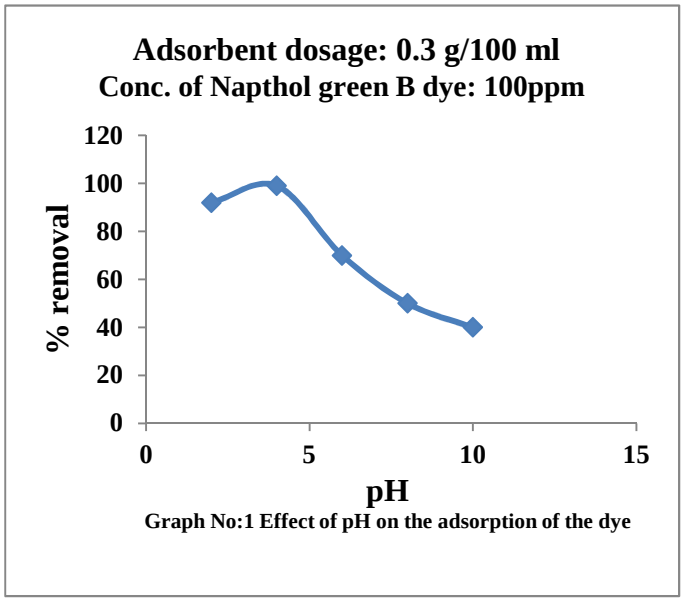
Effect of various physicochemical parameters such as pH, sorbent dosage, equilibration time, initial concentration of the dye and temperature on the sorption nature of the of the Hydrogen peroxide treated activated red mud towards

the anionic dye, Naphthol green B, was studied and the results were presented in the Graph No. 1-8; Table No.2 and 3.

RESULTS AND DISCUSSION

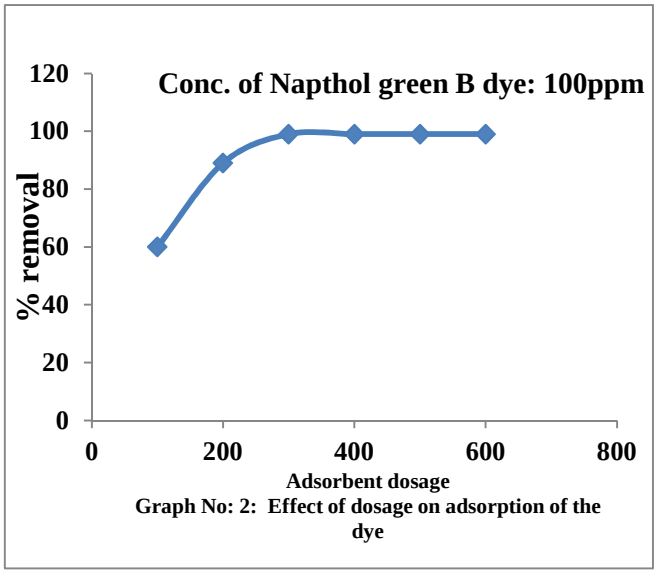
The adsorption nature of the Hydrogen peroxide treated red mud towards the dye has been found to be influenced by various parameters as has been described hereunder.

3.1: Effect of pH: The effect of pH on the adsorption of Naphthol green B dye has been studied by changing the pH from 2 to 10 while maintaining the other optimum conditions of extraction: initial conc. of dye: 100ppm; adsorbent dosage: 0.3gm/100ml, contact time: 40 minutes; rpm: 300 and temperatures: 30^0 C. The results are depicted in Graph No.1. At low pHs: 2 and 4, substantial amounts of the dye have been removed; at pH: 4, 99.0% removal is observed. But with the increase in pH, the % of extraction is decreased.



Graph No:1 Effect of pH on the adsorption of the dye

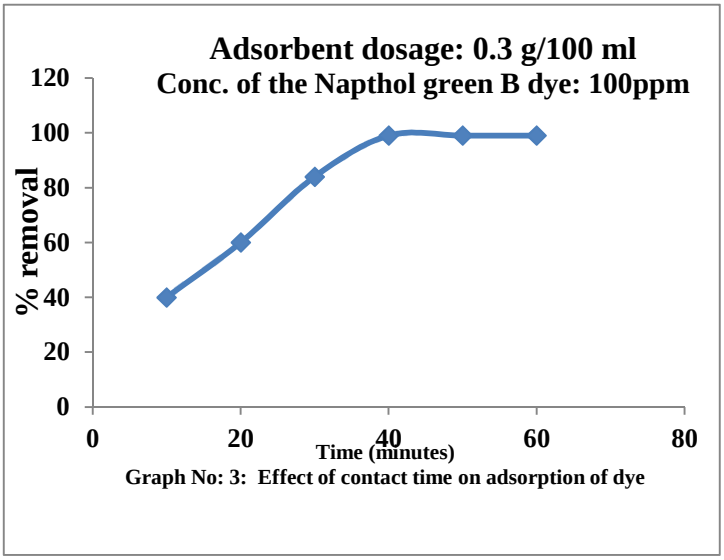
This is expected because at low pH values, the surface –OH groups on the activated red mud are protinated and thereby, the surface of the red mud acquires positive charge and this positive charge imparts affinity of the surface towards anions. Hence, Naphthol green B dye being an anion at pH: 2 and 4, has shown substantial extraction at low pH values. With the increase of pH, deprotination occurs and also the surface –OH groups get dissociated and thereby, endowing the surface a negative charge and this negative charge doesn't favour the adsorption of the anionic dye on to the surface of the red mud and hence, less removal of the dye is observed at high pH values.



Graph No: 2: Effect of dosage on adsorption of the dye

3.2: Effect of adsorbent dosage:
The effect of sorbent concentration on the % removal was investigated by changing the sorbent dosage from 100mg to 500mg/ 100 ml at other optimum extraction conditions (pH: 4, time of equilibration: 40 min, rpm: 300, initial conc. of dye: 100 ppm, temp.:30^{0}C) and the results are presented in the Graph No. 2.

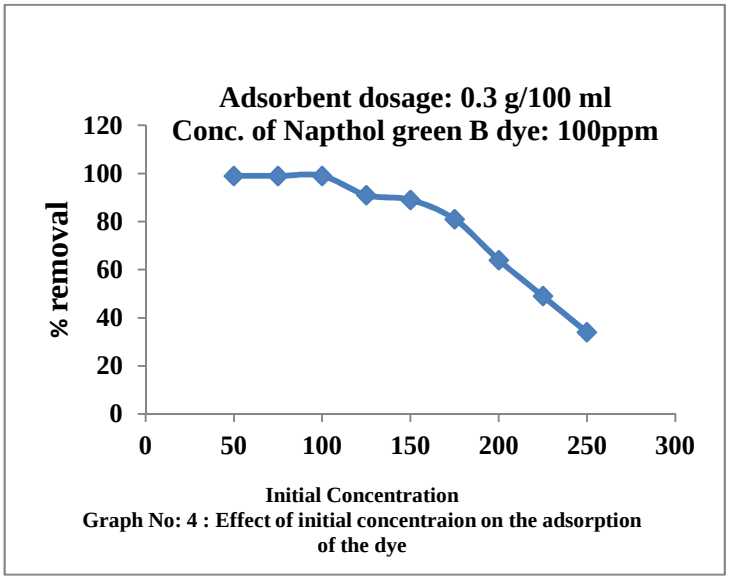
It is observed from the Graph that as the adsorbent dosage is increased from 100 mg to 500 mg/100 ml, % removal of the dye is increased from 60.0% to 99.0%. These observations may be accounted as with the increase in the concentration of the sorbent, the available active sites also increase and hence % removal is more initially. But when the concentration is high, some of the active sites may be blocked and thereby, a study state is resulted and hence at high dosage, % of extraction is marginally affected.



Graph No: 3: Effect of contact time on adsorption of dye

3.3: Effect of Contact time: The influence of contract time on the % removal of the dye has been evaluated by changing the time of equilibration from zero to 60 min at other optimum conditions of extraction namely pH: 4, sorbent dosage: 0.3g/100ml, rpm: 300 and temp. 30^{0}C and the results are presented in Graph No: 3

It is seen from the Graph that % of extraction increases almost linearly up to 40 min and then onwards a steady state is resulted. The maximum extraction is found to be 99.0% at 40 min of equilibration



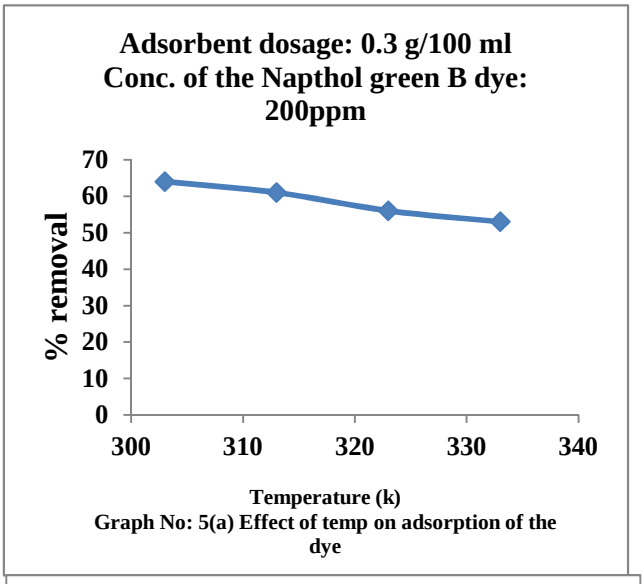
Graph No: 4 : Effect of initial concentraion on the adsorption of the dye

3.4: Effect of initial concentration:

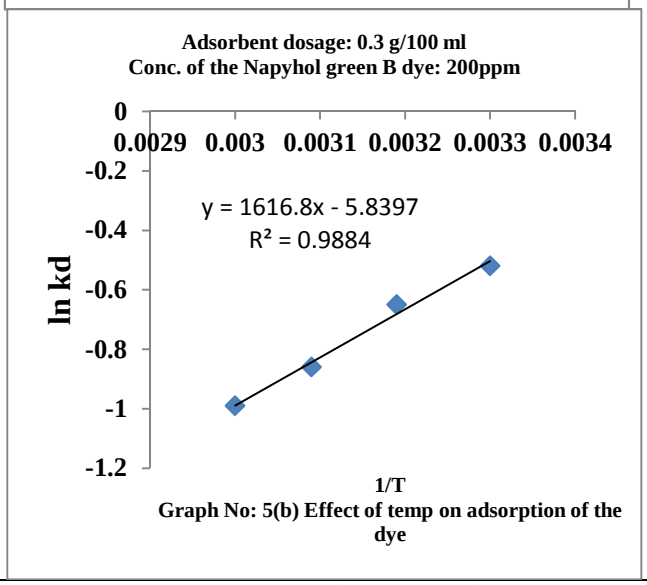
The influence of initial concentration of the dye on the % removal when all the other conditions of extraction are at optimum levels has been probed and the results are depicted in the Graph No.4. It can be noted that as the initial concentration changes from 50 to 250 mg/L, % removal of the dye changes from 99 to 34% for a fixed concentration of the adsorbent, 0.3g/100ml. This is as expected because at low concentrations of the dye, sufficient active sites on the adsorbent are available but at high concentrations of the dye, the demand for active sites is more but the available active sites are limited and hence, the % removal is less.

3.5: Effect of temperature:

The influence of temperature on the % removal of the Naphthol green B dye (200 ppm) has been investigated by changing temperature from 303 to 333 k at optimum extraction conditions (pH: 4, equilibration time: 40 minutes, rpm: 300 and dosage: 0.3 g/100 ml) and the results are shown in the Graph No. 5a & 5b.



Graph No: 5(a) Effect of temp on adsorption of the dye



Graph No: 5(b) Effect of temp on adsorption of the dye

Table No: 2 Thermodynamic Parameters of Adsorption of dye on treated Red mud

ΔH (KJ/mole)	*ΔS (J/mole)*	*ΔG (KJ/mole)*				R^2
-13.44	-48.55	1.26 (303k)	1.75 (313k)	2.23 (323k)	2.72 (333k)	0.988

Thermodynamic parameters namely free energy change (ΔG) (KJ/mole), enthalpy change (ΔH) (KJ/mole) and entropy change (ΔS) (J/mole) were determined using the equations; $\Delta G = -RT \ln K_d$; $\ln K_d = \Delta S / R - \Delta H / RT$; $K_d = q_e / C_e$; $\Delta G = \Delta H - T\Delta S$, where K_d: distribution co-efficient of the adsorption, q_e: the amount of dye adsorbed, C_e: equilibrium conc. of dye, T : the absolute temperature in Kelvin, R: the gas constant. The values of ΔH and ΔS were obtained from the slope and intercept of the plot between $\ln K_d$ and 1/T. [54-58]. The values are given

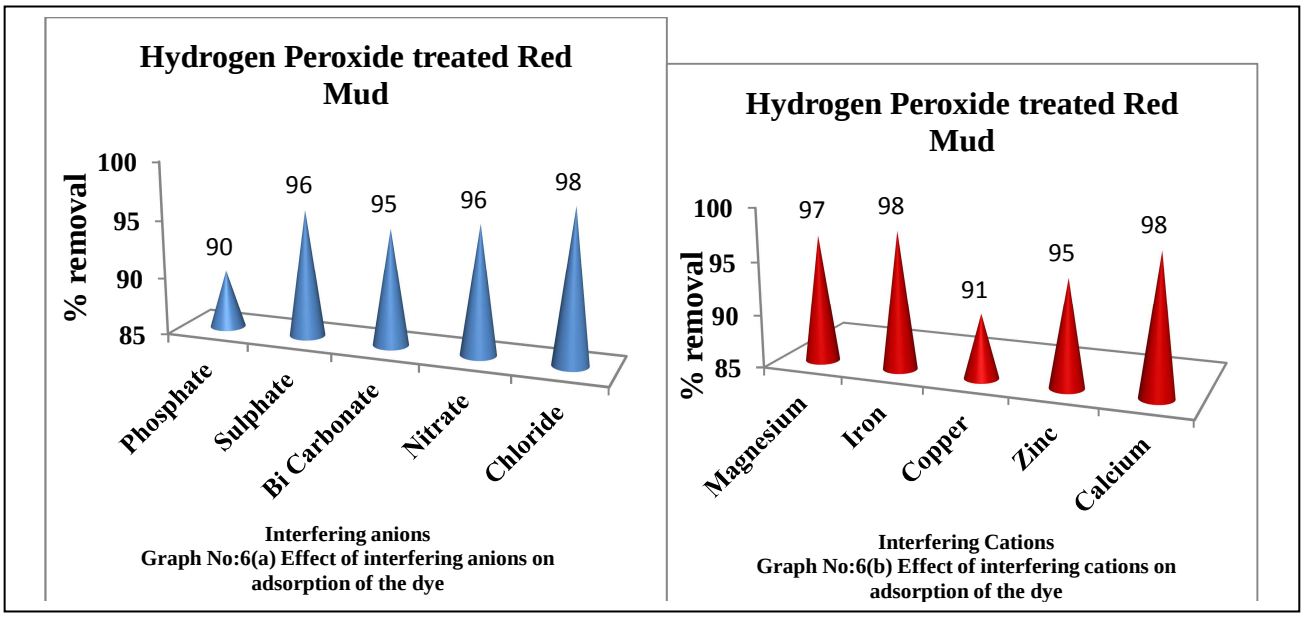
in the Table No: 2. % of extraction decrease from 64% to 53% with the increase in temperature from 303 to 333 k for dye solution (200 ppm concentration).

The positive values of ΔG indicate that the adsorption of the dye is non-spontaneous in nature. However, as $\Delta S < 0$ and $\Delta H < 0$, the process is spontaneous at low temperatures and non-spontaneous at high temperatures. The negative value of ΔH (-13.44 kJ/mol) reflect the exothermic nature of the adsorption process.

3.6: Effect of Co-ions:

% of extraction has been studied in the presence of fivefold excess commonly found co-ions , both anions and cations, in water and the results are presented in the Graph Nos. 6a&b. The % of extractions is less affected. In the case of cations, the interference is of the order:

$Cu^{2+}>Zn^{2+}>Mg^{2+}>Ca^{2+}=Fe^{2+}$, and in the case of anions, the order is : $PO_4^{3-}>HCO^{3-}>NO^{3-}=SO_4^{2-}>Cl^-$



Interfering anions
Graph No:6(a) Effect of interfering anions on adsorption of the dye

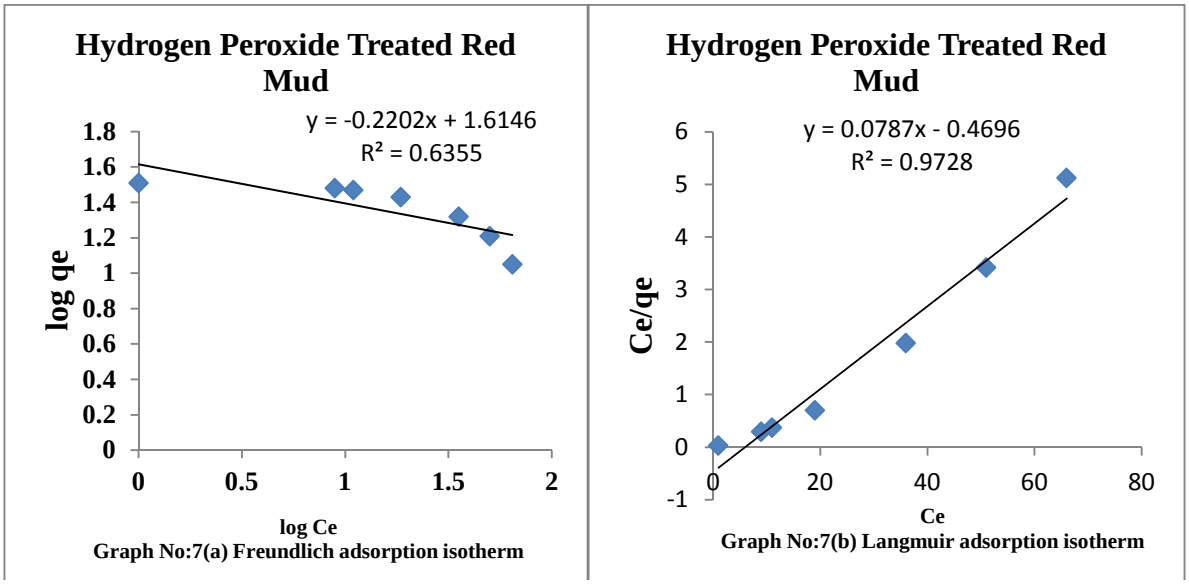
Interfering Cations
Graph No:6(b) Effect of interfering cations on adsorption of the dye

3.7: Adsorption Isothems:

The adsorption nature has been analyzed by using, Freundlich [59], Langmuir [60], Temkin [61] and Dubinin-Radushkevich [62] isotherms. Linear form of Freundlich equation is $\log (q_e) = \log k_f + (\frac{1}{n}) \log C_e$;Linear form of Langmuir equation is $C_e/q_e) = (a_L/k_L)Ce + 1/k_L$. The nature of the adsorption process is unfavorable when $R_L > 1$, linear when $R_L = 1$, favorable when $0 < R_L < 1$ and irreversible when $R_L = 0$ and further, the significant feature of the Langmuir isotherm model is defined by the dimensionless separation factor, $R_L = 1/ (1+ a_L C_i)$ The linear plots of these two adsorption isotherms were as shown in Graph Nos :7a &b and isothermal constants along with the correlation coefficient values are presented in Table 3.

Freundlich isotherm {log (q_e) vs log (c_e)} and Langmuir isotherm (Ce/qe Vs Ce) have been presented in the Graph No. 7a and 7b respectively. The slope (a_L/k_L), intercept ($1/k_L$), and R^2 and R_L values have been calculated and presented in the Table No. 3

With R^2 value of 0.9728, Langmuir isotherm model is more suitable to describe the adsorption process than Freundlich model with R^2 = 0.6355. Further as R_L= 0.0634, lying between zero and one, indicates that the adsorption of the dye is onto the surface of the treated red mud.



Graph No:7(a) Freundlich adsorption isotherm **Graph No:7(b) Langmuir adsorption isotherm**

Further, the adsorption process is analyzed with Linear form of Temkin equation: $q_e = B\ln C_e + B\ln A$ where RT/b = B and Linear form of Dubinin-Radushkevich equation : $\ln q_e = -\beta\varepsilon^2 + \ln q_m$, where $\varepsilon = RT \ln(1+1/Ce)$. The linear plots are shown in Graph. No: 7c & d and isothermal constants along with the correlation coefficient values are presented in Table 3. The mean free energy (E) and heat of sorption (B) are characteristics of adsorption and as E value is less than 8 kJ/mol and B is less than 20kJ/mol, the adsorption is "physisorption" in nature.

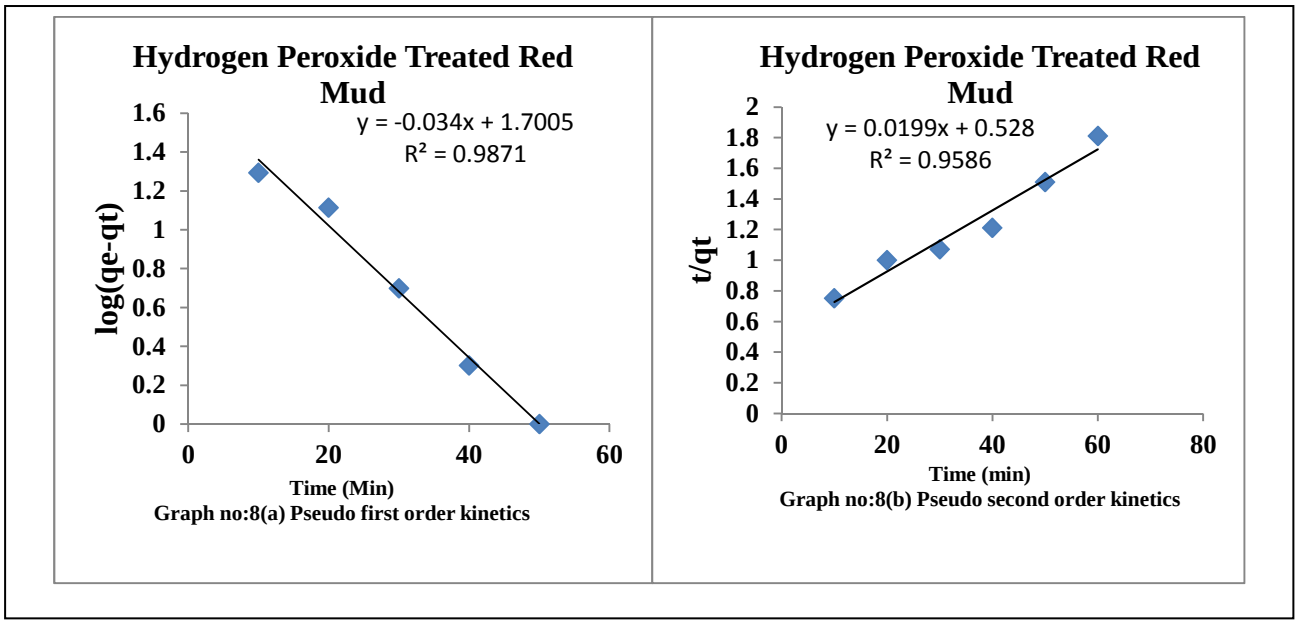
3.8: Adsorption kinetics:

Kinetics of adsorption is s studied using pseudo first-order model [63,64], pseudo second-order model [65,66], Bangham's pore diffusion model[67,68] and Elovich equations[69,70]. The pseudo first-order equation is $\log (q_e - q_t) = \log q_e - k_1 t/2.303$; the pseudo second-order equation is $t/q_t = 1/k_2 q_e^2 - (1/q_e)\, t$; Bangham's pore diffusion equation is $\log [\log (C_i/C_i - q_t m)] = \log (k_o/2.303V) + \alpha \log(t)$; Elovich equation is $q_t = 1/\beta \ln(\alpha\beta) + 1/\beta \ln(t)$

The data of these four kinetic models are presented in Graph Nos: 8-a to d, and rate constants along with the correlation coefficient values are presented in Table 3.

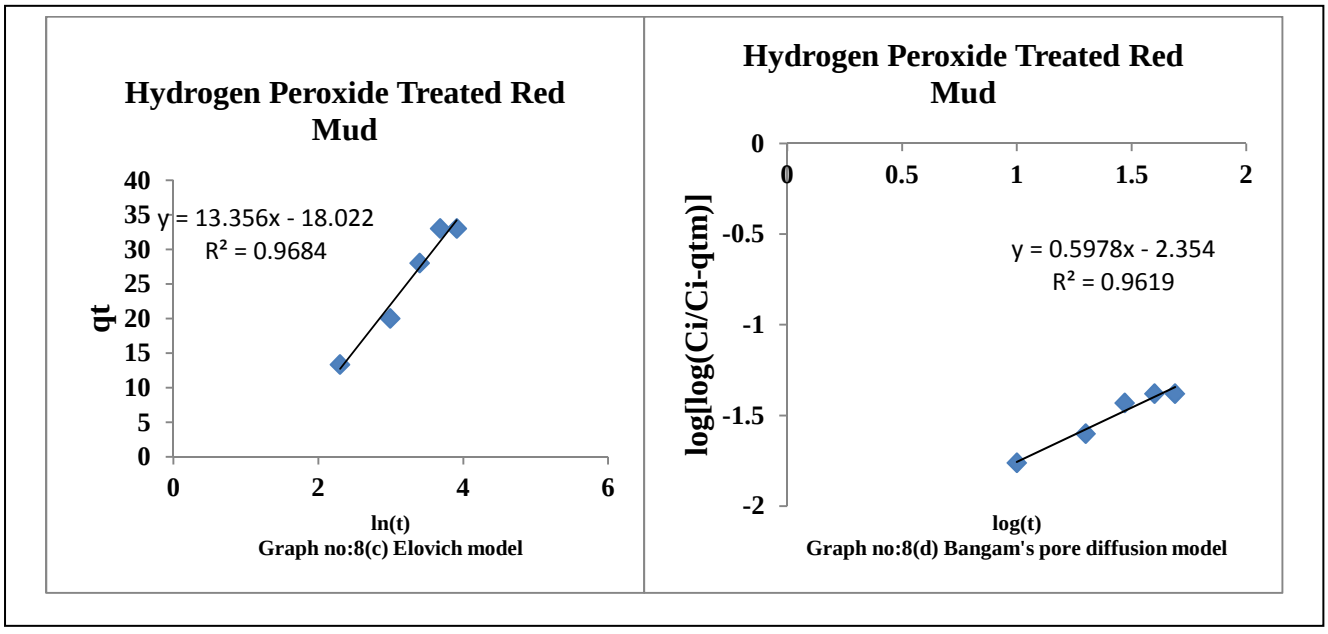
Table 3: Adsorption and Kinetic parameters

S.No.	*Adsorption and Kinetic parameters*		*Slope*	*Intercept*	R^2
1.	Freundlich Isotherm		-0.2202	1.6146	0.6355
2.	Langmuir Isotherm	R_L= 0.0634	0.0787	-0.4696	0.9728
3.	Temkin Isotherm	B=-4.8605	-4.8605	37.479	0.7400
4.	Dubinin-Radushkevich Isotherm	E=0.5 KJ	2E-06	3.0001	0.3028
5.	Pseudo- first-order model		-0.034	1.7005	0.9871
6.	Pseudo-Second-order model		0.0199	0.528	0.9586
7.	Elovich Model		13.356	-18.022	0.9684
8.	Bangham's pore diffusion model		0.5978	-2.354	0.9619



Graph no:8(a) Pseudo first order kinetics

Graph no:8(b) Pseudo second order kinetics



Graph no:8(c) Elovich model

Graph no:8(d) Bangam's pore diffusion model

It may be inferred from correlation coefficient (R^2) values that the kinetics of adsorption follows the order: pseudo first-order > Elovich model> Bangham's pore diffusion > > pseudo second-order. So, the adsorption kinetics is well defined by pseudo first-order and least by pseudo second-order.

3.9: I R Spectral Data:

FTIR data before and after adsorption of the dye are presented in the Fig No. 2.

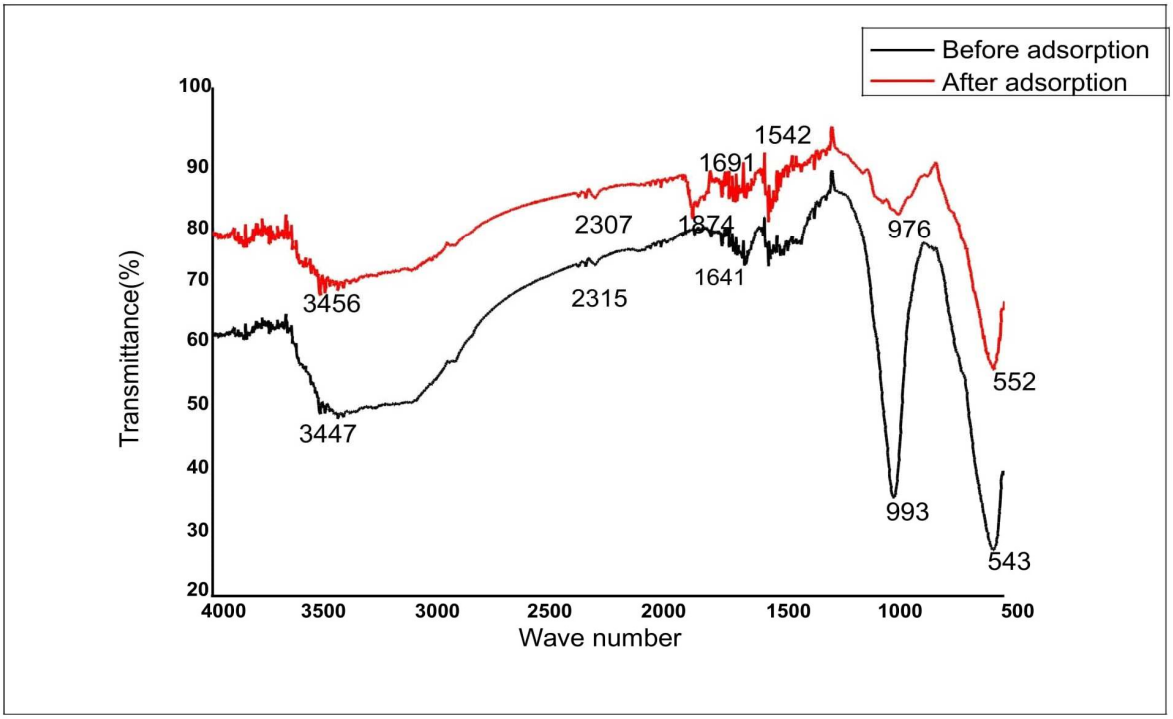


Figure 2: FTIR Spectra of Hydrogen peroxide treated Red mud before and after adsorption of the dye

It is seen from the spectrum that broad band around 3447 cm^{-1} and the narrow band around 1641 cm^{-1} assigned to the -OH stretching and bending vibrations (of adsorbed water and/or surface hydroxyl groups) are shifted to 3456 and 1691 cm^{-1} respectively with decrease of intensity after the dye is onto the surface of the red mud.

Further, the appearance of new small bands in the range 1400 to 1550 cm^{-1} pertain to the aromatic nature and a new band at 1874 cm^{-1}, indicate that the dye is onto the surface of the red mud.

The sifting of band from 993 cm^{-1} (before adsorption) to 976 cm^{-1} (after adsorption) pertaining to asymmetric stretching of Si-O-Si and bending vibrations of Al-O-Si, and shifting of band from 543 cm^{-1} to 552 cm-^{1} pertaining to stretching vibrations of Fe-O reflect the sorption of the dye on to the surface of the red mud.

APPLICATIONS

The developed methodology in this investigation was applied to effluents samples of textile industries at Mangalagiri and Machilipatnam of Andhra Pradesh. The samples were analyzed for the actual amounts of the said dye and then the samples were fed with known amounts of the dye.

Table No:4 Removal of colour in swages of dying industries

S. No.	Water samples	C_i (mg/lit) (initial concentration of dye)	C_f (mg/lit) (concentration of dye after removal)	% Removal
1	Sample 1	50	1	98
3	Sample 2	75	6	92
4	Sample 3	100	17	83

Then these samples were subjected to extraction and the obtained results were presented in the Table No. 4. It is inferred from the table that the procedure developed in this work is effectively removing the Naphthol green B dye from waste waters.

CONCLUSION

- Hydrogen peroxide treated red mud has been investigated for its ability to remove anionic dye, Naphthol green B dye adopting batch methods. Extraction conditions such as pH, adsorbent concentration, equilibration time, temperature and initial concentration of the dye have been optimized for the maximum removal of the dye.

- 99.0% extraction of the Naphthol green B dye is found at pH: 4; agitation time: 40 minutes; 300 rpm, temp: 30^0C, initial conc. of the dye: 100 ppm and sorbent dosage: 0.3 g/100ml.
- Normally found co- ions in waters viz., NO_3^-, CO_3^{2-}, Cl^-, PO_4^{3-}, SO_4^{2-}, Zn^{2+}, Ca^{2+} , Mg^{2+} , Fe^{2+}, and Cu^{2+} have least interfered.
- The sorption nature is analyzed using Langmuir, Freundlich, Temkin and Dubinin-Radushkevich isotherms and noted that the adsorption of the dye onto the surface of the activated red mud is 'physisorption' in nature with monolayer formation.
- The kinetics of adsorption are analyzed using pseudo-first-order, pseudo-second-order, Bangham's pore diffusion and Elovich equations and found that the adsorption follows the pseudo-first-order kinetics.
- On the analysis of the thermodynamic parameters, it is revealed that the adsorption of the dye is an exothermic process and the adsorption is less favored at elevating temperature.
- The investigated methodologies are successfully applied for the removal of anionic dye from the effluents of the textile industries.

Acknowledgement
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