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Fly Ash for Removal of Malachite Green Dye From Wastewater: Kinetic and Isotherm Studies

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Abstract: *The release of toxic dye components from dye manufacturing industries has become a severe threat and causing water contamination. Adsorption is one of the common techniques, employing solid adsorbents such as activated carbon, alumina silica, wood, Fuller's earth, fired clay, and metal hydroxides to remove traces of dye components from water. In India, every year 180 million tons of fly ash is produced and the disposal of the same is a major issue. To address the disposal problem, one of the best practices is to use flue ash as an adsorbent. In the present work flue ash has been used to remove components of malachite green dye from concentrated stock solution. A set of experiments in batch mode were performed to test the potential of flue ash for the removal of Malachite Green and the effects of parameters such as such as time of contact, pH, concentration of adsorbate, adsorbent dosage. Dynamic Light Scattering (DLS) technique is used to determine the average particle size of flue ash. Study on adsorption isotherms and adsorption kinetics was performed and most suitable models for adsorption isotherms and rate kinetics were proposed.*

Keywords: *Malachite Green; Flue ash; DLS; Adsorption; Adsorbent; Adsorbate; Adsorption kinetics; Adsorption isotherms*

I. Introduction

Chemical industries, refineries, textile, plastic and food processing plants discharge harmful effluents. Among these, textile industries are the main source for discharge of dye components. There are a variety of dyes and huge amounts of dyestuff are produced every year for various applications. The classification and applications of dyes are depicted in Fig. 1 a & b. The discharge of effluent water containing heavy metals and toxicdye components into the water bodies leads to contamination and penetrate beneath the soil, making it unsuitable for cultivation (Sushmita 2016). Dye components even in minutequantities affect the marine life and the food cycle by inhibiting the sunlight incident onto the water bodies or by consuming the dissolved oxygen from the water bodies (Gita 2017). To mitigate this effect, synthetic dyes can be substituted with natural dyes.The natural dyes are made from plants and mineral sources, which could be berries, plant roots, wood, bark, fungi, leaves, and lichens(Virendra Kumar Gupta 2019). Synthetic dyes are usually made from coal tar and petroleum and are carcinogenic and mutagenic. India has been one of the largest manufacturers of dyes and pigments from the past 35 years (GhodsiMohammadiIarani 2018).

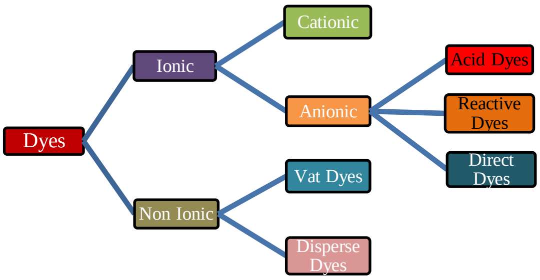


Fig 1(a): Classification of Dyes

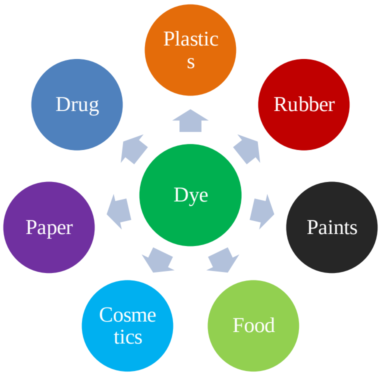


Fig 1(b): Application of dyes

Therefore, it is serious concern to focus on removal/separation of dye components from the effluent waters. The available methods for the removal of dyes from effluent waters are: adsorption, membrane separation processes, coagulation, electrochemical process, reverse osmosis, chemical oxidation, aerobic and anaerobic microbial degradation of dye components (Rummi Devi Saini (2017) &Kok Bing Tan 2015). However, intensive power requirements, evolution of toxic by-products, and high cost makes most of the methods less feasible on large scale plants for the eviction of dye components from the effluent water.

Adsorption is rapidly gaining prominence among the treatment technologies due to low cost and high performance. Effect on environment, economic advantages, and adsorption efficiencies are the main parameters considered by researchers while selecting an adsorbent. A variety of adsorbents derived from agriculture residues, industrial wastes have been employed to remove various hazardous components from effluent water (Kurniawan 2005). Agriculture residues such as coconut shell, coir pith, rice husk, nut shells were processed to activated carbon and are used in adsorption (Shabudeen Syed 2010, Dalia Khalid Mahmoud 2011, Oladoja 2008 &Shawabkeh 2007). Industrial wastes such as fly/flue ash (Gowswami 2014), red mud (Chen 2009), green sand, waste iron, metallic iron, blast furnace slag, titanium oxide (Kok Bing Tan 2015) are some of the examples.

Activated carbon is the mostly used adsorbent for dye removal(Sivakumar 2008 &EwaLorenc-Grabowska 2007]. The major issues with activated carbon as adsorbent are high cost, difficulty in regeneration of adsorbent, and low adsorption capacities led to search to find new adsorbents which are of low cost and have high adsorption capacities. Thus, flue ash obtained from combustion of coal in thermal power plants is abundantly available in India and can be observed as the main source of adsorbent for removal of dye from waste water.

India alone is producing 180 million tons of fly ash every year (ShardaDhadse 2007).Flue ash is one of the coalignition products, composed of the fine particulate matterthat are directed out of the boiler along with the flue gases and is a natural by-product from thermal power plants running with combustion of coal. Electrostatic precipitators (ESP) are installed at the outlet nozzles in order to collect flue ash from the flue gases before they are released into the atmosphere. Flue ashis in spherical shape with diameter in the range 1 µm to 150 µm. Thus low-cost adsorbent like flue ash obtained from coal combustion in thermal power plants is used in the present study to remove malachite green from water.

II. Literature Review

Adsorption

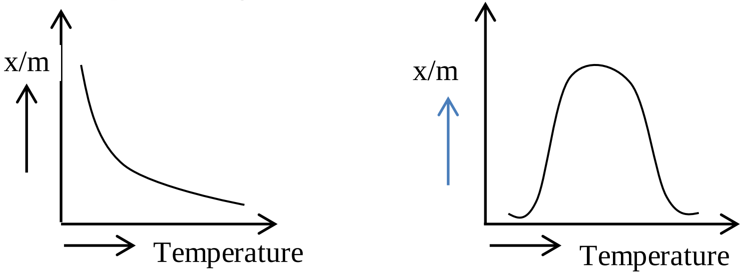
Adsorption is a surface based phenomenon in which one or more than one constituents from the feed get accumulated on the solid adsorbent. The atoms, molecules, and ions which get deposited on to a surface is known asadsorbate and the substance that provide surface for adsorption to takes place is known as an adsorbent. As heat is evolved during the process, adsorption is considered as an exothermic process and ΔH is always negative and is given as the quantity of heat generatedfor one mole of the adsorbategetadsorbed on the surface of adsorbent (MohdAzmier Ahmad 2011). As adsorption goes on the active surface area available for new molecules gradually decreases and leads to decrease in degree of randomness (entropy) of molecules. Adsorption process is carried out at constant temperature and pressure. It is a spontaneous process,thus ΔG is also decreased.

Types of adsorption

There are two major classes of adsorption phenomena based on thebinding forces present between the molecules getting adsorbed and the adsorbent:

(i) Physical adsorption and
(ii) Chemical adsorption

Physical adsorption is a multilayer phenomenon where the binding forces are weak Vander Waals forces. Whereas chemical adsorption (or) chemisorption is a uni-layer phenomenon with strong chemical forces and covalent bonds are present between adsorbate and adsorbent. Moreover, in chemisorption attractive forces exist between only absorbate and adsorbent. ΔH adsorption is 20-40 kJ/mol for physisorption and 200-400 kJ/mol for chemisorption. Physisorption is inversely proportional to temperature and chemisorption first increases and then decreases with increase in temperature (Fig. 2).



Physical adsorption

Chemical adsorption

Fig 2: Temperature effect on adsorption

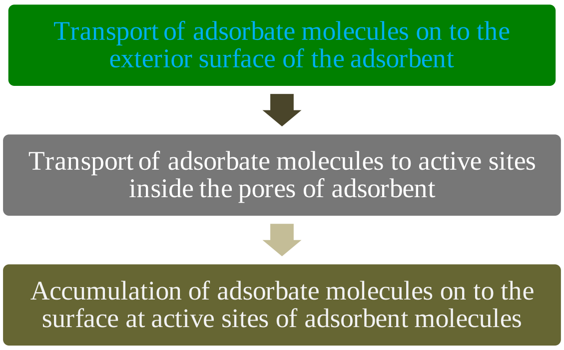


Fig 3: Mechanism of adsorption

Adsorption mechanism is depicted in Fig 3. Major part of adsorption takes place in the pores of adsorbent molecule because of their larger surface area (MohdAzmier Ahmad 2011&Indra Deo Mall 2006).

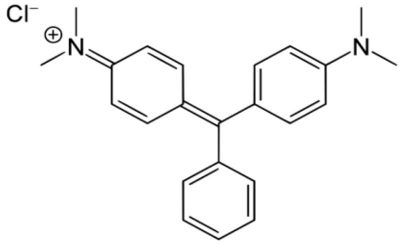
III. Materials and Methods

Materials

Fly ash is collected from Rayalaseema Thermal Power Plant (RTPP), Kadapa district. Fly ash is dried in a hot air oven at 100^{0}C to remove moisture content before it is used in the experiments. Fine particles of flue ash are collected by using molecular sieves of micro pores. Particle size distribution and hydrodynamic radius of flue ash particles is analysed by using Dynamic Light Scattering technique (DLS) which works on the principle of Brownian motion. DLS is used for particles of size less than 5 nm. DLS works on the principle of Brownian motion.

The chemical structure of malachite green dye is shown in Fig 4 and the other information including type of dye, chemical formula and molecular weight are also provided in the following table. Malachite green dye is used as adsorbate. Malachite green dye stock solution of concentration 1000 mg/L was prepared by mixing the accurately weighed quantity of dye with double distilled water. The stock solution was diluted for different dye concentrations ranging from 2 mg/L to 10 mg/L (2, 4, 6, 8, 10 mg/L).

The % absorbance of dye was measured by using UV spectrophotometer at a maximum wave length (λ_{max}) of 618 nm. A standard calibration curve with the values of % absorbance on y-axis and concentration on x-axis was plotted. Concentration value at a given % absorbance value was determined by using equation of the calibration curve.



Name of the dye	Type	Chemical formula	Molecular weight
Malachite green	Cationic dye	$C_{52}H_{54}N_4O_{12}$	927

Fig 4: Structure of Malachite Green

Effect of Various Parameters on % Removal of Malachite Green

Adsorption studies of malachite green were carried out using fly ash as adsorbent. Thebatchexperimentswith a working volume of 200 mL and adsorbent dosage of 1 g/L wereperformed in Erlenmeyerflasks. The speed of rotation of orbital shaker (REMI instruments) for mixing of the contents was maintained at 300 rpm and temperature at 26 ºC. The samples were collected at regular intervals of 5, 10, 15, 20, 25, 30, 35, 40, 50, 60, 90, 100, 120, 130 and 150 minutes till equilibrium is obtained. The samples were filtered and analysed using UV-VIS spectrophotometer (UV 1800 Shimadzu,) for dye concentrationat a wavelength of λ_{max}=618 nm corresponding to highest absorbance.At equilibrium, theamountofdye adsorbed,q_e(mg/g),wascalculatedusingthefollowingequation:

$$q_e = \frac{c_o - c_e}{x}$$

Where,

$q_e =$ Equilibrium capacity of dye on adsorbent (mg/g)

$c_o =$ Initial concentration of dye solution (mg/L)

$c_e =$ Final concentration of dye solution (mg/L)

$x =$ Weight of adsorbent used, g

Batch experiments were performed to study the effect of various parameters including time (5 -140 min), pH (4, 6 and 8), initial concentration of dye (6, 8 and 10 mg/L), and adsorbent dosage (40, 60 and 80 mg/100 mL of solution). In the experiments solution pH was measured using pH meter (ELICO).

Adsorption Kinetics

Mass transfer and chemical reactions are the two key steps in adsorption processes. To determine the adsorption rate controlling step (mass transfer or chemical reaction),the experimental data is tested for pseudo second-order and pseudo first-order kinetic models. A sample dye solution of concentration 10 mg/L and flue ashconcentration of 1 g/Lare used to find the kinetics data. The experiments are carried out for a time of 150 min and samples were collected at regular intervals of time. The % absorbance were obtained using UV-VIS spectrophotometer (λ_{max}=618 nm).From the above obtained experimental data$q_t$and q_e values were calculated.Where

q_t= amount of dye adsorbed per unit amount of flue ash at time t.

q_e= amount of dye adsorbed per unit amount of flue ash at equilibrium time (140 min).

The pseudo-first-order kinetic model (Wan Saime Wan Ngah 2010&Largitte2016) for adsorption is given by;

$$\frac{dq_t}{dt} = k_1(q_e - q_t)$$

Up on integration we get;

$$\ln (q_e\text{-}q_t) = \ln q_e\text{-}\ k_1 t$$

Slope gives $k_1$and intercept gives q_e

The pseudo-second-order kinetic model (Wan Saime Wan Ngah 2010 &Largitte 2016)is given by the following equation

$$\frac{dq_t}{dt} = k_2(q_e - q_t)^2$$

With a hypothesis that the adsorption follows second order chemisorption,up on integration we get;

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

Slope gives q_e and intercept gives k_2.

Where
q_t= Adsorption capacity at time t (mg/g),
k_1=Rate constant of pseudo-first-order adsorption (1/min),
k_2= Rate constant of pseudo-second-order adsorption (g /mg min) and
t = time.

Adsorption isotherms

In order to know how molecules of adsorbate combine with surface of adsorbent adsorption isotherms are studied (Hameed2007, Hameed 2010, Mi-HwaBaek 2010, AshishSartape 2017, Anbia 2011).Freundlich and Langmuir isotherm models are generally used for adsorption studies. Three dye solutions with initial pH values of 4, 6 and 8 and varying adsorbate concentrations of 6 mg/L, 8 mg/L, and 10 mg/L respectively were prepared by maintaining adsorbent concentrationat 1 g/L. All these nine conical flasks containing sample solutions were put into the orbital shaker for 120 minutes and % absorbance's of the samples were determined using UV-VIS spectrophotometer at a wavelength of λ_{max}=618 nm. From theabove experimentally obtained datavalues ofc_e and $q_e$were calculated. By plotting the curves for Freundlich and Langmuir Isotherms with the obtained data, we can determine the best fitting model.

The Langmuir equation is applied only when there are no interactions between the adsorbate – adsorbate molecules (i.e. monolayer adsorption) where only chemical bonds and covalent bonds exist between adsorbate and adsorbent. The Langmuir equation in the linear form is given by:

$$\frac{1}{q_e} = \frac{1}{b q_0 c_e} + \frac{1}{q_0}$$

A graph is drawn between $1/q_e$ vs. $1/c_e$where$q_o$is given by intercept and k_L value is given by slope.

The Freundlich adsorption model in its linear form is given by:

$$\ln q_e = \ln k_f + \frac{1}{n \ln c_e}$$

A graph is drawn betweenln(q_e) vs. ln(c_e) where n value is given by slope and k_f value is given by intercept.Where

q_o= Adsorption capacity of adsorbent (mg/g),
k_f = (constant) capacity of adsorption
b= Langmuir adsorption (L/mg)
n = (constant) intensity of adsorption

IV. Results and Discussion

Particle size distribution of flue ash

The average particle size of flue ash obtained from DLS technique is 261.5 nm. The histogram obtained from DLS method is shown in Fig 5. A calibration graph (Appendix A) of malachite green in water is generated to measure the concentration of malachite green in working samples.

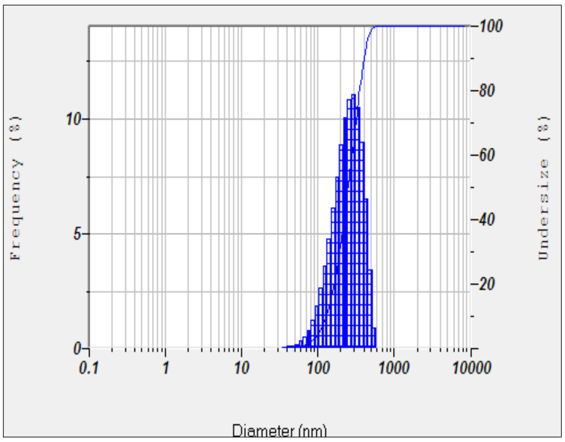


Fig 5: Particle Size distribution histogram of flue ash

Effect oftime of Contact

The effect of time on adsorption of the dye on flue ash is shown in Fig 6. It was observed that the degree of adsorption is high at the beginning stage and gradually decreased. After a time of 120 min the system almost reached equilibrium and there is no much increase in percentage removal of dye can be observed.The equilibrium condition in the present case represents the saturation of the flue ash and there are no active sites for adsorption to take place.

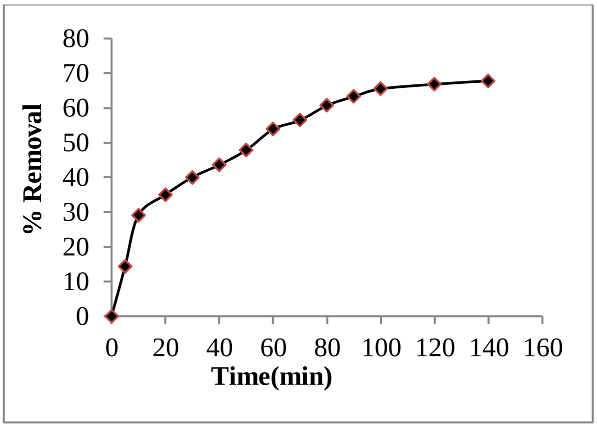


Fig 6 Effect of contact time on % removal of MG

Effect of pH

Solution pH is the other important factor which has a strong influence on adsorption. The effect of pH on % removal of malachite green is shown in Fig 7 and it can be observed that at an initial pH of 4 the % removal was found to be minimal and at a pH of 8a maximum in dye removal was observed.From this result, it can be concluded that the % removal increases as the pH of solution increases. Malachite green is a basic dye which has positive charge. At lower pH the net positive charge in solution is more than the active negatively charged sites on the flue ash causing less adsorption of malachite green at lower pH. The same was observed experimentally and depicted in Fig 7. The percentage adsorption of malachite green at pH 4 is far less than that at pH 8.

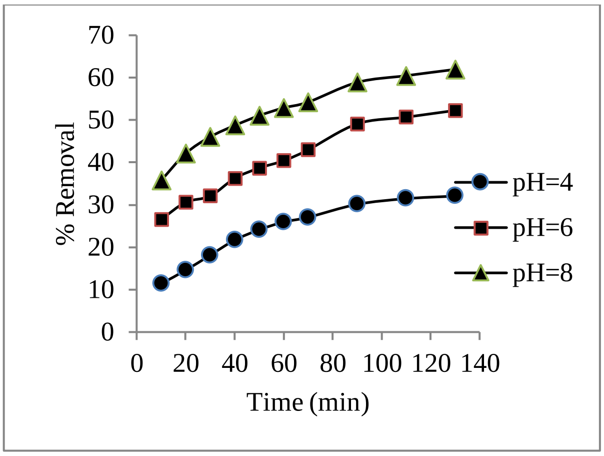


Fig 7 Effect of pH on % removal of MG

As solution pH increases negative charge accumulates in the solution and no competition for the dye to occupy the positively charged active sites on the flue ash. Therefore, with increase in negative charge in the solution side i.e with increase in pH the percentage removal increased.

Effect of Adsorbent Dosage

To study the effect of adsorbent dosage on percentage removal of dye, experiments were conducted to with a fixed dye concentration of 10 mg/L for 120 min. Dosage study will give the true picture of adsorbent capacity in removal of a given dye. Fig 8 shows the percentage removal of dye at three different adsorbent dosages i.e. 40 mg/L, 60 mg/L, and 80 mg/L. It can be observed that the percentage removal increased with increase in adsorbent dosage. This is mainly due to the increase in availability of active surface area with increase in adsorbent dosage.

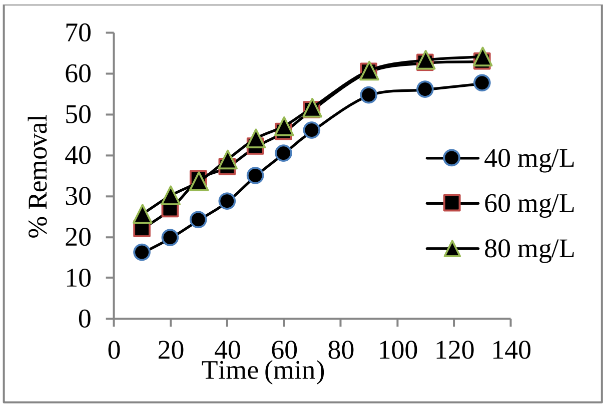


Fig 8: Effect of adsorbent dosage on % removal of Malachite Green

Adsorption Kinetics

Experiments were conducted to study the kinetics of malachite green adsorption onto flue ash using an initial dye concentration of 10 mg/L, adsorbent dosage of 80 mg/L at a constant mixing intensity of 300 rpm and initial pH of 8 for 120 min. Samples were collected at 5, 10, 15, 20, 25, 30, 35, 40, 50, 60, 90, 100, and 120 min and the amount of dye in the solution is estimated. The concentration of dye verses time data is tested for pseudo first order and pseudo second order models and the same was shown in Fig. 9 a & b.

By comparing both Fig 9(a) & (b) it can be said that pseudo first order model fits well for the experimental data than the pseudo second order model. The values of R^2 for pseudo first order fitting (0.9912) is very close to 1 and for pseudo second order fitting is only 0.8872. Therefore, it can be conclude that pseudo-first-order model is the best representing kinetic model for the adsorption of malachite green using flue ash at the given condition.

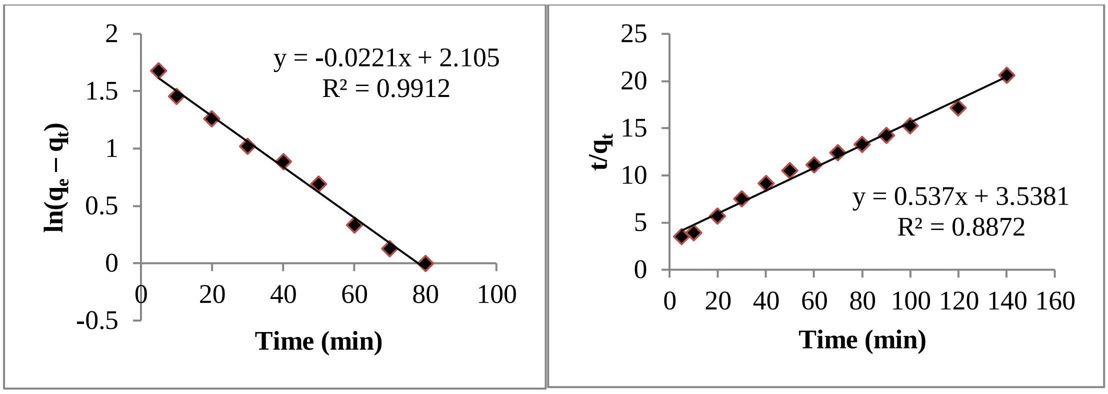


Fig 9 (a): Pseudo first order curve; (b): Pseudo second order curve

Adsorption Isotherm Models

The experimental data obtained at different pH were tested with Langmuir isotherm and Freundlich models and are shown in Fig 10 (a) & (b), respectively. Langmuir isotherm model is given by the following equation.

$$\frac{1}{q_e} = \frac{1}{bq_0c_e} + \frac{1}{q_0}$$

A graph is plotted between $1/q_e$ vs. $1/c_e$ where q_0 is given by intercept and k_L is given by slope. The same is depicted in Fig 10 (a). The values of q_0 and k_L are given in Table 1.

Freundlich isotherm model is given by the following equation.

$$lnq_e = lnk_f + \frac{1}{nlnc_e}$$

A graph is plotted between $\ln(q_e)$ vs. $\ln(c_e)$ where n is given by slope and k_f is given by intercept as shown in Fig 10 (b). The values of n and k_f are given in Table 1.

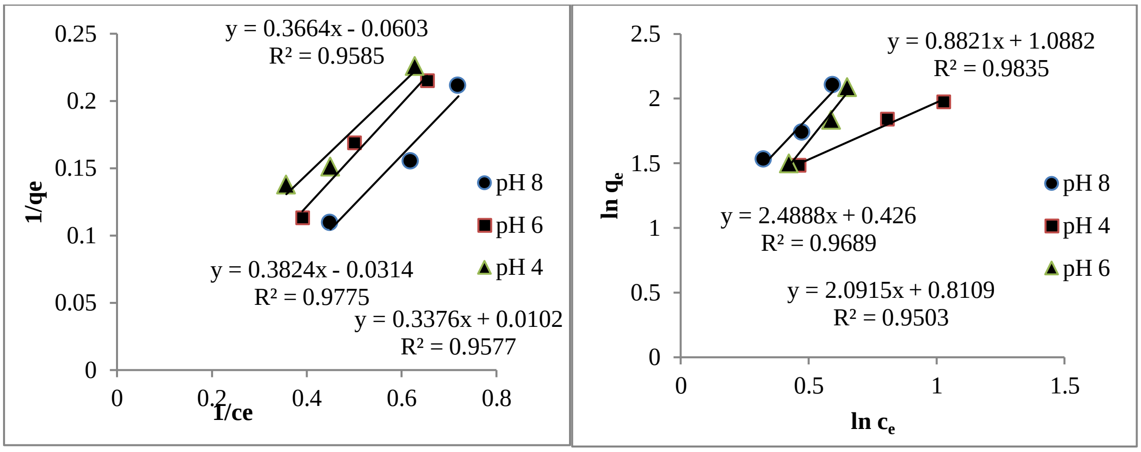


Fig 10 (a): Langmuir isotherm curve; (b):Freundlich isotherm curve

Table 4.1 Langmuir and Freundlich parameters

		pH=4	**pH=6**	**pH=8**
Langmuir Isotherm	**k_L**	0.0302	0.0821	0.1645
	q_0	98.03	31.847	16.583
	R^2	0.9577	0.9775	0.9585

Freundlich Isotherm	**k_f**	2.968	1.531	2.249
	n	1.133	0.401	0.478
	R^2	0.9835	0.9689	0.9503

From the above data it is observed that both the models are well fitted with the experimental data. From the inference based on Langmuir isotherm model, it can be concluded that there is no interaction between adsorbate -adsorbent, the active sites are homogeneously distributed over the adsorbent and monolayer adsorption of malachite green on flue ash surface.From this we can conclude that both the isotherm curves fit well with the adsorption system.

V. Conclusion

The above study and experiment shows the efficiency of flue ash as an adsorbent for dye removal. This article gives the readers a basic idea regarding the effectof various physio-chemical experimental factors on removal of malachite green using flue ash as adsorbent.

The following observations were made:

- Adsorption increases with increase in initial concentration of dye, adsorbent dosage and contact timeand the optimum values are 8 mg/L, 60mg/100mL and 2 hours, respectively.
- Adsorption increased with increase in solution pH.The adsorption of cationic dye is influenced by the presence of negative charge in the solution. The adsorption is greater in pH=8 as compared to pH=4 because of greater net negative charges in former.
- Pseudo first order kinetic model is the best representing model over second order kinetic model. The values of k_1=0.0221 and R^2 = 0.9912.
- Both Langmuir and Freundlichisotherms are well fitted with the experimental data.

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Appendix A: Calibration Plot for Malachite Green