Competitive Adsorption of dyes (congo red, methylene blue, malachite green) on Activated Carbon
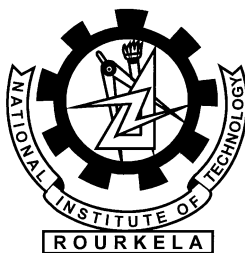
A Project submitted to the
National Institute of Technology, Rourkela

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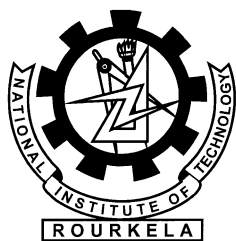*of*
Bachelor of Technology (Chemical Engineering)

By

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CERTIFICATE

This is to certify that the thesis entitled **Competitive Adsorption of dyes (congo red, methylene blue, malachite green) on Activated carbon,** submitted by **Bibek Dash** to National Institute of Technology, Rourkela is a record of bonafide project work under my supervision and is worthy for the partial fulfillment of the degree of Bachelor of Technology (Chemical Engineering) of the Institute. The candidate has fulfilled all prescribed requirements and the thesis, which is based on candidate's own work, has not been submitted elsewhere.

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ABSTRACT

The objective of this work is the study of adsorption of dye solution which is a mixture of three dyes (methylene blue, congo red & malachite green) using commercial activated carbon. Removal of these dyes from aqueous solution using commercial activated carbon has been investigated. Liquid phase adsorption experiments were conducted. Batch adsorption studies are carried out by observing the effect of experimental parameters, namely, pH, amount of adsorbents, contact time and temperature. Adsorption capacity of activated carbon is determined for competitive adsorption of mixture of dyes. Optimum conditions for dye removal are studied like pH value, contact time required, amount of adsorbent, temp, etc. The results generated by this project work can be used for determination of optimum conditions for adsorption of mixture of dyes in aqueous solutions. Dyes are present in mixture form in various Industrial effluents like Textile Industries, Sewage water, Water treatment plants. This work can have use in Design of adsorption columns for dyes removal.

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CHAPTER-01
INTRODUCTION

Introduction

Dyes have long been used in dyeing, paper and pulp, textiles, plastics, leather, cosmetics and food industries. Colour stuff discharged from these industries poses certain hazards and environmental problems. These coloured compounds are not only aesthetically displeasing but also inhibiting sunlight penetration into the stream and affecting aquatic ecosystem. Dyes usually have complex aromatic molecular structures which make them more stable and difficult to biodegrade. Furthermore, many dyes are toxic to some microorganisms and may cause direct destruction or inhibition of their catalytic capabilities.

Textile industry use dyes and pigments to colour their product. There are more than 100,000 commercially available dyes with over 7×105 tonnes of dyestuff are produced annually.

Many types of dye are used in textile industries such as direct, reactive, acid and basic dyes. Most of these dyes represent acute problems to the ecological system as they considered toxic and have carcinogenic properties, which make the water inhibitory to aquatic life .Due to their chemical structure, dyes possess a high potential to resist fading on exposure to light and

water. The main sources of wastewater generated by the textile industry originate from the washing and bleaching of natural fibers and from the dyeing and finishing steps. Given the great variety of fibers, dyes and process aids, these processes generate wastewater of great chemical complexity and diversity, which are not adequately treated in conventional wastewater treatment Plant.

Numerous studies have been conducted to assess the harm impacts of colorants on the ecosystem. It was found that colorants may cause problems in water in several ways: (i) dyes can have acute and/or chronic effects on exposed organisms with this depending on the dye concentration and on the exposure time; (ii) dyes are inherently highly visible, minor release of effluent may cause abnormal coloration of surfacewaters which captures the attention of both the public and the authorities; (iii) the ability of dyes to absorb/reflect sunlight entering the water, this has drastic effects on the growth of bacteria and upsets their biological activity; (iv) dyes have many different and complicated molecular structures and therefore, are difficult to treat and interfere with municipal waste treatment operations; (v) dyes in wastewater undergo chemical and biological changes, consume dissolved oxygen from the stream and destroy aquatic life; (vi) dyes have a tendency to sequester metal ions producing micro toxicity to fish and other organisms.

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There are various conventional methods of removing dyes including coagulation and flocculation, oxidation or ozonation and membrane separation . However, these methods are not widely used due to their high cost and economic disadvantage. Chemical and electrochemical oxidations, coagulation are generally not feasible on large scale industries. In contrast, an adsorption technique is by far the most versatile and widely used. The most common adsorbent materials are: alumina silica, metal hydroxides and activated carbon. As proved by many researchers, removal of dyes by activated carbon is economically favorable and technically easier. Activated carbon is widely used as an adsorbent due to its high adsorption capacity, high surface area, microporous structure, and high degree of surface respectively.

CHAPTER-2
LITERATURE REVIEW

Literature Review

Activated carbon, also called activated charcoal or activated coal, is a form of carbon that has been processed to make it extremely porous and thus to have a very large surface area available for adsorption or chemical reactions.

Activated carbons are highly developed internal surface area and porosity, sometimes described as solid sponges. The large surface area results in a high capacity for adsorbing chemicals from gases liquids. The most widely used commercial active carbons have a specific surface area of the order of 800-1500 m2/g, as determined typically by nitrogen gas adsorption. Difference in pore size affects the adsorption capacity for molecules of different shapes and sizes, and thus is one of the criteria by which carbons are selected for a specific application. Porosity is classified by IUPAC into three different groups of pore sizes. They are:

Micropores: width less than 2 nm

Mesopores: width between 2 and 50 nm

Macropores: width greater than 50 nm

In 2005 Indra Deo Mall, Vimal Chandra Srivastava, Nitin Kumar Agarwal, Indra Mani Mishra studied the Adsorptive removal of malachite green dye from aqueous solution by bagasse fly ash and activated carbon-kinetic study and equilibrium isotherm analyses. Batch adsorption studies were conducted to evaluate the effect of various parameters such as pH, adsorbent dose, contact time and initial MG concentration on the removal of MG. The initial pH of the dye solution strongly affected the chemistry of both the dye molecules and adsorbents in an aqueous solution. Equilibrium reached in about 4 h contact time. The adsorption followed pseudo-second-order kinetics.

In 2005 B.H. Hameed, A.T.M. Din, A.L. Ahmad studied Adsorption of methylene blue onto bamboo-based activated carbon. Kinetics and equilibrium studies Bamboo, an abundant and inexpensive natural resource in Malaysia was used to prepare activated carbon by physiochemical activation with potassium hydroxide (KOH)and carbon dioxide(CO_2) as the activating agents at 850 ∘Cfor 2 h. The adsorption equilibrium and kinetics of methylene blue dye on such carbon were then examined at 30 ∘C. Adsorption isotherm of the methylene blue (MB) on the activated carbon was determined and correlated with common isotherm equations. The equilibrium data for methylene blue adsorption well fitted to the Langmuir equation, with maximum monolayer adsorption capacity of 454.2 mg/g. Two simplified kinetic models including pseudo-first-order and pseudo-second-order equation were selected to follow the adsorption processes. The adsorption of methylene blue could be best described by the pseudo second-order equation. The kinetic parameters of this best-fit model were calculated and discussed.

In 2006 Ewa Lorenc-Grabowska, Gra_zyna Gryglewicz studied Adsorption characteristics of Congo Red on coal-based mesoporous activated carbon Adsorption of Congo Red dye (CR) on bituminous coal-based mesoporous activated carbon (AC) from aqueous solutions was studied. The ACs used differed significantly in terms of total surface area, pore volume distribution and surface charge properties. The mesopore contribution to the total pore volume ranged from 52 to 83%. The adsorption tests were performed under static conditions at solution pH 7.8e8.3. The pH at the point of zero charge (pHPZC) for ACs used was over 10. It was found that the higher the fraction of mesopores with a size between 10 and 50 nm, the shorter the time to achieve the equilibrium stage for CR adsorption. The kinetics of adsorption in view of two kinetic models,

i.e. the pseudo-second-order model and the intraparticle diffusion model, was discussed. The pseudo-second-order kinetic model describes the adsorption of CR on mesoporous activated carbon very well. The correlation coefficients ranged from 0.980 to 0.991. The equilibrium adsorption data were interpreted using Langmuir and Freundlich models. The adsorption of CR was better represented by the Langmuir equation. The monolayer adsorption capacity of ACs was found to increase with increasing both the mesopore volume and the mesopore contribution to their porous texture.

In 2007 R. A. Shawabkeh and E. S. M. Abu-Nameh studied Absorption of Phenol and Methylene Blue by Activated Carbon from Pecan Shells. Activated carbon is produced from pecan shells by chemical activation using phosphoric acid. This activation is followed by the treatment with sodium dodecyl sulfate to prepare the surface for the adsorption of phenol and methylene blue from aqueous solution. The results showed a great ability for methylene blue removal with sorption capacity of 410 mg/g at pH 9 and solution concentration of 35 mg/l, while moderate adsorption was obtained for phenol with a capacity of 18 mg/g at pH 11 and the same solution concentration. The increase or decrease in solution pH has a favorable effect on the sorption of both adsorbates. Langmuir and Freundlich models were used to fit the experimental data.

In 2009 Tabrez A. Khan, Imran Ali, Ved Vati Singh and Sangeeta Sharma studied the Utilization of Fly ash as Low-Cost Adsorbent for the Removal of Methylene Blue, Malachite Green and Rhodamine B Dyes from Textile Wastewater. Fly ash was utilized as a potential low-cost adsorbent for the removal of methylene blue, malachite green and rhodamine B from artificial textile wastewater. The adsorbent was characterized by its physico-chemical analyses,

porosity, surface area, ignition loss measurements and scanning electron micrograph. Adsorption studies were carried out in a batch process with different concentrations of dyestuffs, pH, temperature and contact time. The removal of methylene blue, malachite green and rhodamine B varied from 0.228 to 0.814, 0.219 to 0.644, and 0.184 to 0.618 mgg^{-1} respectively when the initial dye concentration was raised from 5 to 20 mgL^{-1}. The amount of dye adsorbed (mgg^{-1}) was found to increase with increase in the contact time; with 80 minutes for malachite green and rhodamine B and 100 minutes for methylene blue. The equilibrium data closely followed both Langmuir and Freundlich isotherms, but the latter isotherm fitted the data better.

CHAPTER-3
MATERIALS AND METHODS

Materials and methods

1. ADSORBENT:

Coconut shell activated carbon, is used as adsorbent for dye removal. It is a form of carbon that has been processed to make it extremely porous and thus to have a very large surface area available for adsorption or chemical reactions.

The word activated in the name is sometimes substituted by active. Due to its high degree of microporosity, just one gram of activated carbon has a surface area of 1000 m^2, as determined typically by nitrogen gas adsorption. The samples were washed several times and dried in an oven at 50°C overnight and was ground into fine powder form before being used.

Specifications:

Size 12x40 ASTM ,oversize 0.6%, undersize 0.3%

Iodine No. 1050 mgm/gm

Hardness 98.60%

Moisture 2.677%

Ash 2.15%

2. GLASSWARE AND APPARATUS USED

All glass wares (Conical flasks, Measuring cylinders, Beakers, Petri plates and Test tubes etc.) used are of Borosil/Rankem. The instruments and apparatus used throughout the experiment are listed below:

Table 1: List of Instruments used during the whole experiment their make and function

Instruments	Make	Function
Electronic weight balance	Sartorius	To measure weight
PH meter	EuTech Instruments	Measurement of pH
Spectrophotometer(UV/Vis)	Jasco(V-530)	absorbance
shaker	Environmental Orbital Shaker	To stir the content
oven	Shivaki	To dry the samples
shaker	Environmental orbital shaker incubator	To shake the samples in dye solution

3. ADSORBATE:

Mixture of three dyes each with conc. 50mg/lt is used for experiment purpose.

1.The malachite green dye used during was manufactured by Merck. It is a basic cationic dye.

The solution is green in colour. Chemical formula: $=C_{52}H_{54}N_4O_{12}$, Molecular Weight =
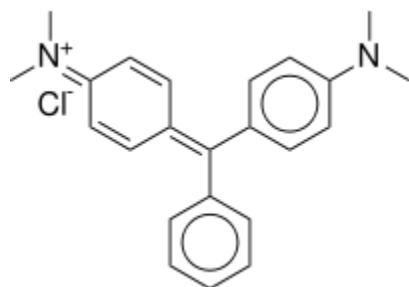
927.00. $\lambda_{max} = 617$nm.

Fig:1 Structure of malachite green

2. The methylene blue dye used was discovered by Caro in 1878.It is a basic cationic dye, heterocyclic aromatic chemical compound with molecular formula: $C_{16}H_{18}N_3SCl$, Molecular Weight=319.85. $\lambda_{max} = 663$nm.
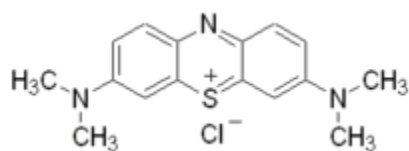
Fig:2 Structure of methylene blue

3. The Congo red dye was first synthesized in 1883 by Paul Bottiger who was working then for the Friedrich Bayer Company in Elberfeld, Germany. Due to a color change from blue to red at pH 3.0-5.2, congo red can be used as a pH indicator. It is the sodium salt of benzidinediazo-bis-1-naphthylamine-4-sulfonic acid (formula: $C_{32}H_{22}N_6Na_2O_6S_2$; molecular weight: 696.66 g/mol). It is a secondary diazo dye. $\lambda_{max} = 497$nm

Fig:3 Structure of congo red

Methods:

Adsorption Studies:

1. Effect of contact time:

150 ml of dye solution with dye concentration (50mg/L) is to be prepared in a conical flask with adsorbent concentration (0.5g/150ml) and kept inside the shaker. Dye concentration to be estimated spectrophotometrically at the wavelength corresponding to maximum absorbance, λ_{max}, using a spectrophotometer (JASCO UV/Vis-550). The samples to be withdrawn from the incubator shaker (Environmental orbital Shaker Incubator, DENEB INSTRUMENTS) at predetermined time intervals and the dye solution should be separated from the adsorbent by the help of a micropipette. The absorbance of solution is then measured. The dye concentration is to be measured after 5, 10, 20, 30,60, 90,120mins until equilibrium reaches. A graph is to be plotted with q_e vs time. The q_e is expressed as

$$q_e = \frac{C_0 - C_e}{X}$$

Where, q_e = Amount of dye adsorbed per unit mass of adsorbent (mg/g).

   C_0= Initial dye concentration (mg/L).

   C_e= Final dye concentration (mg/L).

   X = Dose of adsorbent (g/L).

2. Effect of initial pH:

   150ml of dye solution was prepared in a conical flask with dye conc. 50mg/L and adsorbent conc.(1g/150ml) and initial pH of the conical flask is to be measured. The pH of the dye solutions was adjusted with dilute HCl (0.05N) or KOH (0.05N) solution by using a pH meter (EUTECH Instrument, pH 510).150 ml of dye solution was prepared taking three dyes and the pH of solution is changed from 2 to 10.The flasks were put inside the incubator shaker

(120rpm fixed through out the study) maintained at $27^{o}C$ and the final concentration of dye was measured using UV spectrophotometer and the calibration plot of the dye after 2 hours. A graph is to be plotted with q_e vs initial pH.

3. Effect of adsorbent dose:

150ml of dye solution was prepared in different conical flasks with dye conc.(50mg/L) and adsorbent concentration 0.5,1,2,5,8g/150ml. The final dye concentration readings were taken after putting the 4 flasks inside the shaker for 2 hours. A plot of q_e vs adsorbent dose is taken.

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4. Effect of temperature:

150 ml of dye solution was prepared in conical flask with dye concentration 50mg/L and adsorbent dose (1g/L) and put inside the incubator shaker. The temperature was maintained at 20°C. The final dye concentration readings were taken at 5, 10, 20, 30, 60,120mins. The same procedure was followed for temperatures 30°C and 40° C.

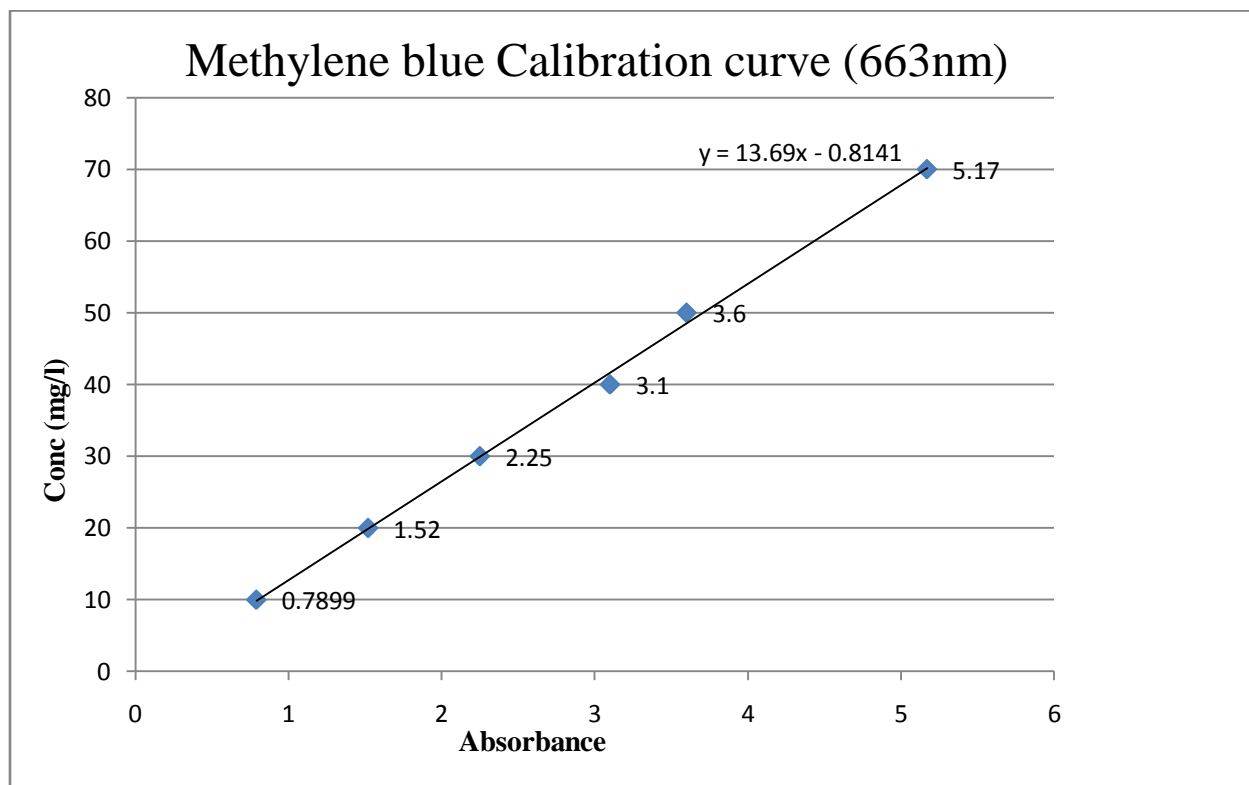A plot of q_e vs time at different temperatures is obtained.
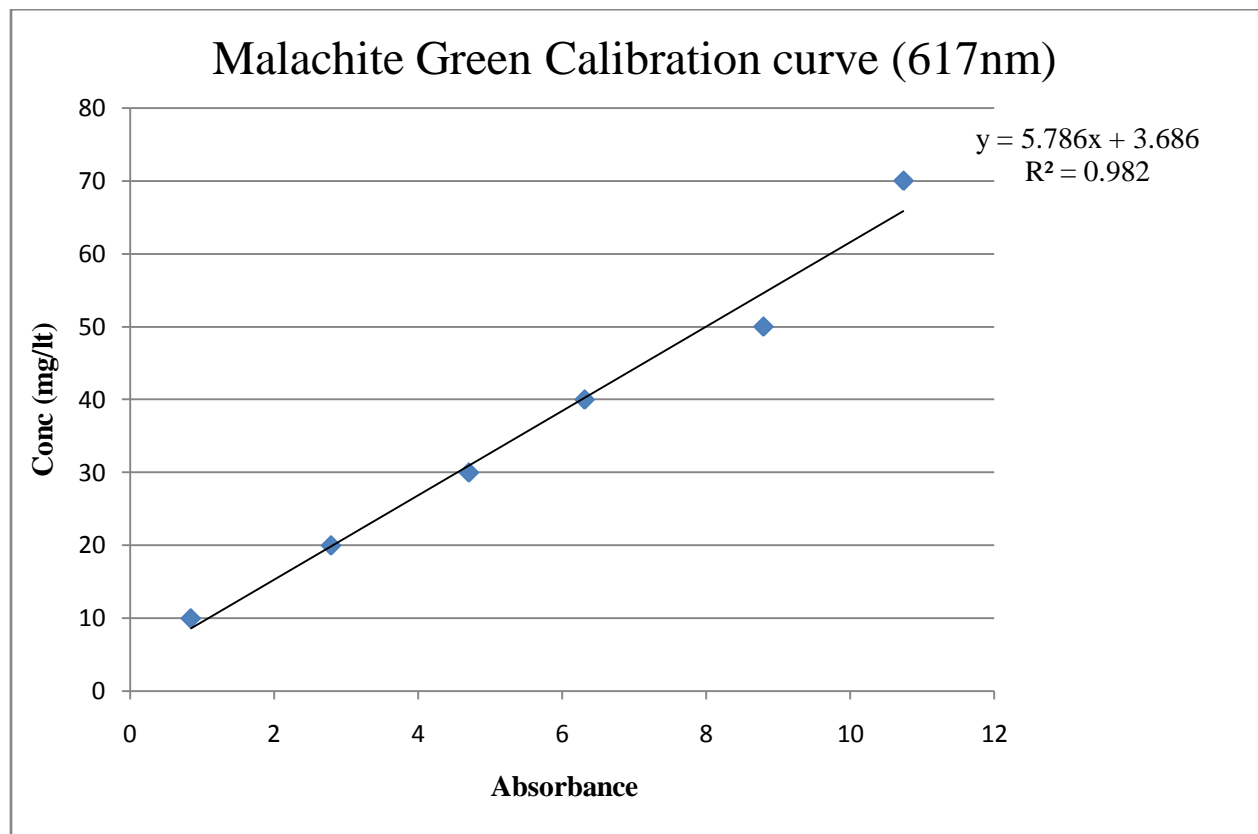
CHAPTER-4
RESULTS AND DISCUSSIONS
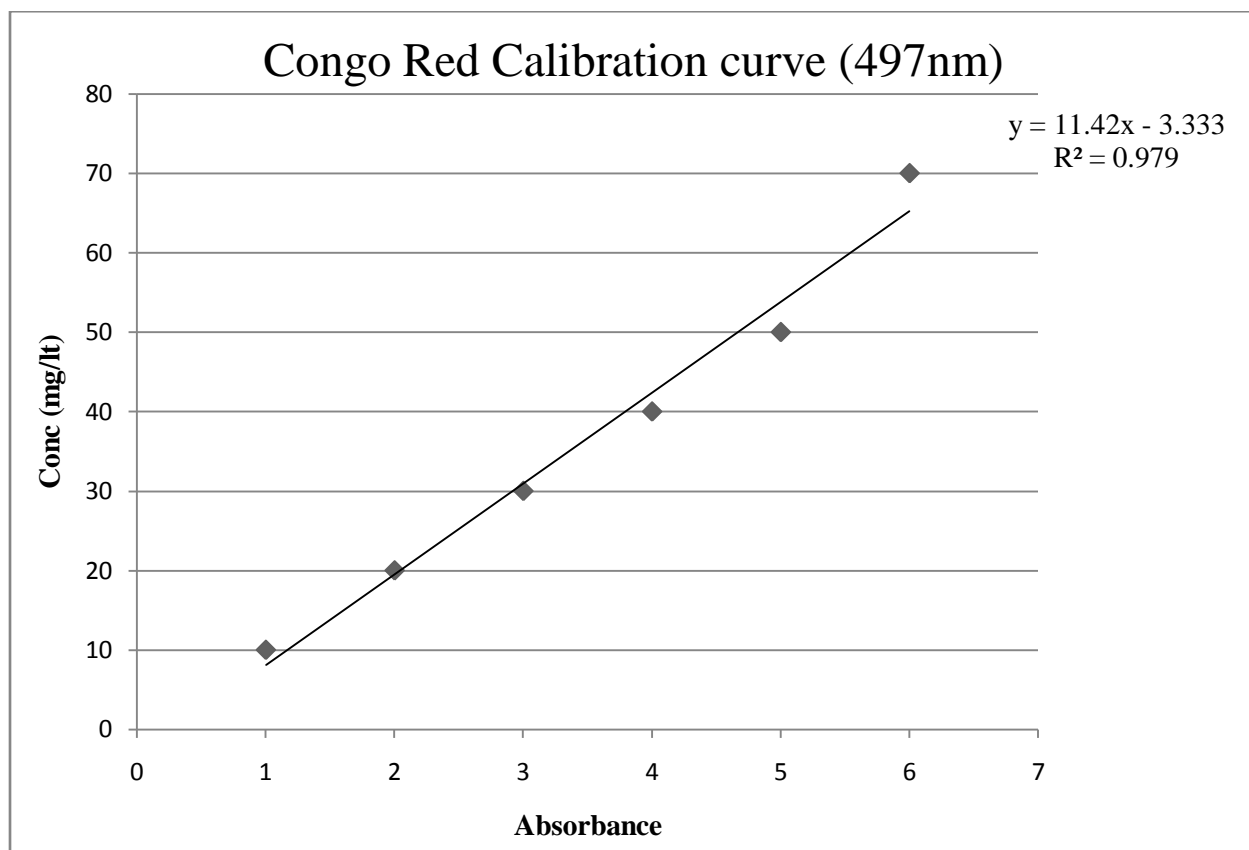
RESULTS AND DISCUSSIONS:

1.CALIBRATION PLOTS :

For Methylene blue dye:

Methylene blue Calibration curve (663nm)

y = 13.69x - 0.8141

For Malachite green dye:

Malachite Green Calibration curve (617nm)

$y = 5.786x + 3.686$
$R^2 = 0.982$

Conc (mg/lt) vs Absorbance

For Congo red dye:

Congo Red Calibration curve (497nm)

y = 11.42x - 3.333
R² = 0.979

Conc (mg/lt)

Absorbance

4.1 Adsorption Studies:

4.1.1 Effect of Contact time:

The effect of contact time can be seen from Fig.4.1.1.1. for the dyes. It is clear that the extent of adsorption is rapid in the initial stages and becomes slow in later stages till saturation is allowed. The final dye concentration did not vary significantly after 2 hours from the start of adsorption process. This shows that equilibrium can be assumed to be achieved after 2 hours (120 min). It is basically due to saturation of the active site which do not allow further adsorption to take place .
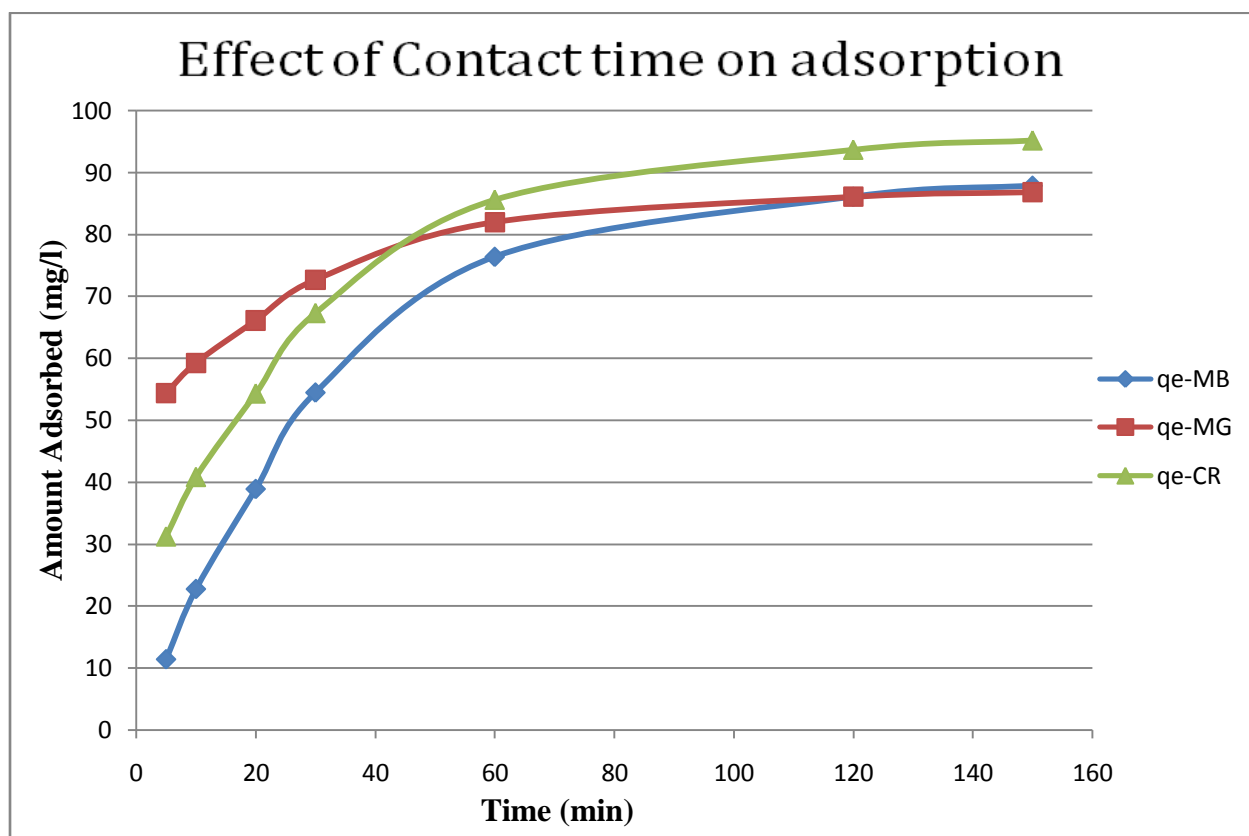

Fig:4.1.1.1 Effect of contact time on adsorption of dye solution on Activated carbon at Co-50 mg/L, Temp- Room Temperature, pH- solution pH, Speed-120 rpm, adsorbent dosage- 0.5 g/150ml

4.1.2 Effect of initial pH of the solution:

The effects of initial pH on dye solution of three dyes removal were investigated by varying the pH from 2 to 10. At pH - 2 the removal was minimum but it increased along with increasing initial pH of dye solution. For malachite green it was maximum at pH = 9 as we see in the fig 4.1.2.1. In case of methylene blue higher the pH, greater is removal by adsorption .For congo red there is no significant change in amount adsorbed after pH 7. Infact adsorption found to decrease with increase in pH of solution. The adsorption of these positively charged dye groups on the adsorbent surface is primarily influenced by the surface charge on the adsorbent which in turn is influenced by the solution pH. The result showed that availability of negatively charged groups at the adsorbent surface is necessary for the adsorption of basic dyes to proceed which we see at pH -2 is almost unlikely as there is a net positive charge in the adsorption system due to the presence of H_3O^+ . Thus as the pH increased, more negatively charged surface was available thus facilitating greater dye removal .We see that the trend is increasing with increasing pH.
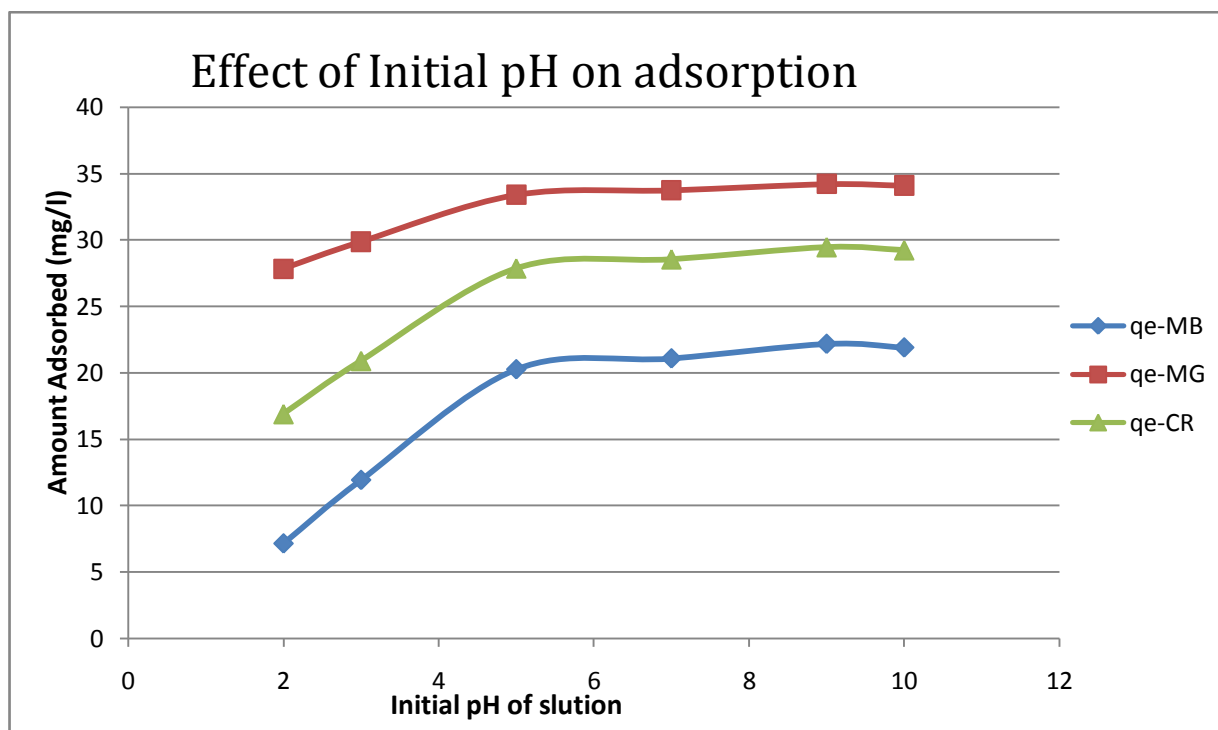
Fig: 4.1.2.1 Effect of initial pH of the Dye solution at Co-50 mg/L, Temperature- room temperature, speed- 120 rpm, Adsorbent dosage-1 g/150ml

4.1.3 Effect of adsorbent dosage:

From fig 4.1.3.1 we see that the optimum dose for the dye is 6g/150ml. Though at 8g/150ml, there is slight increase in q_e value but if we get nearly the same result as we get at adsorbent dosage of 5g/150ml then going for 8g/150ml will be expensive and loss of adsorbent. It is obvious as with increasing amount the active sites for adsorption of mixture of three dyes

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increases which results in an increase in removal efficiency. The decrease in adsorption capacity with an increase in the adsorbent concentration could be ascribed to the fact that some of the adsorption sites remained unsaturated during the process.

Effect of Adsorbent dose on adsorption

Malachite Green----->

Congo Red

<-----Methylene blue

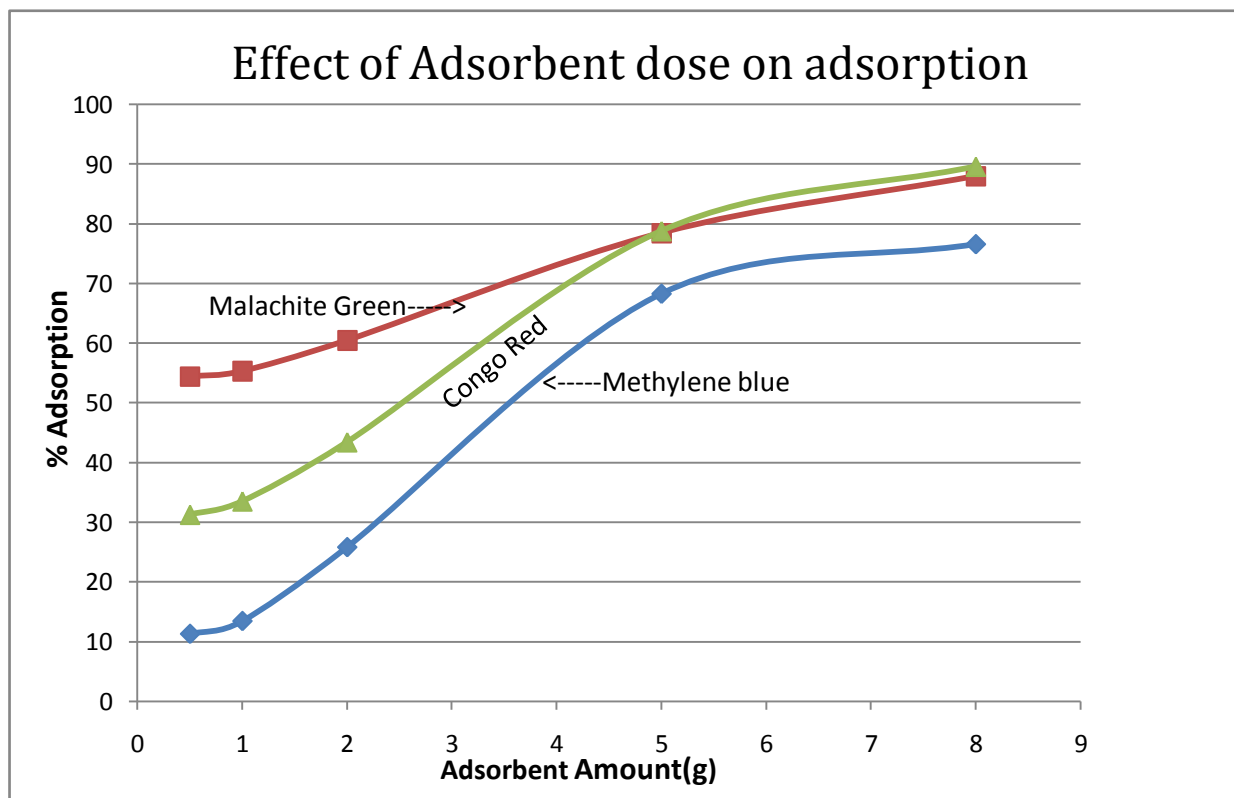% Adsorption

Adsorbent Amount(g)

Fig: 4.1.3.1 Effect of Amount of activated carbon on dye solution adsorption at Co- 50 mg/L, pH- solution pH, Temperature- room temperature, Speed-125 rpm.

4.1.4 Effect of Temperature:

The effect of temperature on adsorption of dye solution with initial concentration of 50mg/L at pH=solution pH at temperatures 20, 30 and 40°C on has been determined. The result of time rate studies for the adsorption of the three dyes malachite green, methylene blue and congo red at different temperature has been shown in the figures below.
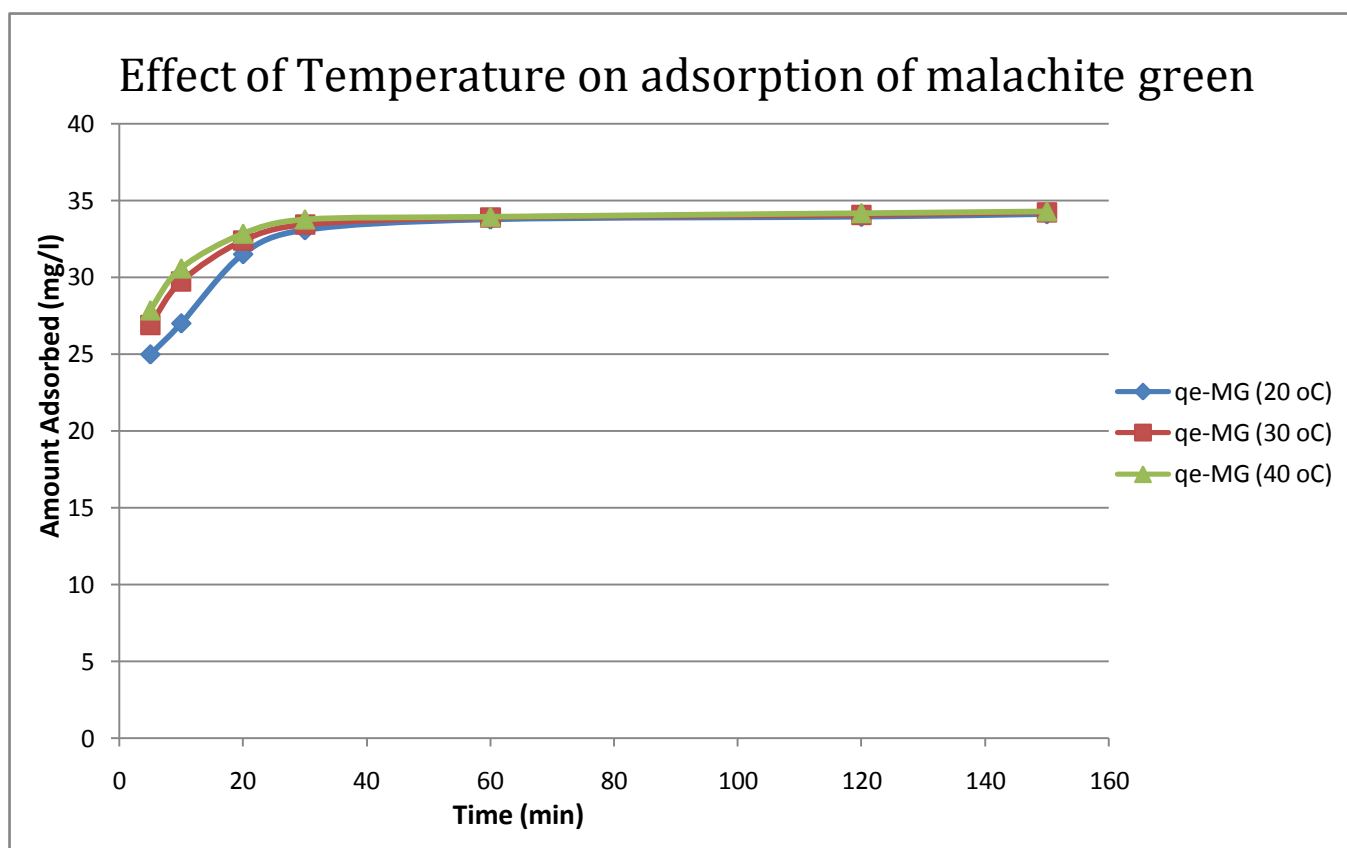

Fig: 4.1.4.1 Effect of Temperature on Malachite Green adsorption on Activated carbon at Co- 50 mg/L, pH-solution pH, speed-120 rpm and adsorbent dosage-1g/150ml
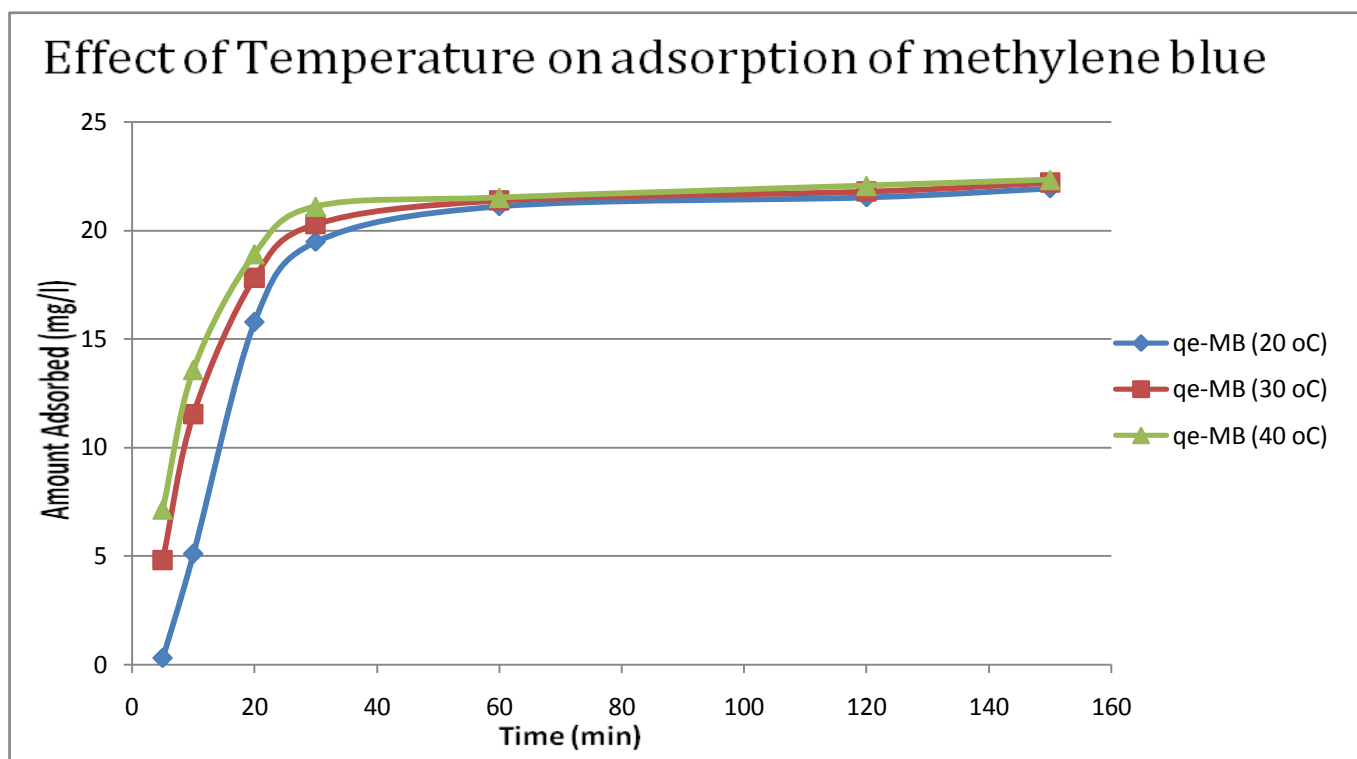
Fig: 4.1.4.2 Effect of Temperature on Methylene Blue adsorption on Activated carbon at Co- 50 mg/L, pH-solution pH, speed-120 rpm and adsorbent dosage-1g/150ml
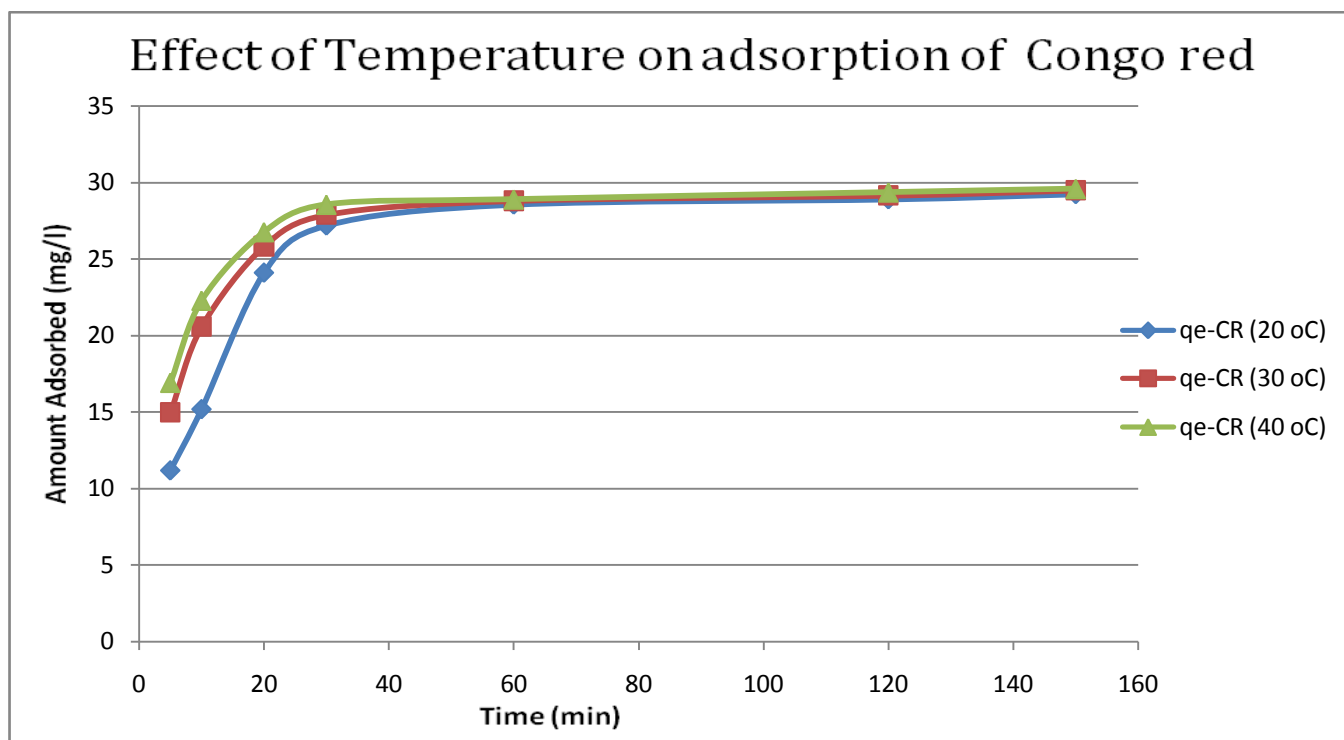
Fig: 4.1.4.3 Effect of Temperature on Congo Red adsorption on Activated carbon at Co- 50 mg/L, pH-solution pH, speed-120 rpm and adsorbent dosage-1g/150ml

Results indicate that the adsorption capacity of activated carbon for the three dyes (methylene blue, malachite green and congo red) increased with temperature. This may be a result of increase in the mobility of the large dye ion with temperature. An increasing number of molecules may also acquire sufficient energy to undergo an interaction with active sites at the surface. Furthermore, increasing temperature may produce a swelling effect within the internal structure of the activated carbon enabling large dyes to penetrate further.

CHAPTER-5
CONCLUSION

CONCLUSION

Removal of dyes, mixture of methylene blue, malachite green and congo red from aqueous solutions by adsorption with activated carbon has been experimentally determined and the following observations are made:

- The percentage of colour removed increase with increasing adsorbent dosage, increase with increasing contact time and varied with dye solution pH.

- The adsorption rates increases with increasing temperatures.

- Optimum contact time for equilibrium to be achieved is found to be 2 hours (120 min). It is basically due to saturation of the active site which do not allow further adsorption to take place.

- For malachite green maximum adsorption found to be at pH = 9. In case of methylene blue higher the pH, greater is removal by adsorption .For congo red there is no significant change in amount adsorbed after pH 7. Infact adsorption found to decrease with increase in pH of solution. The adsorption of these positively charged dye groups on the adsorbent surface is primarily influenced by the surface charge on the adsorbent which in turn is influenced by the solution pH.

- The result showed that availability of negatively charged groups at the adsorbent surface is necessary for the adsorption of basic dyes to proceed which we see at pH -2 is almost unlikely as there is a net positive charge in the adsorption system due to the presence of H_3O^+ $^.$Thus as the pH increased, more negatively charged surface was available thus facilitating greater dye removal .

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- Optimum adsorbent dose for the dye is 6g/150ml. It is obvious as with increasing amount the active sites for adsorption of mixture of three dyes increases which results in an increase in removal efficiency. The decrease in adsorption capacity with an increase in the adsorbent concentration could be ascribed to the fact that some of the adsorption sites remained unsaturated during the process and agglomeration of activated carbons as a result all the surface area is not available for adsorption process.

- Optimum temperature is 30 ^{o}C .The adsorption capacity of activated carbon for the three dyes (methylene blue, malachite green and congo red) increased with temperature. This may be a result of increase in the mobility of the large dye ion with temperature. An increasing number of molecules may also acquire sufficient energy to undergo an interaction with active sites at the surface. Furthermore, increasing temperature may produce a swelling effect within the internal structure of the activated carbon enabling large dyes to penetrate further.

CHAPTER-6
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