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STUDY ON THE REMOVAL CHARACTERISTICS OF PHENOL FROM AQUEOUS SOLUTION BY COAL FLY ASH

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ABSTRACT

Coal fly ash from Suratgarh and Kota Thermal Power Stations, Rajasthan was used to effectively remove phenol from aqueous solution. Batch studies were performed to evaluate the adsorption process and the effect of contact time, pH, adsorbent dosage and initial phenol concentration on removal process was investigated. Equilibrium adsorption data were correlated with Langmuir and Freundlich adsorption isotherm models. The adsorption data were best fitted for Freundlich model than Langmuir model. Adsorption capacity K (mg/g) was found to be 4.00 x 10^{-2} *mg/g for fly ash from Suratgarh thermal power station and 3.60 x* 10^{-2} *mg/g for fly ash from Kota thermal power station. Fly ash from Suratgarh Thermal power station has comparatively higher adsorption capacity due to smaller particle size and larger surface area as examined by particle size analysis of fly ash samples. This work proved that fly ash from Rajasthan thermal power stations can be used as an effective low-cost adsorbent for the removal of phenol from aqueous solution.*

Keywords - ***Coal Fly Ash, Phenol, Freundlich Isotherm Model, Adsorption Capacity***

I. INTRODUCTION

Phenolic pollution due to wide distribution and detection of phenol in the effluent of various industrial operations is a very important area of research, since phenolic wastewater is harmful for aquatic and human life. According to the standard set by United State Environmental Protection Agency (USEPA) surface water must contain less than 1 microgram/litre [1] phenol whereas effluents discharged in water bodies contain 3-10,000 mg/litre of phenol which is objectionable and toxic to animal and plant life [2]. Phenols have been considered as priority organic pollutants by the US environmental protection agency (EPA) and can cause considerable damage to the environment [3]. Phenol is potential human carcinogen and is fatal by ingestion or skin absorption and may cause severe irritation to eyes and respiratory tracts. As it is a highly harmful compound, there is a necessity to develop improved, cost effective and environmentally safe disposal methods for proper treatment of industrial effluents containing phenols prior to their discharge into the environment [4].Various treatment processes available for the removal of phenols include: ion exchange solvent extraction, oxidation, phase transfer catalysis, biological treatment, hot gas or steam stripping, granular activated carbon processes and reverse osmosis, the electro-Fenton method, combined applications of flotation and coagulation processes [5-8]. But most of these methods often suffer from some serious drawbacks including high capital and operational cost, regeneration cost and formation of hazardous by-products which create disposal problems. Among various methods used in phenolic wastewater treatment, liquid – phase adsorption has been shown to be an efficient and well-established technique due to its simplicity of design, ease of operation, effectiveness and the availability of a wide range of adsorbents. Granular/powdered activated carbon as adsorbent is often preferred for the removal of organic compounds due to its high adsorption capacity, however due to high cost and the irreversible nature of adsorption other alternative low cost adsorbents suggested become very attractive from a cost point of view [9, 10]. Therefore, materials which are produced as agricultural or industrial by-products such as rice husk, saw dust, vegetable and fruit peels, clay, prawn waste, carbonized bark, peat, fly ash, bentonite, zeolite, leaves, etc. have attracted attention of researchers owing to their easy availability and cost effectiveness [11-13].The potential of fly ash, a by-product of thermal power plants, as a natural adsorbent for adsorption of phenol has been investigated by many researchers [14-16], thereby not only reducing its harmful impact on the environment but also solving the problem of disposal associated with its massive production in our country.The high carbon content, adsorptive capacity attributed to its hydrophilic surface which is highly porous with large surface area and pore volume owing to the presence of high amounts of SiO_2 and Al_2O_3 makes fly ash a good candidate for adsorption of phenols and other organic compounds. Therefore, coal fly ash can be investigated as a suitable low-cost adsorbent for adsorption of phenol from aqueous solution thereby minimizing the adverse impacts of industrial wastewaters as well as large amounts of coal fly ash.

In this paper, batch experiments were designed for the adsorption process, and the effects of contact time, pH, initial phenol concentration and adsorbent dosage on adsorption process were evaluated. The optimum removal conditions were also discussed for phenol removal.

II. MATERIALS AND METHODS

Coal fly samples collected from Suratgarh and Kota Thermal Power Stations, Rajasthan were dried in the electric oven at 120 ^{0}C for at least 24 hours and stored in vacuum desiccators for further use without any pre-treatment. Phenol of Analytical grade supplied by Merck, India was used as an adsorbate. The stock solutions were prepared in deionized water. Fresh solutions were prepared on a daily basis with the initial concentration and pH of the adsorbate calculated before the start of any experiment. The standard calibration curve of known concentrations of phenol was plotted by finding out the absorbance at the characteristic wavelength of λmax 269.4nm using UV-visible spectrophotometer (Hitachi V-500 UV/VIS double beam). The final phenol concentration was evaluated spectrophotometrically from the calibration curve at 269.4 nm. The equilibrium adsorption uptake Qe (mg/g) and percentage removal of phenol from the aqueous solution was determined using the following equations:

$$Qe = V\,(Ci - Ce)\,/m \qquad \text{(Eq 2.1)}$$

$$\%\ Removal = 100\,(\,Ci - Ce\,)/\,Ci \qquad \text{(Eq 2.2)}$$

Where Ci is initial adsorbate concentration (mg/l), Ce is equilibrium adsorbate concentration (mg/l), V is the volume of solution (l), and m is the mass of adsorbent (g).

III. RESULTS AND DISCUSSIONS

3.1 Characterization of the Fly Ash Samples

3.1.1 Chemical Composition of Fly Ash

Chemical composition of fly ash samples for assessing the quality of fly ash was analysed for following inorganic compounds viz. silicon dioxide, aluminium trioxide, ferric oxide, calcium oxide, sulphur trioxide and loss on ignition (LOI) by standard methods of complexometric titration and gravimetric analysis. For the sake of easy reference fly ash from Suratgarh(TPS-1) and Kota(TPS-2) thermal power station are denoted as FA-1 and

FA-2 respectively. The chemical composition of the fly ashes collected from Suratgarh and Kota thermal power stations are given in Table 3.1.

Table 3.1: Chemical Composition of Suratgarh (TPS-1) and Kota (TPS-2) Fly Ash

Components or Property	Suratgarh Fly ash (%)	Kota Fly ash (%)
Silicon dioxide (SiO_2)	58.23	56.07
Aluminium trioxide (Al_2O_3)	29.11	26.69
Ferric oxide (Fe_2O_3)	5.72	8.03
Calcium oxide (CaO)	1.3	1.2
Sulphur trioxide (SO_3)	0.67	0.18
Loss on Ignition (LOI)	0.13	0.8

It is evident from the above results that the fly ash is predominantly composed of SiO_2 and Al_2O_3 with small amounts of Fe_2O_3 which together account for 93.06%and 90.79% by mass of the total ash content from TPS-1 and TPS-2 respectively. CaO content of fly ash has a relatively low value of 1.3 and 1.2 for TPS-1 and TPS-2. According to the ASTM C618, this fly ash can be classified as class F for having a less than 10% CaO content and a greater than 70% content of SiO_2, Al_2O_3 and Fe_2O_3 altogether [17]. The loss on ignition (LOI), a measure of unburnt carbon in the fly ash was reported to be having a low value of 0.13 and 0.8 for TPS-1 and TPS-2 respectively. These LOI values can be used as an indicator for the efficiency of the combustion chamber at the thermal power station.

3.1.2 Particle Size Analysis

Particle size distribution of fly ash samples was analysed using particle size analyser (CILAS PARTICLE SIZE ANALYSER 920L) which provides diffraction data and stores it as volume percentage against the particle size as shown in Fig 3.1 and 3.2.

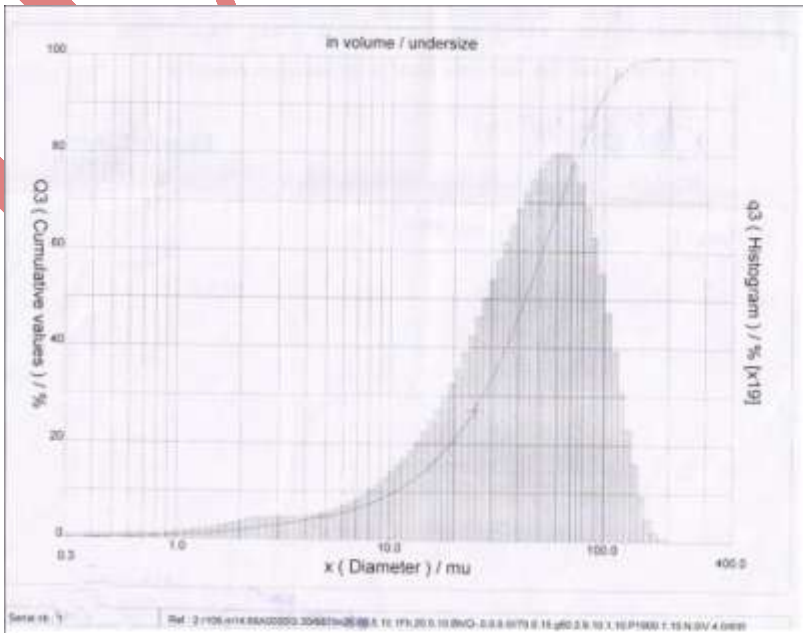

Fig 3.1. Particle Size Distribution of Fly Ash from TPS-1

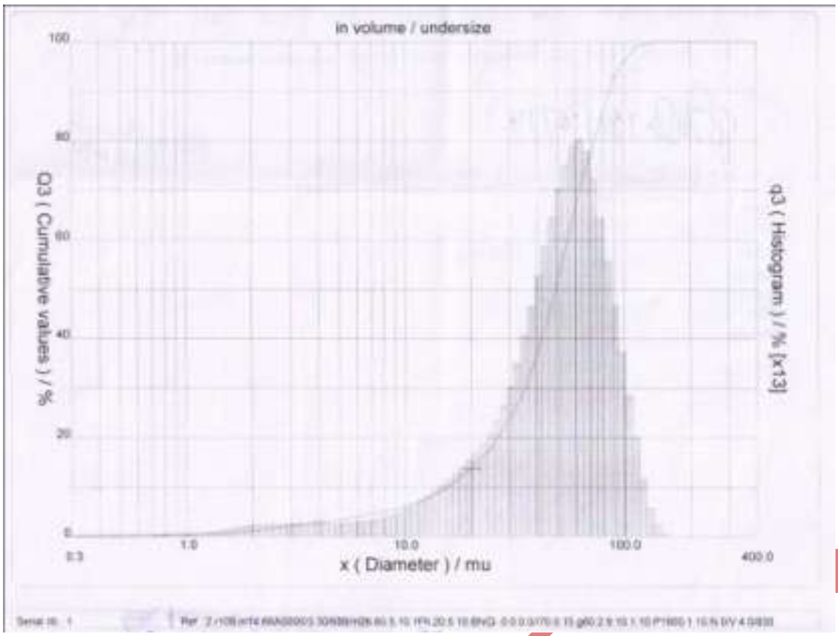

Fig 3.2. Particle Size Distribution of Fly Ash from TPS-2

Results of particle size analysis of fly ash samples collected from TPS-1 and TPS-2 are represented in Table 3.2:

Table 3.2: Particle Size Analysis of FA-1 and FA-2

Sample	**Particles finer than (Vol %)**				**Diameter (µm) at**				
	10 (mu)	5 (mu)	2 (mu)	1 (mu)	10 Vol% of particles (d_{10})	50Vol% of particles (d_{50})	90 Vol% of particles (d_{90})	100 Vol% of particles (d_{100})	Mean Dia (mu)
TPS-1	9.34	4.84	1.88	0.78	10.60	42.79	92.19	180.00	48.35
TPS-2	6.19	3.60	1.27	0.40	15.35	48.74	85.90	140.00	50.52

Particle size distribution shows weather a material is fine, coarse, well graded or poorly graded and helps in the classification of the coal ashes. The particle size distribution of fly ash mainly depends on firing condition, initial pulverization of coal and sedimentation in lagoons, which has the greatest influence on the grading of fly ash [18]. Granulometric data for fly ashes and their size fractions in Table 3.2 show that $d_{(90)}$ and $d_{(50)}$ values are 92.19 and 42.79 for FA-1 and 85.90 and 48.74 for FA-2, respectively. The particle size of the fly ash from TPS-1 ranges from 0.30 to 180 µm and from TPS-2 ranges from 0.30 to 140 µm. It can be seen that there are some significant differences in particle size distribution of both the fly ashes. Fly ash from TPS-1 has a higher percentage of finer particles than TPS-2 as indicated by granulometric data in Table 3.2. The size of fly ash particles plays an important role in determining its adsorptive capacity. It was observed that for effective adsorption smaller particle size and larger surface area gives higher adsorbate removal at equilibrium [19]. With smaller particle size, equilibrium is more easily achieved and nearly full adsorption capability can be attained owing to reduction in internal diffusion and mass transfer limitation for penetration of the adsorbate into the adsorbent. The effective specific surface area decreases with increasing particle size, as a result of which the saturation adsorption per unit mass of the adsorbent will also decrease [20]. Therefore, FA-1 exhibited higher adsorption capacity in comparison to FA-2 as increase in percentage of finer particles in FA-1 resulted in the increase in total surface area which provided more active sites for the adsorption of phenol at smaller particle sizes [21, 22].

3.2 Batch Studies

3.2.1 Influence of Contact Time

The effect of contact time on the adsorption of phenol using fly ash was studied by varying the contact time from 30 to 180 minutes at an adsorbent dosage of 20 g/l and initial phenol concentration of 100 mg/l.

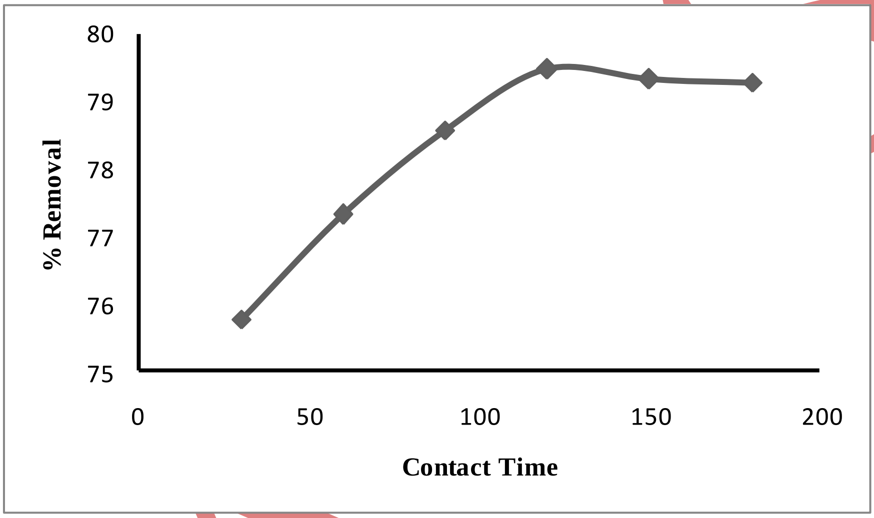

Figure 3.3 Effect of contact time on % Removal of phenol by FA-1 (Initial phenol concentration of 100mg/l and fly ash dosage of 20g/l)

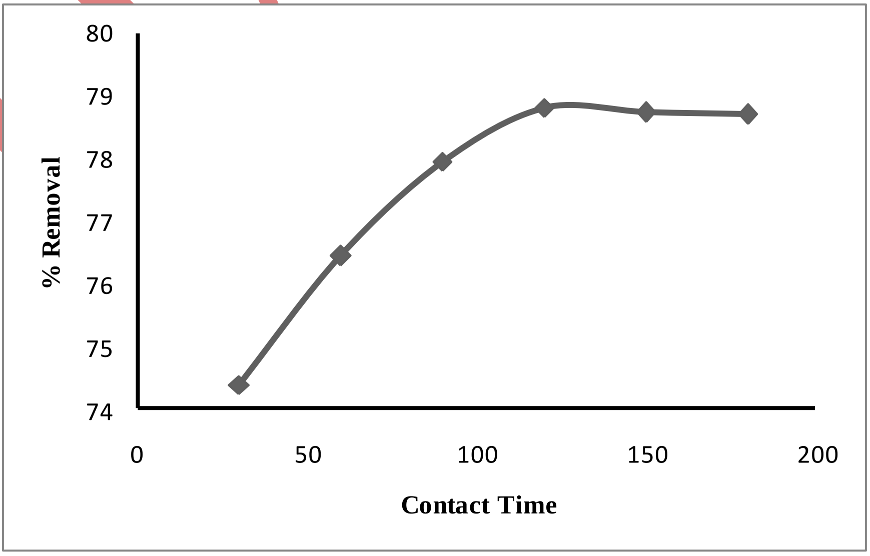

Figure 3.4 Effect of contact time on % Removal of phenol by FA-2 (Initial phenol concentration of 100mg/l and fly ash dosage of 20g/l)

It can be seen from fig. 3.3 and 3.4 that the amount of phenol adsorbed onto fly ash increases with time and about 75.76% and 74.38% of phenol had been removed by FA-1 and FA-2 respectively, within the first thirty minutes of agitation. The results showed that equilibrium time required for the adsorption of phenol on both types of fly ash was almost 120 minutes (2 h), after which the process almost approaches equilibrium with smooth and continuous curves leading to saturation. More than 70% of phenol was removed within the first thirty minutes of agitation as a result of the large number of vacant sites existing on the surface of the fly ash, available for adsorption during the initial agitation stages. Richa et al. reported that an equilibrium time of 2 h was necessary for the maximum removal of phenol [23]. Phenol removal increases with increasing mixing contact time and attains equilibrium in 120 min was reported by Ihsan et al. using sawdust as an adsorbent [24].

3.2.2 Influence of pH

Solution pH has a significant effect on the uptake of phenol, since it determines the surface charge of fly ash and the degree of ionization and speciation of the adsorbate in solution [25]. Figure 3.5 and 3.6 shows the effect of % removal of phenol on FA-1 and FA-2 at pH values ranging from 4-9 at an adsorbent dosage of 20 g/l and initial phenol concentration of 100 mg/l. The percent removal increased from pH 4 to 6 and decreased from pH 6 to 9.The maximum removal occurs at pH 6 and thereafter a significant decline in removal efficiency was observed with further increase in pH. The adsorbed amount decreased with increasing pH value, which can be attributed to the phenol ionization to form phenolate ions and at the same time the presence of hydroxyl ions on the adsorbent preventing the uptake of phenolate ions [26].

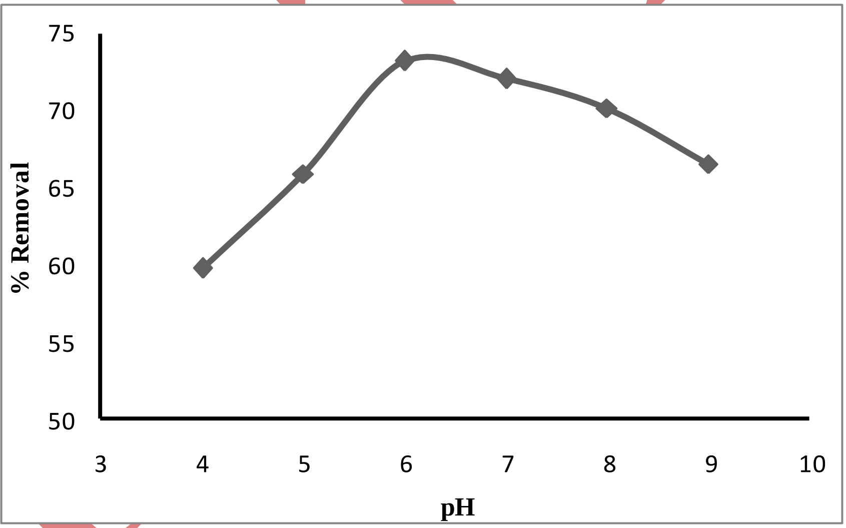

Figure 3.5 Effect of pH on % Removal of phenol by FA-1 (Initial phenol concentration of 100mg/l and fly ash dosage of 20g/l)

Hence, the results given above suggest that phenol removal efficiency decreased with increasing pH value, with maximum removal of 73.13% and 72.67% at pH 6 by FA-1 and FA-2 respectively. Therefore, from the above results, pH 6.0 was selected as the optimum pH for further experiments. Sunil et al. observed % removal of phenol to be maximum at corresponding pH value of 6.5 [27]. Similar behaviour has been reported during the adsorption of phenol onto activated carbon by Ekpete et al. [28], onto agriculture waste by Tagreed [29] and onto rise husk by Mahvi et al. [30].

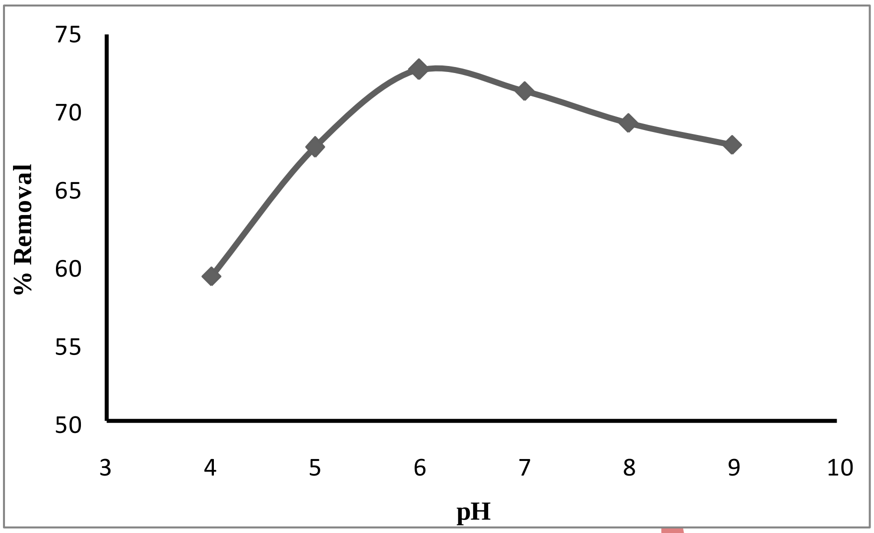

Figure 3.6 Effect of pH on % Removal of phenol by FA-2 (Initial phenol concentration of 100mg/l and fly ash dosage of 20g/l)

3.2.3 Influence of Adsorbent Dosage

The influence of fly ash dosage on phenol removal was investigated by conducting the experiments at adsorbent dosages of 10 to 60 g/l at pH 6 and initial phenol concentration of 100 mg/l. Figure 3.7 and 3.8 show the influence of FA-1 and FA-2 on the % removal of phenol. It was found that the phenol removal increased by increasing adsorbent dosage. After equilibrium, it was found that 88.73% and 82.92% removal was obtained by FA-1 and FA-2 respectively at 40 g/l of adsorbent dosage.

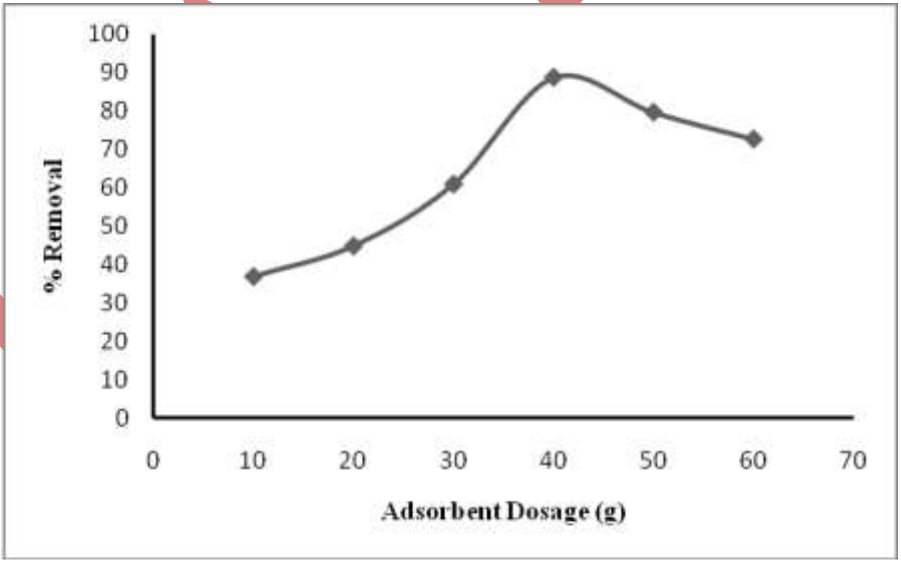

Figure 3.7 Effect of Adsorbent Dosage on % Removal of phenol by FA-1 (Initial phenol concentration of 100mg/l, pH 6 and contact time of 2h)

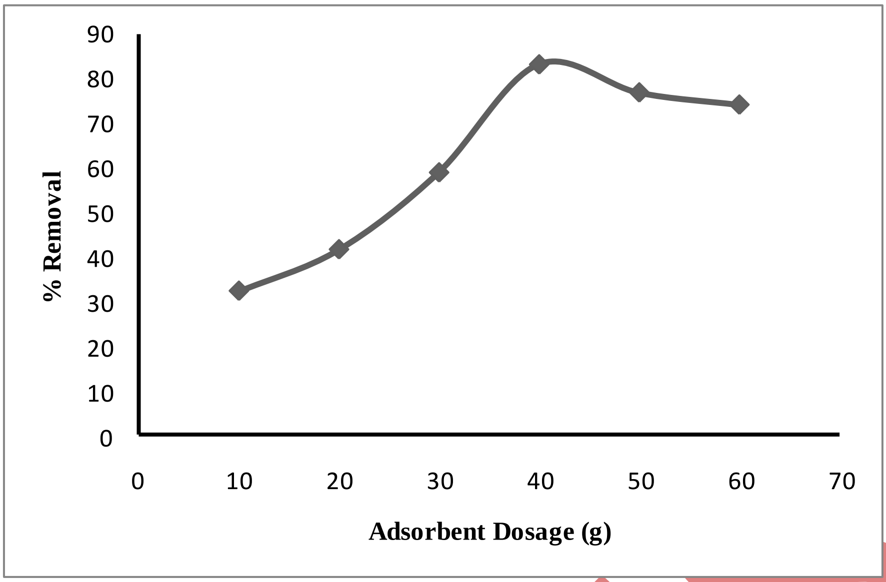

Figure 3.8 Effect of Adsorbent Dosage on % Removal of phenol by FA-2 (Initial phenol concentration of 100mg/l, pH 6 and contact time of 2h)

Initial increase of percentage removal can be attributed to the increase in surface area as well as active sites for adsorption resulting from the increased adsorbent dosage [31]. However at higher adsorbent doses overcrowding of phenol molecules may take place thereby preventing the diffusion through the actual adsorption sites [32]. Bhumica et al. observed that removal of phenol increased initially up to 40 g/l of fly ash and decreased on further increase in dosage [33]. Therefore, considering Qe and R%, adsorbent dose of 40.0 g/l was found to be the optimum fly ash dosage and was used for further experiments.

3.2.4 Influence of Initial Phenol Concentration

The initial phenol concentration provides an important driving force to overcome all mass transfer resistances of adsorbate between the aqueous and solid phases. The effect of initial phenol concentration on the phenol removal is shown in Figure 3.9 and 3.10 by FA-1 and FA-2 respectively. Percentage of phenol removal decreases with increasing concentration from 20 to 210 mg/l for both the types of fly ashes. The % removal of phenol decreased from 90.28% to 55.76% and 88.59% to 60.61% as the initial phenol concentration increased from 20 to 120 mg/l for FA-1 and FA-2 respectively.

At the initial conc. of 20 mg/l high amount of phenol is removed due to an increased number of vacant sites on the adsorbent available at the initial stage. As concentration of initial phenol is increased there is a decrease in percentage removal of phenol due to the accumulation of adsorbate particles on the surface of fly ash. Similar behaviour was also reported by Singh et al. [34] and Uddin et al. [35]. Since percentage removal starts to decrease after 20 mg/l indicating less favourable sites in adsorption [36], therefore 20 mg/l was fixed as the optimum initial phenol concentration for sorption experiments.

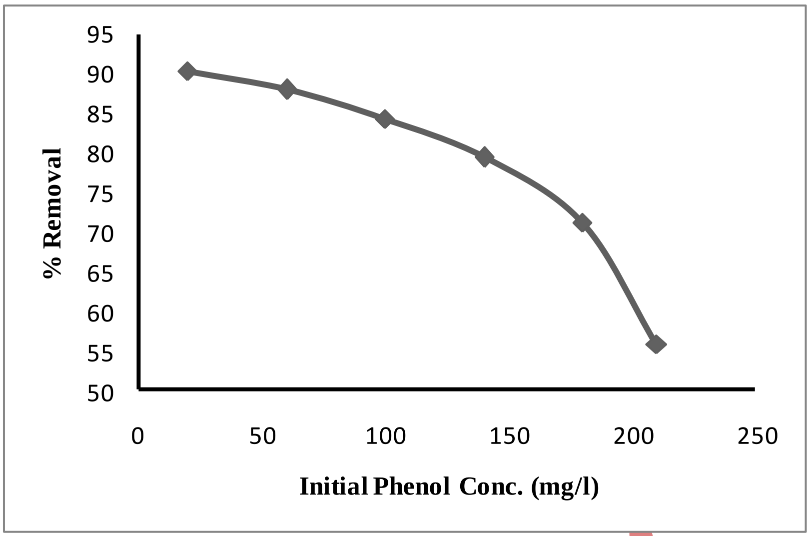

Figure 3.9 Effect of Initial Phenol Conc. on % Removal of phenol by FA-1 (Adsorbent dosage of 40 g/l, pH 6 and contact time of 2h)

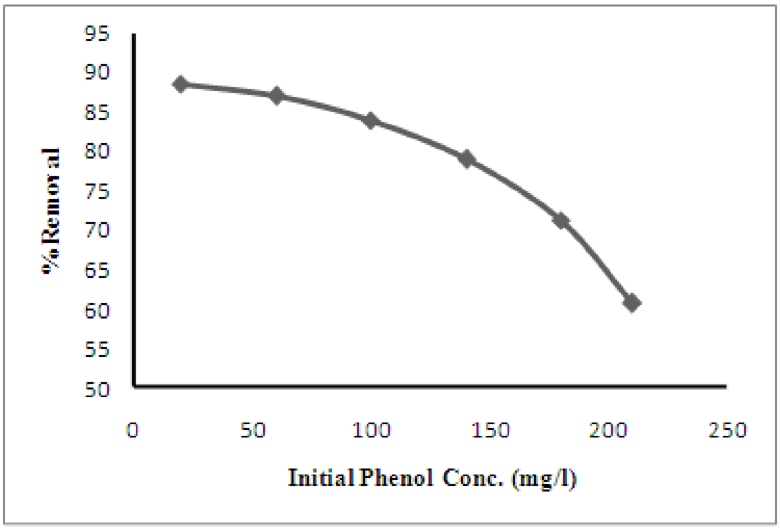

Figure 3.10 Effect of Initial Phenol Conc. on % Removal of phenol by FA-2 (Adsorbent dosage of 40 g/l, pH 6 and contact time of 2h)

3.3 Adsorption Equilibrium Study

3.3.1 Langmuir isotherm model

Langmuir isotherm describes quantitatively the formation of a monolayer adsorbate on the outer surface of the adsorbent containing a finite number of identical sites, and after that no further adsorption takes place. Thereby, it represents the equilibrium distribution of phenol between the solid and liquid phases [37]. Langmuir equation is represented as:

$$1/Qe = 1/Qo + (1/b_LQo)\ (1/Ce) \qquad \text{Eq. 3.1}$$

where,

Ce = equilibrium concentration of adsorbate in the solution (mg/l)
Qe = amount of adsorbate adsorbed per mass of adsorbent at equilibrium (mg/g)
Qo = maximum adsorption capacity

b_L = adsorption equilibrium constant related to the sorption energy between the adsorbate and adsorbent (l/mg)

Langmuir isotherms were obtained by agitating the adsorbent of fixed dosage at different initial phenol concentrations for a contact time greater than equilibrium time. Thus a plot of Ce/Qe vs Ce should be linear if Langmuir adsorption were operative, permitting the calculation of maximum amount of solute (Qo) adsorbed from the isotherm parameters. The experimental data obtained in this study seems to fit using Langmuir isotherm.

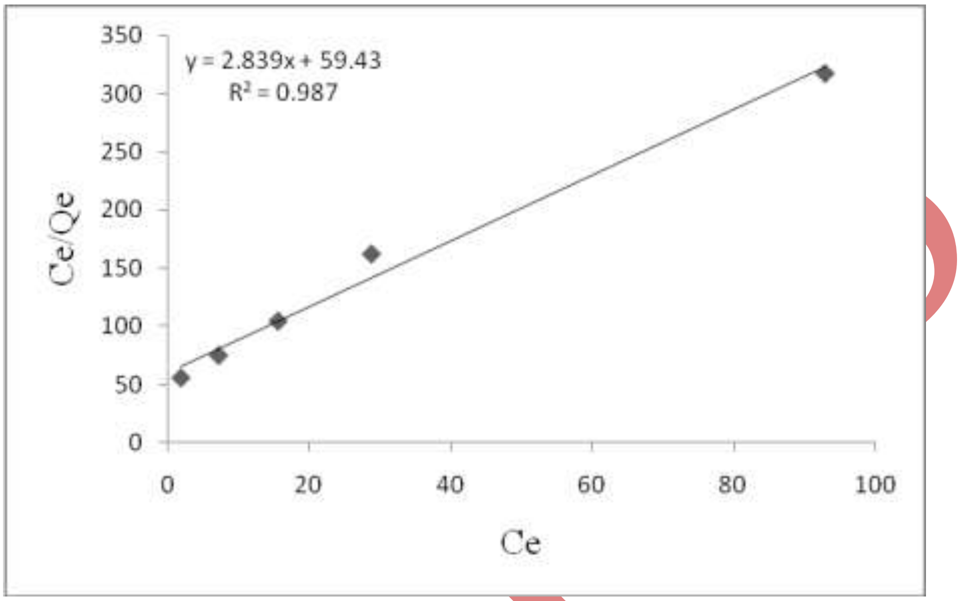

Figure 3.11 Langmuir adsorption isotherm of phenol by FA-1 (pH 6, adsorbent dosage 40 g/l and contact time 2 hours)

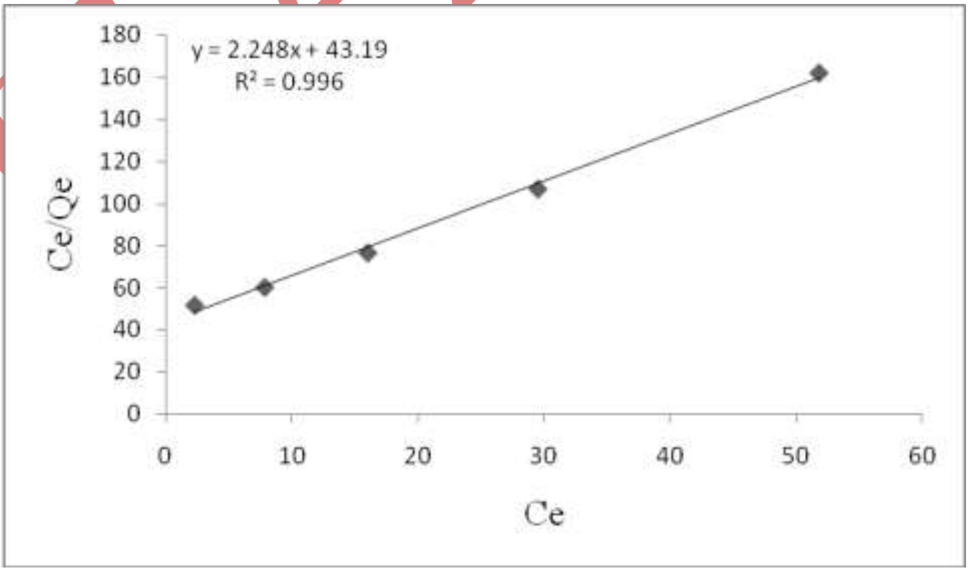

Figure 3.12 Langmuir adsorption isotherm of phenol by FA-2 (pH 6, adsorbent dosage 40 g/l and contact time 2 hours)

Table 3.3 Parameters of Langmuir Isotherm for phenol-FA-1 and FA-2 System

Fly Ash	**Qo (mg/g)**	**b(l/mg)**	**R^2**	**R_L**
FA-1	0.352	0.047	0.987	0.175
FA-2	0.444	0.052	0.996	0.161

From the data of Langmuir isotherm a dimensionless equilibrium parameter called separation factor, R_L, which describes the favourable nature or feasibility of adsorption process could be evaluated. R_L is calculated according to the Eq. 3.2:

$$R_L = 1/1+bCo \qquad \text{Eq. 3.2}$$

Where Co = initial adsorbate concentration (mg/l)
b = Langmuir constant (l/mg)

According to Hall et al. [38], value of R_L indicates the type of isotherm as follows:

Table 3.4 Type of isotherm for R_L parameter

R_L	Type of isotherm
RL > 1	Unfavourable
RL = 1	Linear
0 < RL < 1	Favourable
RL = 0	Irreversible

The values of R_L are less than 1 and greater than 0 indicating favourable adsorption in case of both FA-1(0.175) and FA-2 (0.161). The regression values as shown in Table 3.3 depicts that the linear form of the Langmuir isotherm appears to produce a reasonable model for the sorption of phenol on FA-1 and FA-2.

3.3.2 Freundlich isotherm model

Freundlich isotherm is used to describe adsorption in aqueous systems and supposes that the ratio between the amount of adsorbed phenol and the concentration of phenol in the liquid phase is not a constant at different concentrations. Freundlich isotherms were obtained by agitating the phenol solution of different initial concentrations with fixed dosage of the adsorbent for a contact time greater than the equilibrium time.

The Freundlich equation can be described by the following linearised form [39]:

$$\text{Log } Qe = \log K + (1/n) \log Ce \qquad \text{Eq. 5.5}$$

where,

Qe = amount adsorbed (mg/g)

Ce = equilibrium concentration (mg/l)

K = adsorption capacity

n = adsorption intensity

A plot of log Qe against log Ce gives straight line, the slope and intercept of which corresponds to 1/n and log K.

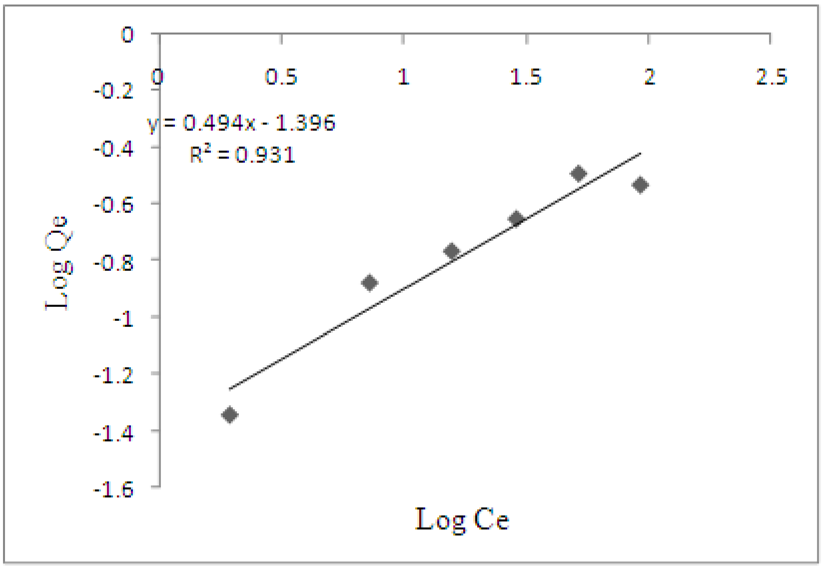

Figure 3.13 Freundlich adsorption isotherm of phenol by FA-1(at pH 6, adsorbent dosage 40 g/l and contact time 2 hour)

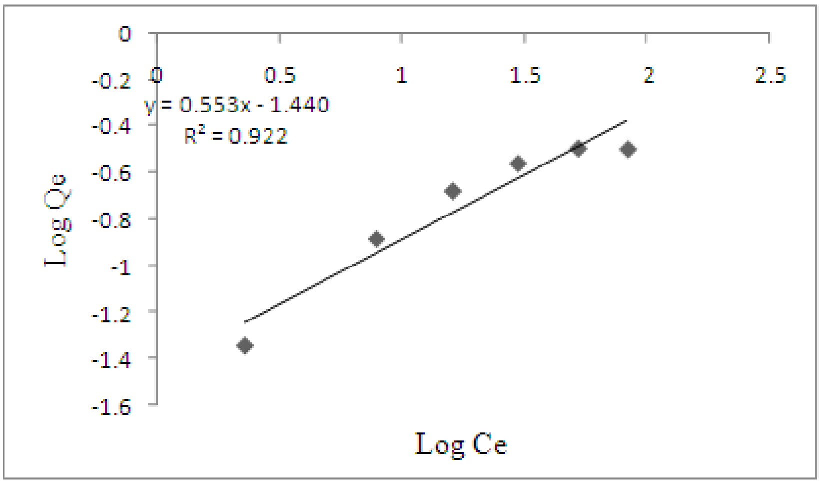

Figure 3.14 Freundlich adsorption isotherm of phenol by FA-2 (at pH 6, adsorbent dosage 40 g/l and contact time 2 hours)

Table 3.5 Parameters of Freundlich Isotherm for phenol-FA-1 and FA-2 System

Fly Ash	K (mg/g)	n	R^2
FA-1	0.040	2.024	0.931
FA-2	0.036	1.808	0.922

Freundlich isotherm constants determined from the plot of log Qe versus log Ce are summarized in Table 3.5. The values of the Freundlich constants (K and n), indicating capacity and intensity of adsorption, respectively, are greater than unity for adsorption of phenol by both the types of fly ashes fulfilling the Freundlich isotherm condition of favourable adsorption. Freundlich isotherm model seems to fit the experimental adsorption data better than Langmuir adsorption isotherm model.

IV. CONCLUSIONS

The following conclusions can be drawn from this research work:

1. Equilibrium time required for the adsorption of phenol on both the types of fly ash was almost 120 minutes (2 h), after which the process almost approached equilibrium with smooth and continuous curves leading to saturation.
2. The maximum removal occurs at pH 6 and thereafter a significant decline in removal efficiency was observed with further increase in pH.
3. It was found that the phenol removal increased by increasing adsorbent dosage and fly ash dosage of 40.0 g/l was found to be the optimum fly ash dosage.
4. Since percentage removal starts to decrease after 20 mg/l indicating less favourable sites in adsorption, therefore 20 mg/l was found to be the optimum initial phenol concentration for sorption experiments.
5. FA-1 exhibited higher adsorption capacity in comparison to FA-2 as increase in percentage of finer particles in FA-1 resulted in the increase in total surface area which provided more active sites for the adsorption of phenol at smaller particle sizes.
6. The Freundlich isotherm model fitted the experiment data better than Langmuir isotherm model, indicating higher adsorption capacity for FA-1 as compared to FA-2.

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