Adsorption of copper and lead in single and binary metal system onto *Fumaria indica* biomass

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A R T I C L E   I N F O

A B S T R A C T

Among heavy metals, lead and copper pose a significant threat to the due to their toxicity, incremental accumulation in the food chain and persistence in the ecosystem. Lead and copper are introduced into water from various industries and other human activities. Present study was aimed to remove lead and copper from aqueous media using *Fumaria indica* biomass as a function of metal ions initial concentration. Both metal removal was investigated in single and binary system. For comparison isotherm models i.e., Langmuir and Freundlich were applied on experimental adsorption data. Results showed that the copper behavior was different in single and binary metal system, whereas lead adsorption remained the same in both single and binary metal systems. The Freundlich isotherm model fitted well to the adsorption data of both single and binary metal system. The thermodynamic parameters i.e., ΔG^0, was measured to get insight into adsorption phenomenon and negative value of ΔG^0 suggest that the adsorption process was spontaneous in nature. Results showed that in multi-metal system the adsorption of specific metal ion may change and during metal adsorption from industrial wastewater this effect must be taken in account before generalization of results.

Capsule Summary: The lead and copper removal from aqueous media using *Fumaria indica* biomass in single and binary metal system was evaluated as a function of metal ions initial concentration.

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INTRODUCTION

An accelerated release of heavy metals into the aquatic environment causes serious water pollution problems because of their toxicity, persistence and bioaccumulation in food chain (Iqbal et al., 2013; Jamal et al., 2015; Qureshi et al., 2015; Sayed, 2015). Several industrial operations have lead to water pollution by introducing metals as toxic contaminants in water. Now a days, due to over population, water resources are depleting, therefore, there is a need to re-evaluate the pattern water utilization to save water reservoirs (Bilal et al., 2014; Iqbal and Bhatti, 2014, 2015; Iqbal et al., 2014; Iqbal and Nisar, 2015).

Major problem is contamination of the environment with harmful heavy metals from industrial wastewater (Manzoor et al., 2013). Metal ions are serious environmental pollutant normally encountered in industrial waste water (Ullah et al., 2013). Lead is introduced into natural waters due to a variety of industrial wastewaters such as paper and pulp, mining, electroplating, lead smelting and metallurgical finishing, dyeing, storage-battery and automotive industries. Similarly, copper and its compounds are widely used in many industries (electrical, electroplating, paper manufacturing, pesticides, herbicides and tannery industries) and wastewater discharged from these industries is a major source of this metal. The continuous intake of copper by human beings leads to necrotic changes in the liver and kidney, mucosal
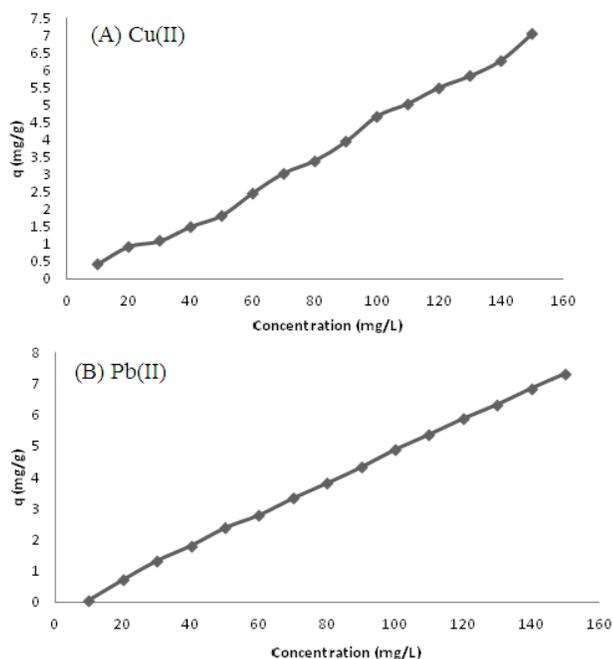
Fig. 1: (A) Effect of copper initial metal concentration (B) Effect of lead initial metal on adsorption onto *Fumaria indica* in single metal system

irritation, wide spread capillary damage, depression, weakness, lethargy, anorexia, gastrointestinal irritation and lung cancer (Manzoor et al., 2013).

Adsorption using biomass has been considered as a potential technique for the removal of toxic heavy metals and high cost of commercial adsorbent makes the adsorption process as an expensive one. Therefore, cost effective, efficient alternate adsorbents for the adsorption of heavy metals from aqueous media are required (Rangabhashiyam and Selvaraju, 2015). Biosorbents derived from agricultural wastes have been proved very effective for the removal of metals from aqueous solution. The agricultural based waste adsorbents are less costly, more effective, available round the year and easy to process and are recyclable (Iqbal et al., 2013; Manzoor et al., 2013; Rangabhashiyam and Selvaraju, 2015; Ullah et al., 2013). Therefore, present study was undertaken for the adsorption of copper and lead from aqueous media using *Fumaria indica* plant biomass. The principle objective of research was to compare the adsorption of both metal ions in single and binary metal system. The equilibrium modeling and thermodynamic studies was performed both for single and binary metal system to get insight into adsorption mechanism and phenomena involved.

MATERIAL AND METHODS

Reagents and instruments

All chemicals and reagents such as $CuSO_4 \cdot 5H_2O$, $Pb(CH_3COO)_2$, HCl, NaOH, acetone, and Pb(II) and Cu(II) standards used were of analytical reagent grade and purchased from Fluka and Merck. Stock solution of Pb(II) was prepared by dissolving salts

of lead and copper in double distilled water and the working solutions were obtained by diluting stock solution. Aqueous copper and lead solutions were prepared by diluting $CuSO_4 \cdot 5H_2O$ and $Pb(CH_3COO)_2$ in distilled water. All glassware used for experimental purposes was washed in 60% (v/v) nitric acid and subsequently rinsed with de-ionized water to remove any possible interference by other metals.

Plant material

The *Fumaria indica* plants were collected from different botanical garden, UAF, Pakistan and authenticated at Ayub Agricultural Research Institute Faisalabad. Plant material was washed with tap water to remove any dust or foreign particles attached to biomass and thoroughly rinse with distilled water. The biomass was dried at room temperature for fifteen days. Dried biomass chopped, grinded (Moulinex, France) and finally, sieved though Octagon siever (OCT-DIGITAL 4527-01) for the removal of uneven particle and to obtain adsorbent with a uniform and known particle size. The fraction with 0.25 mm diameter was selected, stored in airtight plastic containers and used for sorption experiments.

Biosorption procedure

The adsorption experiments were carried out in 250 mL Erlenmeyer flasks containing 100 mL of copper and lead solutions. The flasks with adsorbent dosage of 0.1 g were incubated at room temperature at 120 rpm for 24 h. After adsorption, samples were filtered using Whatmann 32 filter paper and the metal concentrations were determined. Each experiment was carried out in triplicates. For single metal solution, solutions of fixed volume (100 mL) with varying concentrations were added in conical flasks, mixed thoroughly with 0.1 g biosorbent dose and set in shaker (PA 250/25. H) at room temperature, 120 rpm for 24 h. For binary metal solution, 100 mL volume of solutions with varying concentration (25, 50, 75,100,125 and 150 mg L^{-1}) of both metal was used and other experimental setup was same. After stipulated time, samples were filtered, the lead and copper concentrations were determined using AAS (A Analyst 30, Perkin Elmer). The amount of metals adsorbed was calculated by the simple concentration difference method. The biosorption capacities were estimated using the amount of ions retained on unit mass of biomass using the relation (Eq. 1).

$$q \text{ (mg/g)} = [(C_0 - C) \cdot (V/1000)]/m \qquad (1)$$

Where C_0 is the initial concentration of metal ions (mg/L), C is the equilibrium concentration (mg/L), V is the volume of solution (mL) and m is the biosorbent mass (g).

Statistical analysis

Data reported represents the mean of three independent experiments. The correlation coefficient (R^2) values of the linear form of Langmuir isotherm, Freundlich isotherm were also determined using statistical functions of Microsoft Excel, 2007 (version Office XP, Microsoft Corporation, USA).
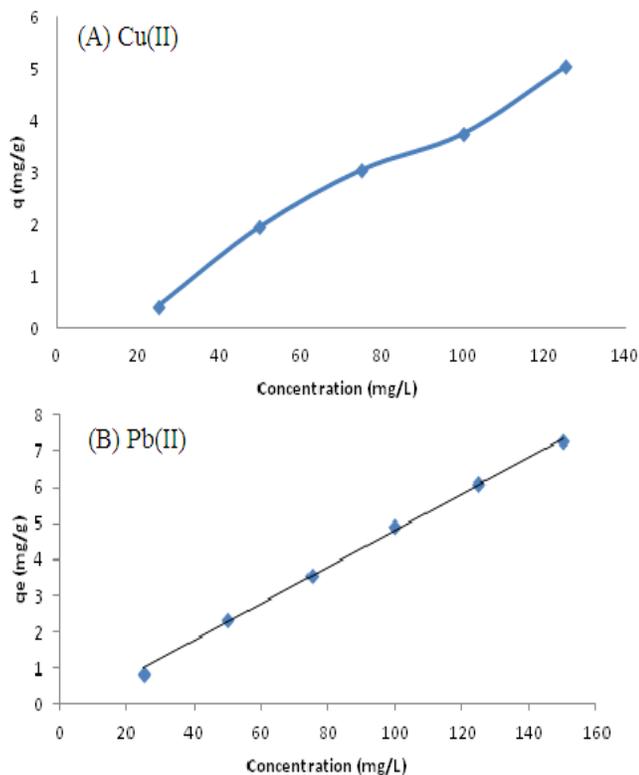
Fig. 2: (A) Effect of copper initial metal concentration (B) Effect of lead initial metal on adsorption onto *Fumaria indica* in binary metal system

RESULTS AND DISCUSSION

In single metal system, the effect of initial metal ions concentration was studied in the range of 10 to 150 mg/L using adsorbent dose 0.1 g, agitation speed 120 rpm, contact time 24 h at room temperature. The adsorption capacity increased as the metal ions concentration increased. However, the percentage recovery decreased by increasing the lead and copper initial concentrations. The results of metal adsorption onto *Fumaria indica* are shown in Fig. 1ab for copper and lead, respectively. In concentration study range, the maximum adsorption of copper was found to be 7 mg/g by *Fumaria indica* biomass, whereas for lead the adsorption capacity was 7.5 mg/g. Results showed that adsorption of both metal ions depends upon the initial concentration because at lower metals ion concentration, the adsorption capacity was also low and it was increased as the initial concentration increased. It is well known that the initial concentration acts as an important driving force to overcome mass transfer resistance of ions between the aqueous and solid phases (Iqbal et al., 2013; Manzoor et al., 2013; Ullah et al., 2013). The initial concentration of copper and lead to the available sorption sites is low at lower concentration; therefore sorption may become independent of initial concentration. At

higher concentration, the available sites might occupied and subsequently, the removal depends on the initial concentration (Aksu and Isoglu, 2006; Manzoor et al., 2013; Ullah et al., 2013). It was observed that by increasing the concentration there was a sharp increase in the adsorption initially, followed by slower adsorption later on. This is due to availability of the adsorption sites which initially lead to the increased adsorption, which on saturation of the adsorption sites decreased gradually (Pandey and Mishra, 2011). This is due to increase in number of metal ions competing for available binding sites and due to lack of binding sites for complexation at higher metal ion concentration. At lower concentration almost all the metal ions can interact with binding sites facilitating maximum adsorption (Gupta and Balomajumder, 2015). The decrease in metal removal with an increase in initial concentration was attributed to limited adsorption sites and intra-particle diffusion (Moussavi and Barikbin, 2010). At lower concentration, the ratio of available surface to the initial copper and lead concentrations were higher and at higher concentration, the ratio was low; hence the metal ions removal decreased at higher concentration at fixed adsorbent dose and volume of solution. According to (Yu et al., 2003) at low concentration of sorbate, the ratio of the initial number of moles of metal ions to the available surface area was larger and subsequently, the fractional biosorption becomes independent of initial concentrations. However, at higher concentrations the available sites of biosorption become fewer, and hence the percentage removal of metal ions depends upon the initial concentration. Similar trend of adsorption has also been reported previously as a function of initial metal ion concentration onto dead fungal biomass of *Phanerochaete crysosporium* (Marandi, 2011), immobilized *C. elegans* (Abdel-Razek, 2011) and dried biomass of *Cyanobacterium Oscillatoria* laete-virens (Das, 2012), wheat shell (Saha et al., 2012). pistachio hull (Moussavi and Barikbin, 2010), *R. damascena* phytomass (Iqbal et al., 2013), *Chlorella* species biomass (Kanchana et al., 2011), boiled mucilaginous seeds of *O. americanum* (Lakshmanraj et al., 2009), *Aspergillus niger* NUA101 (Ghosh et al., 2015), banana peel (Memon et al., 2009), orange (Citrus cinensis) waste (Marín et al., 2009), walnut hull (Wang et al., 2009) and dead fungal biomass of *Phanerochaete crysosporium* (Marandi, 2011). In binary metal ion system, the adsorption of both lead and copper ions was also studied. The adsorption capacity of *Fumaria indica* biomass was found different in binary metal system. The results of copper and lead adsorption in binary metal system are shown in Fig. 2. From results it is clear that the copper adsorption onto *Fumaria indica* biomass decreased, whereas the lead adsorption onto *Fumaria indica* biomass remained same. The adsorption conditions in single and binary metal system were same and in binary metal system the copper adsorption was decreased and lead adsorption was not affected, which indicates that there is no effect of co-metal ions on lead adsorption, whereas copper adsorption may decrease in the presence of co-metal ions.

Adsorption isotherm

To gain more insight into the adsorption of copper and lead onto *Fumaria indica* biomass, the results of equilibrium experiments of both single metal and binary metal system were subjected to

159

Table 1: Langmuir and Freundlich parameters for single metal system

Metal	Langmuir parameters			Exp	Freundlich parameters		
	q_{max}	b	R^2		q_{max}	1/n	R^2
	(mg/g)				(mg/g)		
Pb	9.15	0.1332	0.6856	7.32	39.15	1.6327	0.9078
Cu	6.618	0.0775	0.6582	7.055	13.007	0.6618	0.9155

Table 2: Langmuir and Freundlich parameters for binary metal system

Metal	Langmuir parameters			Exp	Freundlich parameters		
	q_{max}	b	R^2		q_{max}	1/n	R^2
	(mg/g)				(mg/g)		
Pb	51.54	0.0366	0.4647	7.26	9.91	0.8695	0.8309
Cu	0.074	0.323	0.888	5.052	4.081	0.3684	0.979

Langmuir and Freundlich isotherm models. The optimization of an adsorption process requires an understanding of the driving forces that govern the interaction between the adsorbate and the adsorbent. In order to compare the design of an adsorption system for the removal of copper and lead ions, it was important to establish the most appropriate correlation for the equilibrium. The Langmuir and Freundlich models are the most frequently used simple isotherm describing the equilibrium (Shroff and Vaidya, 2011). The isotherm models, Langmuir and Freundlich were applied to understand the copper and lead adsorption behaviour onto *Fumaria indica* biomass. Langmuir adsorption model assumes that adsorption occurs at specific homogeneous adsorption sites within the adsorbent and intermolecular forces decrease rapidly with the distance from the adsorption surface (Manzoor et al., 2013; Ullah et al., 2013). The model further based on the assumption that all the adsorption sites are energetically identical and adsorption occurs on a structurally homogeneous adsorbent. The Langmuir adsorption isotherm is represented in Eq. 2 and linear form of this isotherm is expressed in Eq. 3. Where q_{max} (mg/g) and b (L/mg) are Langmuir constants related to the maximum adsorption capacity and the heat of adsorption, respectively (Manzoor et al., 2013; Ullah et al., 2013).

$$q_e = \frac{q_{max} \times b \times C_e}{1 + b \times C_e} \qquad \text{(Eq. 2)}$$

$$\frac{C_e}{q_e} = \frac{C_e}{q_{max}} + \frac{1}{q_{max} \times b} \qquad \text{(Eq. 3)}$$

Freundlich isotherm model can be used to describe the sorption on heterogeneous surfaces as well as a multilayer sorption. It assumes that the uptake of adsorbate ions occurs on a heterogeneous adsorbent surface (Ewecharoen et al., 2008). The Freundlich model is empirical in nature which further assumes that the stronger binding sites are occupied first and that the

binding strength decreases with increasing degree of site occupation (Srividya and Mohanty, 2009). The logarithmic form of Freundlich model is given in Eq. 4 (Freundlich, 1906).

$$\log q_e = log K_F + \frac{1}{n} \log C_e \qquad \text{(Eq. 4)}$$

Where K_F and *n* are the Freundlich rate constants designated as adsorption capacity and adsorption intensity, respectively, which can be obtained from slope and intercept of the plot of $\log q_e$ against $\log C_e$. The value of *n* in the range 1–10 is another benchmark to assess the good adsorbent–adsorbate interaction (Kumar et al., 2012). The magnitude of the exponent, 1/*n* gives an indication of the favorability of adsorption. The values of *n* > 1 represents favorable adsorption condition (Pandey and Mishra, 2011) and this empirical constant indicating adsorption intensity (L mg^{-1}) and depends on the temperature and properties of the adsorbate and the adsorbent. C_e is the residual concentration of solute remaining in the solution (mg L^{-1}), q_e is the amount of adsorbate adsorbed by a unit mass of adsorbent at equilibrium (mg g^{-1}). Data fitted with Langmuir and Freundlich isotherm models is given in Table 1 and Fig. 3 for single metal system. The higher R^2 and q_{max} values suggested that the Freundlich isotherm describes the sorption process well as compared to Langmuir model isotherm model for single system. The q_{max} and R^2 values obtained in case of Freundlich isotherm were 39.15 and 0.9078 for lead and these values were 13.007 and 0.9155 for copper and in case of Langmuir isotherm model both q_{max} and R^2 values were considerably lower versus Freundlich isotherm. The Langmuir isotherm model indicates the formation of monolayer coverage of heavy metal ions on the outer surface of biosorbent. The maximum biosorption capacity (q_{max}, mg/L), which is a measure of the adsorption capacity to form a monolayer was found lower in case of Langmuir isotherm. The Freundlich isotherm model was employed to describe the adsorption on
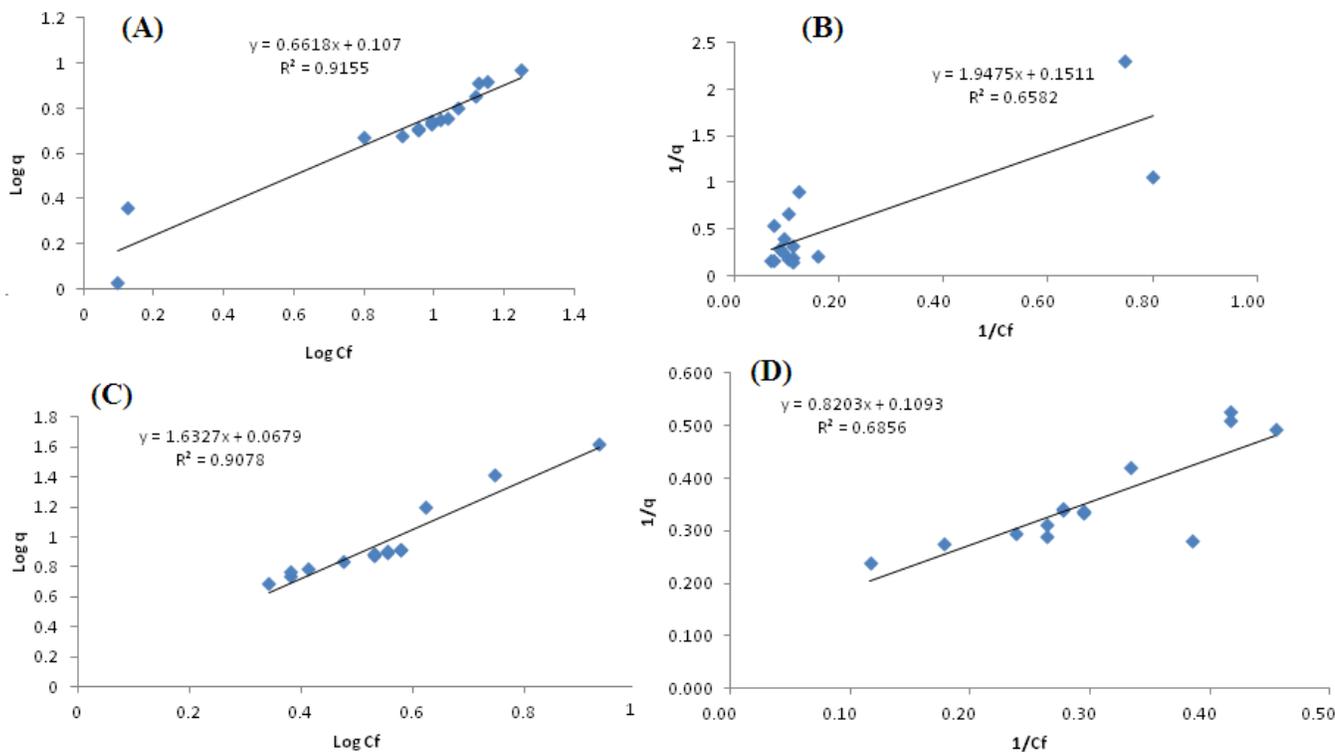
Fig. 3: Frendlich isotherm for copper in single metal system, (B) Langumir isotherm for for copper in single metal system, (C) Frendlich isotherm for lead for single metal system, (D) Langumir isotherm for lead for single metal system

heterogeneous surface which is not restricted to the formation of monolayer and the fractional values of $1/n$, from Freundlich isotherm model (Table 1), suggest the heterogeneity of biosorbent surface and simultaneously indicate a favorable biosorption of metal ions. The values of correlation coefficients (R^2) obtained in case of Freundlich isotherm model was higher than those obtained in case of Langmuir model, which indicate that the Freundlich model fitted well to the lead and copper adsorption data. These findings are in line with previous studies for the adsorption of metal ions onto different biomasses i.e., rose waste biomass (Iqbal et al., 2013), immobilized *C. elegans* (Abdel-Razek, 2011), *P. ostreatus* spent biomass (Carol et al., 2012), *Chlorella* species biomass (Kanchana et al., 2011), boiled mucilaginous seeds of *O. americanum* (Lakshmanraj et al., 2009), dead fungal biomass of *P. crysosporium* (Marandi, 2011) and banana peel (Memon et al., 2009). The binary metal system was also subjected to both isotherm model i.e., Langmuir and Freundlich isotherm model. All condition of adsorption was similar to single metal system except metals ions. In second set of adsorption binary metal system was checked. The results obtained of isotherm models for binary system are shown in Table 2 and Fig. 4 and 5 both for copper and lead, respectively. The q_{max} and R^2 values were 9.91 and 0.8309 for lead and 4.081 and 0.979 for copper in case of Freundlich isotherm. Similar to single metal system, in binary metal system the Langmuir model was not fitted to the experimental data. The higher q_{max} and R^2

values indicate that Freundlich isotherm model fitted well to the binary metal system.

Thermodynamic Study

In an effort to understand the nature of the process for copper and lead adsorption, the thermodynamic parameter Gibbs free energy ($\Delta G°$) was calculated using Eq. 5. Where, K_c is the equilibrium constant

$$\Delta G^0 = -RTlnK_c \hspace{2cm} \text{(Eq. 15)}$$

The $\Delta G°$ was found to be negative, which indicates that the adsorption process was spontaneous in nature. The ΔG^0 were between 0-20 kJ/mole i.e, -5.95 to -11.20 kJ/mole. In general, the values of energy ΔG^0 in between 0 and -20 kJ/mol reveals that the adsorption process was physisorption in nature, while the values between -80 and -400 kJ/mol correspond to chemisorptions (Wu et al., 2013).

CONCLUSION

In present study, lead and copper from aqueous media using *Fumaria indica* biomass as a function of metal ions initial concentration was investigated in single and binary metal system. Results showed that the copper adsorption decreased in binary metal system versus single metal system and lead adsorption did not change in binary metal systems. The Freundlich isotherm models fitted well to the adsorption data in both single and
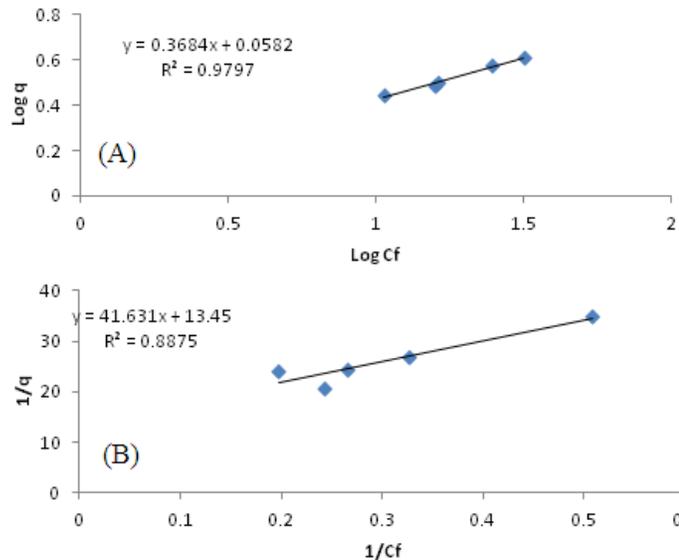
Fig. 4: (A) Frendlich isotherm for copper in binary metal system and (B) Lagumir adsorption isotherm for copper in binary metal system
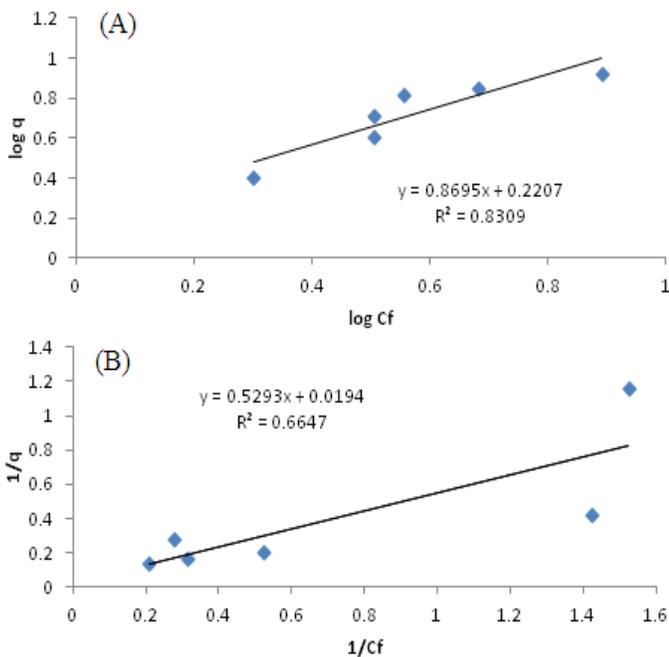

Fig. 5: (A) Frendlich isotherm for lead in binary metal system and (B) Langumir isotherm for lead in binary metal system

binary metal system. The negative value of ΔG^0 suggests that the adsorption process was spontaneous in nature. Results showed that in binary metal system the adsorption of specific metal ion may change and in future studies the effect of other process variable i.e., pH, adsorbent dose, contact time and temperature should be studied both for single and binary metal system.

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