



Isotherm Modelling of Pb Sorption by Lime and SSP from Contaminated Waste Waters

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Abstract: Lead (Pb) pollution from various industrial activities results in many health issues in plants, animals and human beings. Removal of Pb using inorganic chemicals like lime and phosphates makes it unavailable and prevents its entry into the food chain. This study deals with the removal of lead by chemical sorbents viz., lime and single super phosphate (SSP) from aqueous solutions through a batch incubation experiment. The effect of sorbent dosage, initial Pb²⁺ concentrations, and incubation time intervals on Pb adsorption was described using isotherm models. It was inferred that, SSP had higher Pb adsorption capacity (6.08 mg g⁻¹) than lime (4.96 mg g⁻¹) which increased with increasing time intervals from 0 to 6.6 mg g⁻¹ for SSP and 5.59 mg g⁻¹ for lime. The adsorption capacity of SSP increased from 1.14 to 12.7 mg g⁻¹ in SSP and 0.99 to 10.1 mg g⁻¹ in lime when the initial Pb concentration increased from 100 to 2000 mg kg⁻¹. Langmuir adsorption isotherm explained the sorption process better than Freundlich model for both the sorbents. Hence, it could be concluded that, SSP is the effective sorbent which can be utilized for the removal of Pb from wastewater and adsorption mechanism was well described by Langmuir adsorption isotherm.

Keywords: Lead, Lime, SSP, Adsorption isotherm, Contaminated water

Pollution refers to the presence of undesirable materials, in the ecosystems beyond the permitted levels that can adversely disturb life. Increased industrialization activities involving the use of chemical substances like hydrocarbons, pesticides, chlorinated hydrocarbons and heavy metals serve as the prime anthropogenic source which is the key contributor for pollution (Rodríguez et al 2020). Lead is one of the ubiquitous and hazardous environmental pollutants. Lead poisoning in humans causes severe damage to the kidneys, nervous system, reproductive system, liver, and brain (Ara and Usmani 2015). Despite its toxicity, lead is widely used for different applications, in particular as electrodes in the lead-acid batteries, photovoltaic cells, glasses, paint, etc. (Kumar et al 2022). As a result, the levels of Pb in the environment continue to grow, making the development methodologies inevitable for the remediation of Pb-contaminated waters and soil (Alsafran et al 2023).

Remediation of metal-contaminated media is a time-consuming and expensive process. Existing methods for heavy metal removal from waste water include chemical precipitation, ion exchange, membrane filtration, and adsorption (Wang et al 2014, Tavakoli et al 2017 and Rajendran et al 2022). Adsorption is relatively suitable technique to reduce the Pb load in wastewater, since

adsorbing materials have higher metal binding capacity. The major advantages of adsorption over other methods are: better efficiency, low cost, and minimization of chemical and biological sludge (Uddin, 2017 and Chakraborty et al 2022). Application of adsorption isotherm models and their adsorption parameters aids in delineating the mechanism of adsorption pathways, adsorbent capacities and the extent of dependence on adsorbent's surface properties for Pb sorption, and ultimately in designing competent adsorption systems.

The geochemical behaviour of Pb indicates that phosphate and carbonate, when present in sufficient amounts, forms lead phosphates and carbonates which are highly insoluble thus reducing Pb leachability and bioavailability. Using soluble phosphates and carbonates to adsorb and immobilize Pb is a cost-effective, efficient and viable remedial approach. Several insoluble Pb orthophosphate and carbonate minerals have formed after reaction with phosphate (Mouflih et al 2005). Many studies have been conducted using natural apatite and rock phosphate as a phosphate source (Mouflih et al 2005 and Minh et al 2012). Single super phosphate is a commercial P fertilizer containing 16% water soluble P₂O₅ which is widely used in agricultural sector. Zhao et al (2018) suggested that, calcium phosphate addition promote the precipitation of lead

phosphate particles which are less soluble than lead carbonate and provide equilibrium dissolved lead concentrations lesser than the toxic limits. Among the carbonates, calcium carbonate (CaCO_3) is one of the most abundant low-cost materials on earth which is biocompatible and biodegradable, meeting all the desirable criteria for an adsorbent to be used on large-scale. Previous studies on the use of various polymorphs of CaCO_3 like calcite, aragonite, and amorphous CaCO_3 for adsorbing heavy metals, unfortunately demonstrated that, natural CaCO_3 has lesser efficiency in removing heavy metal ions (Zhang et al 2018).

However, limited study has been conducted to compare the efficiencies of lime (pure calcium carbonate) and soluble calcium phosphate (SSP). Thus, this study aims to investigate and compare the parameters that affect amount of Pb adsorbed at varying contact time, initial Pb concentrations, and adsorption capacity of lime and SSP. Furthermore, to ascertain the best fitting isotherm model for describing Pb adsorption by SSP and lime and to know the mechanisms involved in Pb removal to choose the best model to justify the Pb removal from waste water.

MATERIAL AND METHODS

Lead (Pb) standards and sorbents: The Pb standards were prepared using analytical grade lead nitrate ($\text{Pb}(\text{NO}_3)_2$) salt and about 2000 mg L^{-1} stock solution was prepared. Using the stock, different concentrations viz., 100, 250, 500, 1000 and 1500 mg L^{-1} of Pb^{2+} was prepared. The fertilizer grade single super phosphate (SSP) was procured from Tamil Nadu Co-operative Marketing Federation (TANFED), Ramnathapuram, Coimbatore and commercial grade lime was used as sorbents in the study.

Batch sorption experiment: Batch sorption experiment was conducted at room temperature (25°C) with the chemical sorbents for assessing their adsorption potential at different dosage, contact time and Pb concentration. The sorption studies were conducted in 50 ml centrifuge tubes with different doses of sorbents (1, 2.5 or 5 g) added to 25 ml of known Pb concentration solution. The centrifuge tubes with sorbents and Pb solution at different concentration was agitated on a mechanical shaker at 160 rpm for an appropriate contact time of 24, 48, 72, 96 and 120 hours, and centrifuged for 3 min at 5000 rpm. The supernatant was filtered using Whatman No.42 filter paper and analysed for residual Pb concentration using atomic absorption spectrophotometer (Model: GBC AvantaPM) with air-acetylene flame. Sufficient care was taken to prevent the loss of sorbents while separating the supernatant. The experiment was conducted in duplicates with a blank simultaneously to quantify the Pb adsorbed by the sorbents

from solution. The quantity of Pb^{2+} adsorbed (q_e , mg g^{-1}) was estimated (Ray et al 2020) using the following equation (1)

$$q_t = V * \frac{C_0 - C_f}{m} \text{----- (1)}$$

where ' C_0 ' and ' C_f ' are the concentrations of Pb^{2+} (mg L^{-1}) in initial and final solutions, respectively, 'V' is the volume of Pb^{2+} solutions added (25 ml) and 'm' is mass of the sorbent (g). **Adsorption isotherms:** The data obtained from batch experiments were fitted to isotherms models like Langmuir and Freundlich to identify the best fitted model so as to explain the retention and release characteristics of Pb in waste water by SSP and lime.

Langmuir isotherm: The adsorption isotherm model outlined by Langmuir is furnished in the equation given below:

$$q_e = \frac{q_m K_L C_e}{1 + K_L C_e} \text{----- (2)}$$

Where, ' q_m ' is the maximum monolayer coverage capacity (mg g^{-1}), ' q_e ' is the amount of Pb adsorbed per gram of adsorbent at equilibrium (mg g^{-1}), ' C_e ' is the concentration of Pb at equilibrium (mg L^{-1}) and ' K_L ' is the constant representing the affinity of active binding sites (L mg^{-1}). The values of ' q_m ' and ' K_L ' were obtained from slope and intercept of plot between ' C_e/q_e ' vs. ' C_e ' (Langmuir 1918).

Freundlich isotherm: The adsorption isotherm proposed by Freundlich is commonly used for describing adsorption on heterogeneous surface (Dada et al 2012) which is represented in the equation (3) below:

$$q_e = K_F C_e^{1/n} \text{----- (3)}$$

Where ' K_F ' is the constant related to adsorption capacity (mg g^{-1}) and ' n ' is the intensity of adsorbent. The values of ' n ' and ' K_F ' were obtained from slope and intercept of Freundlich plot of $\log q_e$ vs. $\log C_e$. The favourability of adsorption is indicated by the magnitude of exponent ' n '.

Statistical analysis: The linear forms of six kinetic equations were fitted to the kinetic experimental data. The R^2 values and isotherm parameters were subsequently obtained from the fitted equations. The constants and parameters of all the isotherms were calculated using OriginPro 2022. The data is subjected to statistical analysis using SPSS software and simple variance analysis was done using factorial completely randomized block design with three factorial arrangement (Pb^{2+} concentration, sorbent dose and incubation time interval) and two replications. The least significance test was used to detect the variations between means at $p < 0.005$.

RESULTS AND DISCUSSION

Lead (Pb^{2+}) adsorption as influenced by various factors:

Sorbent dose: The amount of Pb^{2+} adsorbed increased with

increasing sorbent dosage (1.0 to 5.0 g/25 ml) irrespective of the sorbent studied (Fig. 1a). The amount of Pb^{2+} adsorbed decreased from 10.6 to 2.8 $mg\ g^{-1}$ in SSP and 8.07 to 2.47 $mg\ g^{-1}$ in lime when the sorbent dose increased from 1 to 5 g. The adsorption capacity decreased with the increase in adsorbent dosage for both adsorbent. As the sorbent dosage increased, the ratio of number of adsorption sites to the number of heavy metal ions would increase and there would be plenty of unabsorbed adsorption sites (Yarkandi 2014). It might also be the result from the aggregation of sorbents particles at higher dosages, thereby resulting in a decline in the surface area of adsorbent and also an increase in the diffusion path length (Rezaei et al 2022 and Ayodele et al 2016).

Initial Pb concentration: The amount of Pb^{2+} adsorbed increased with increasing initial Pb concentrations and the amount of Pb^{2+} adsorbed was significantly higher in SSP than lime for varying Pb concentrations (Fig. 1b). The amount of adsorbed Pb^{2+} varied from 1.14 to 12.7 $mg\ g^{-1}$ in SSP and 0.99 to 10.1 $mg\ g^{-1}$ in lime. The increase in Pb concentration resulted in increased number of available Pb ions per binding site in the adsorbent thus bringing a higher probability of Pb ion binding to the adsorbent. The probability of chemical interaction between the adsorbent and the adsorbate was enhanced by higher availability of Pb molecules in solution (Ahmad et al 2012). Increasing initial Pb concentration, increases the interaction between the metal ions and the sorbents, thus enhancing the availability of binding sites on the surface of sorbents (Mandal et al 2016 and Bulut et al 2018). These results corroborate with the findings reported by Yarkandi (2014) and Rezaei et al (2022).

Time: The Pb^{2+} adsorption capacity was studied at different incubation time intervals for both the sorbents (Fig. 1c) and observed an increase in adsorption capacity with the advancement of time for both sorbents. The adsorption capacity was found to increase from 0 to 5.33 $mg\ g^{-1}$ for SSP and 4.1 $mg\ g^{-1}$ for lime within 24 hours of incubation. The initial faster adsorption was due to a rapid diffusion of ions from solution to external adsorbent surface, and the slower adsorption capacity in the second stage was possibly the result of diffusion of Pb ions into the adsorbent material (Harja et al 2017). Upon saturation of adsorption sites, equilibrium is attained and adsorption capacity tends to increase at lesser rates (Shah et al 2018). Similar observations were noted in this study where there is a slow and steady increase in the adsorption rates (5.33 to 6.6 and 4.1 to 5.59 $mg\ g^{-1}$ for SSP and lime respectively) from 24 to 120 hrs. These results are in line with the findings of Harja et al (2017) and Shah et al (2018).

Adsorption isotherms: The data obtained from the

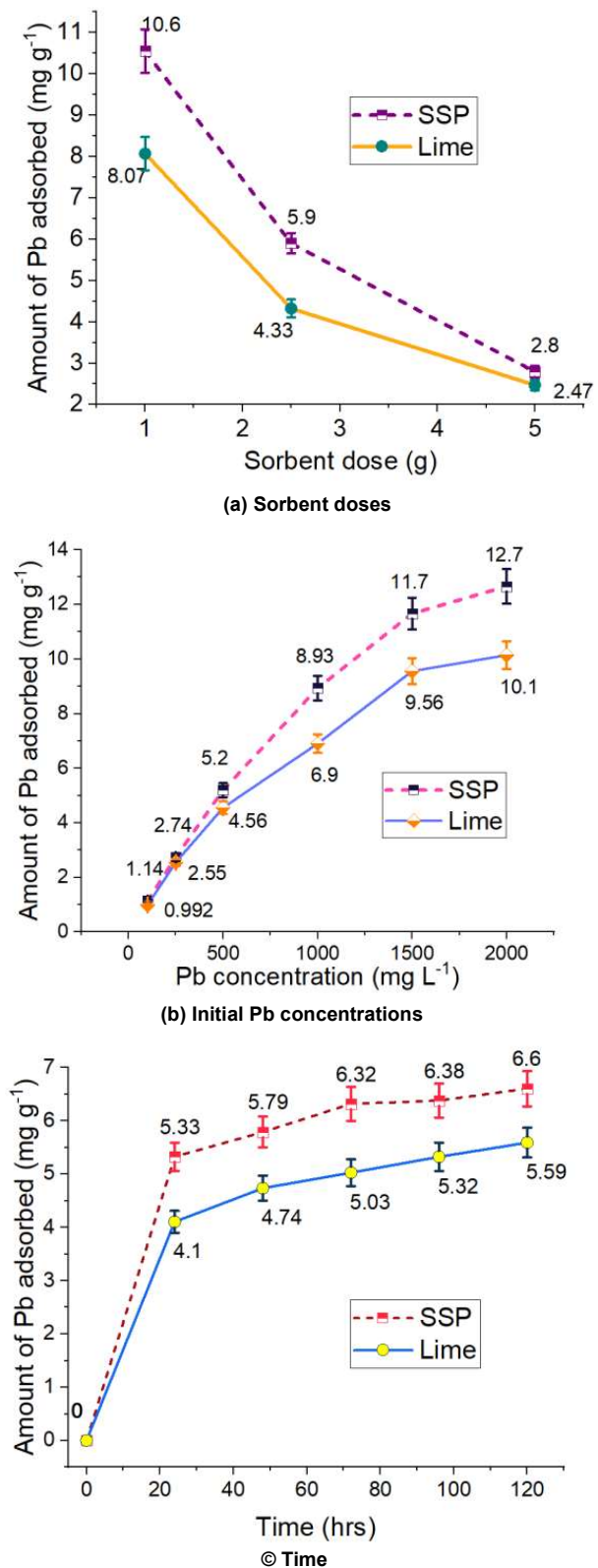


Fig. 1. Adsorption rate (%) by SSP and Lime as influenced by (a) Sorbent doses (b) Initial Pb concentrations (c) Time. Bars represent standard error; data are average of two replications; comparison of mean was done at 5% level

experiment was fitted to Langmuir and Freundlich isotherm models to identify the best model to explain the Pb adsorption characteristics of the sorbents (Table 1).

The coefficient of determination varied from 0.96** to

0.99** for SSP and 0.96** to 0.99** for lime indicating that, the Langmuir isotherm explains the adsorption process well and indicated that the adsorption takes place in a homogeneous monolayer (Kamal et al 2021) (Fig. 2, 3). Testing with the

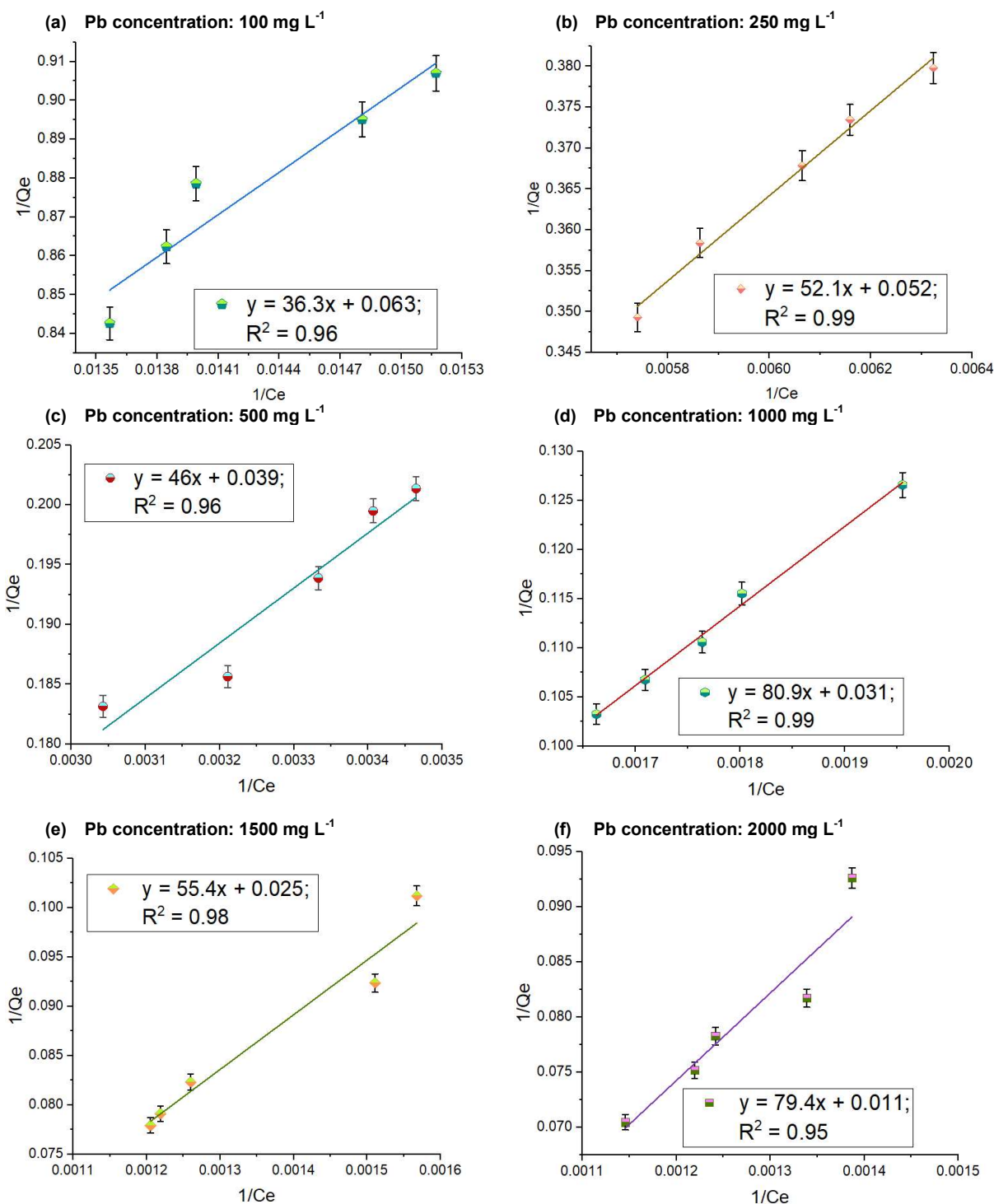


Fig. 2. Langmuir Adsorption Isotherm for describing the effect of SSP in removing Pb as influenced by varying initial Pb concentrations. Bars represent standard error; data are average of two replications; comparison of mean was done at 5% level

Langmuir isotherm where ' q_m ' is the maximum monolayer coverage capacity and ' K_L ' is the constant representing the affinity of active binding sites revealed a maximum monolayer coverage capacity (' q_m ') of 15.3 to 95.2 and 12.7 to

27.2 mg g^{-1} in SSP and lime respectively. The monolayer coverage capacity was observed to increase with increasing initial Pb concentrations. However, the ' K_L ' constant varied from 1.76 to 0.19 $\times 10^{-3} \text{ L mg}^{-1}$ for SSP and 1.29 to 0.50 $\times 10^{-3}$

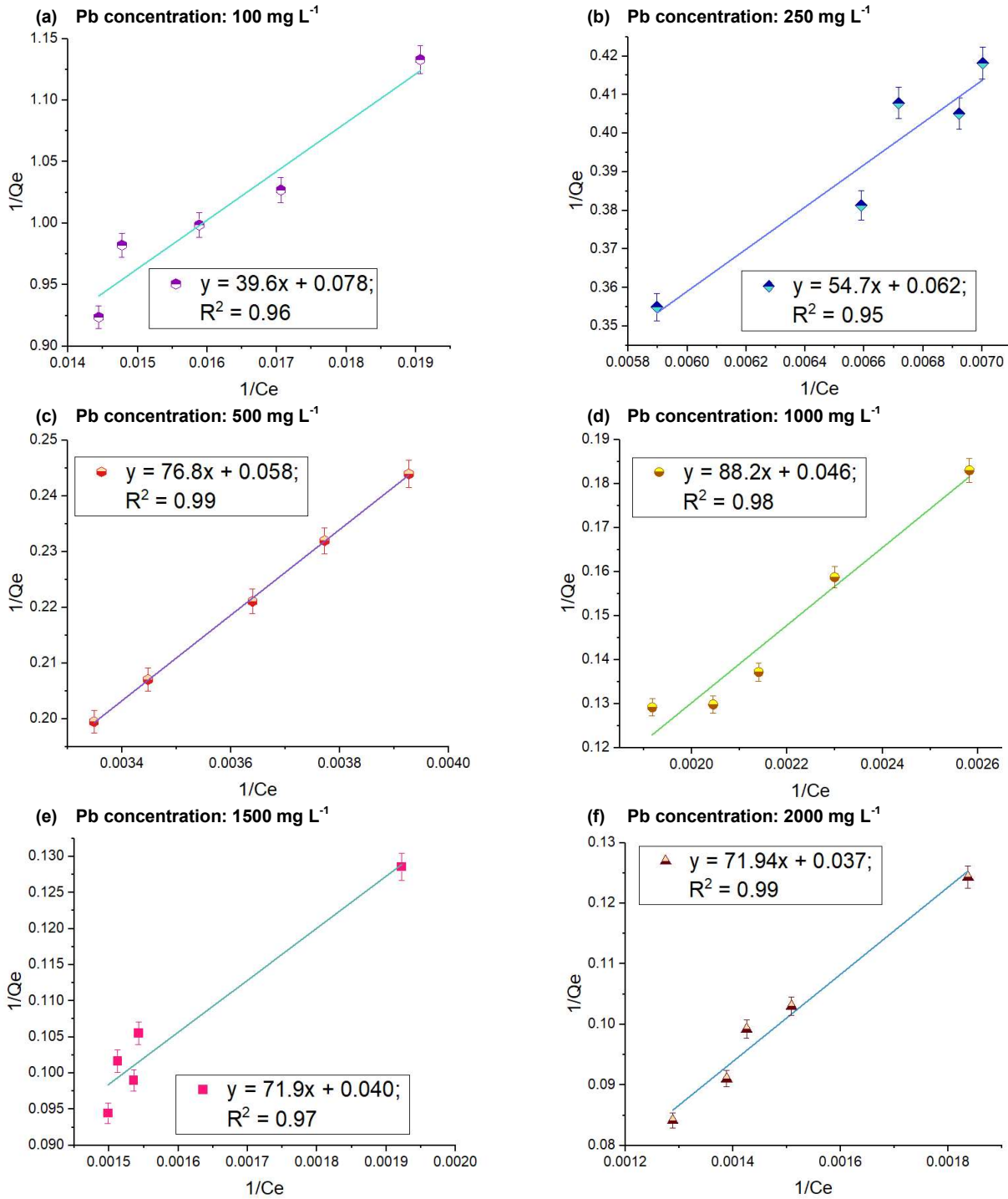


Fig. 3. Langmuir Adsorption Isotherm for describing the effect of lime in removing Pb as influenced by varying initial Pb concentrations. Bars represent standard error; data are average of two replications; comparison of mean was done at 5% level

for lime and higher binding affinity was observed at lesser initial Pb concentrations and declined with increasing initial Pb concentrations. This confirms that the affinity of Pb towards both the sorbents decreased with increasing initial Pb concentrations (Mouflih et al 2005, Mouflih et al 2006).

As regards to Freundlich isotherm, the coefficient of determination, (R^2) varied from 0.92** to 0.99** for SSP and 0.91** to 0.99** for lime showing that this model could be used to explain the adsorption process and the adsorption takes place on a heterogeneous surface (Fig. 4, 5). The

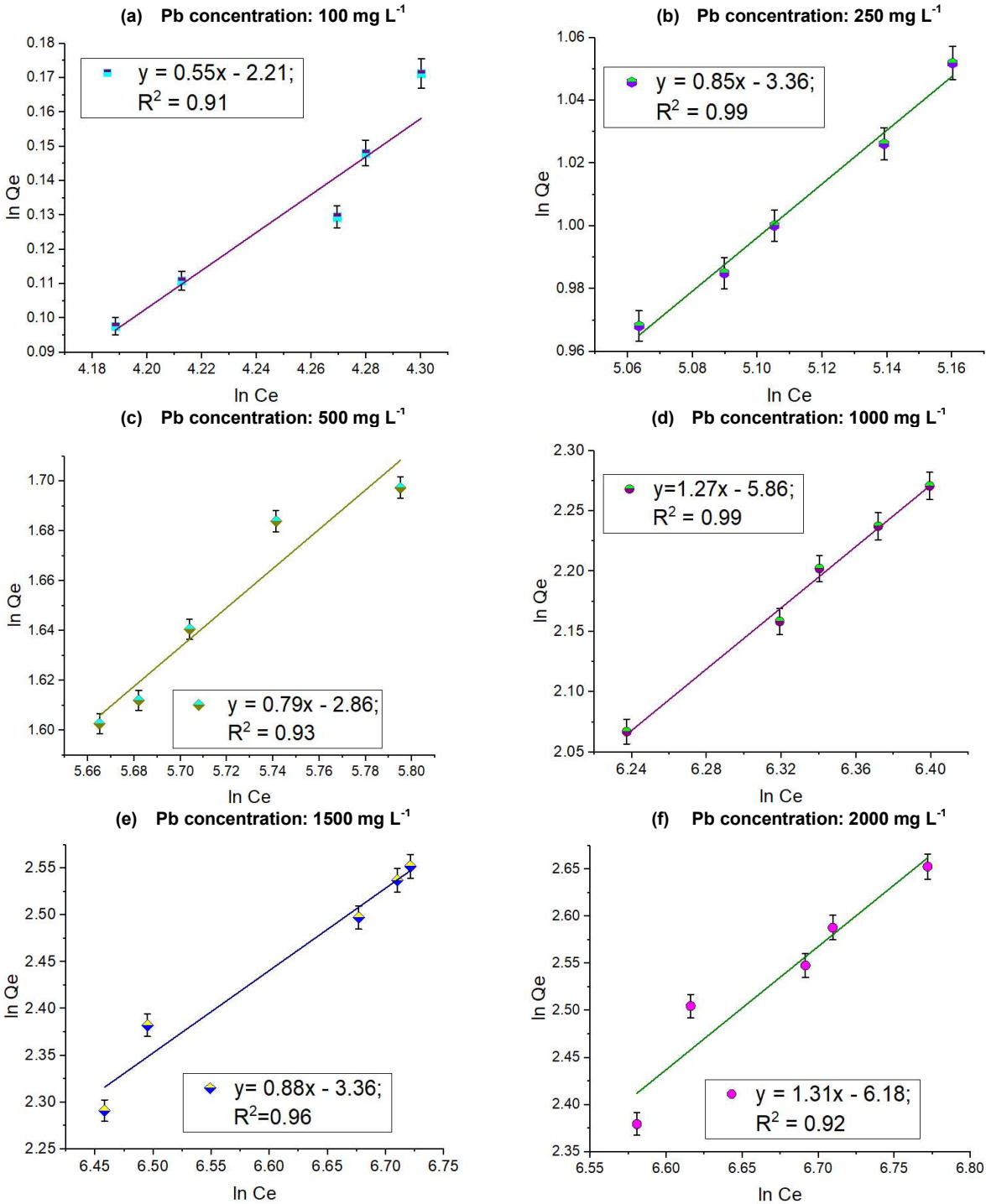


Fig. 4. Freundlich Adsorption Isotherm for describing the effect of SSP in removing Pb as influenced by varying initial Pb concentrations. Bars represent standard error; data are average of two replications; comparison of mean was done at 5% level

Freundlich isotherm assumes that the amount of adsorbed material increases with the increasing concentration of adsorbed material in solution. ' K_f ' is the adsorption capacity constant (mg g^{-1}) and ' η ' is the intensity of adsorbent. From

the estimated results, the intensity of adsorbent (' η ') varied from 0.78 to 1.69 for SSP and 0.78 to 1.57 for lime. Adsorption capacity constant ' K_f ' varied between 0.002 to 0.092 mg g^{-1} and 0.003 to 0.071 mg g^{-1} in SSP and lime

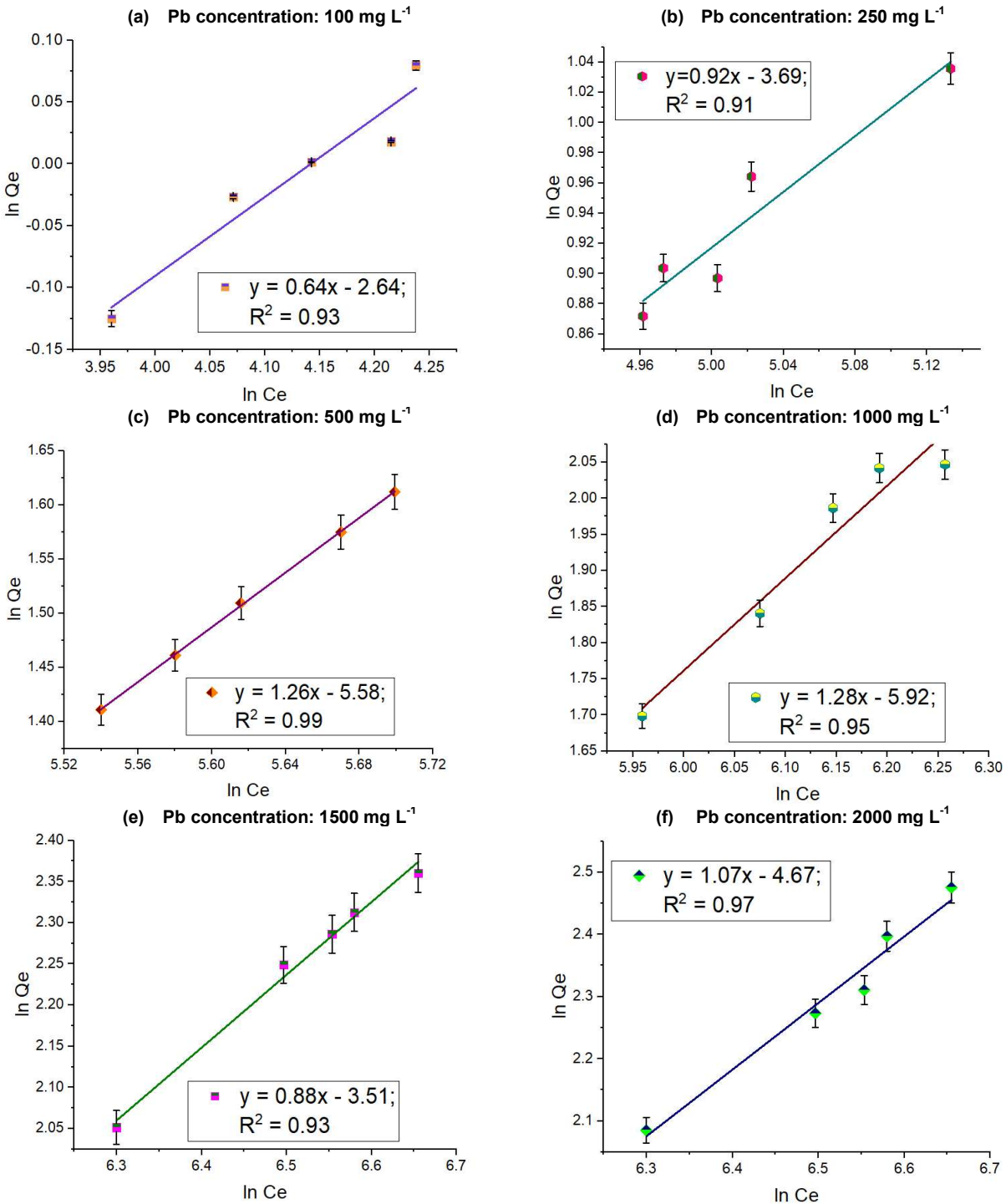


Fig. 5. Freundlich Adsorption Isotherm for describing the effect of lime in removing Pb as influenced by varying initial Pb concentrations. Bars represent standard error; data are average of two replications; comparison of mean was done at 5% level

Table 1. Description of Pb sorption by SSP and Lime using Freundlich and Langmuir isotherms

Sorbents	Pb concentration (mg L ⁻¹)	K _F	η	R ²	Q _m	K _L × 10 ⁻³	R ²
SSP	100	0.092	1.69	0.91**	15.8	1.76	0.96**
	250	0.034	1.17	0.99**	18.9	1.02	0.99**
	500	0.059	1.28	0.93**	25.2	0.86	0.99**
	1000	0.003	0.79	0.99**	32.2	0.38	0.99**
	1500	0.036	1.14	0.96**	40.4	0.30	0.98**
	2000	0.002	0.78	0.92**	95.2	0.19	0.95**
Lime	100	0.071	1.57	0.93**	1.98	1.29	0.96**
	250	0.025	1.09	0.91**	1.12	0.50	0.95**
	500	0.004	0.79	0.99**	0.75	0.75	0.99**
	1000	0.003	0.78	0.95**	0.52	0.52	0.98**
	1500	0.008	0.92	0.93**	0.55	0.55	0.97**
	2000	0.009	0.92	0.97**	0.51	0.51	0.99**

respectively. High K_F and η values indicate higher affinity of sorbent to sorption and desired levels of sorption process. These results are in line with the findings of Cekim et al (2015), Okoli et al (2017), Ozsin et al (2019) and Thabede et al (2020).

CONCLUSION

This study demonstrates that both SSP and lime showed higher Pb²⁺ sorption and removal potentials from aqueous solutions, however, SSP had higher Pb removal capacity than lime. The adsorption capacity increased with increasing Pb²⁺ concentrations and successive time intervals for both the sorbents. The models tested for sorbent-metal ion interactions showed that, Langmuir model explained the Pb²⁺ adsorption mechanism precisely explaining that adsorption takes place in a monolayer on a homogeneous sorbent surface. Hence it could be concluded that, SSP is the effective sorbent used for removing Pb²⁺ from the aqueous systems.

AUTHOR CONTRIBUTION

The research work was carried out and the first draft was written by Ms. G. Mohanapriya, Ph D. Scholar. Dr. T. Chitdeshwari, Professor (SS&AC) designed the methodology and edited the manuscript as Chairperson of the advisory committee. Dr. R. Shanmugasundaram, Dr. M. Maheswari and Dr. A. Senthil as advisory committee members edited the manuscript draft.

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