



## Reuse of alum sludge for phosphorus removal from municipal wastewater

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### ABSTRACT

In this study, the efficiency of alum sludge for removing phosphorus from synthetic and municipal wastewater (MWW) was investigated. Orthophosphorus (OP) and condensed phosphorus (CP) were used as model pollutants. Alum sludge (A and B) from two local treatment plants was collected, dewatered, dried, and processed before use. Batch experiments were performed to determine OP and CP removal under equilibrium conditions. Sludge dose, contact time, and pH were optimized for both sludges. Adsorption efficiencies of OP and CP were determined by using Langmuir and Freundlich adsorption isotherm models. The maximum adsorption capacity ( $Q_0$ ) for sludge A was found to be 4.86 mg/g for OP, and 4.21 mg/g for CP at 12 g/L of sludge dose, 90 min of contact time, and pH 4. For sludge B,  $Q_0$  was 1.58 mg/g for OP and 4.71 mg/g for CP at 30 g/L of sludge dose, 80 min of contact time, and pH of 5.5. Results showed that pH of wastewater significantly affected adsorption capacity and better removal was achieved within pH range of 4.0–5.5. Optimized conditions for sludge A and B were applied on MWW which provided over 90% of OP and 70–80% of CP removal. Sludge B performed better than sludge A in case of domestic wastewater. This study concluded that alum sludge as being a great resource for phosphorus removal from wastewater, and could be applied to streams feeding water supply reservoirs for prolonged oligotrophic conditions.

*Keywords:* Alum sludge; Wastewater; Orthophosphorous; Condensed phosphorus; Adsorption

### 1. Introduction

With greater understanding of impacts of wastewater on all living organisms and water bodies, the standards of wastewater are becoming stringent every day. In addition to organic matter and suspended solids, nutrients such as nitrogen (N) and phosphorus (P) require removal to meet treated wastewater standards.

Nutrient-rich wastewater released directly into water bodies leads to a number of environmental issues such as taste and odor problems, eutrophication leading to death of aquatic life, developing esthetically unpleasant sites, and enhanced water treatment cost [1,2]. Cyanobacteria are the indicator for eutrophication and are well known for fixing molecular nitrogen from air. Thus, nitrogen coming in the form of nitrite, nitrate, and ammonium ions from wastewater is not needed by these Cyanobacteria [3,4]. Therefore, lowering

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phosphorus concentration would be a major step towards delaying eutrophication and survival of water bodies.

Municipal wastewater (MWW) in developing countries is either discharged untreated or treated only to primary levels. Thus, MWW injects substantial amount of phosphorus into water bodies. Secondary and tertiary biological and physico-chemical treatment processes are generally expensive, and difficult to maintain and operate especially in developing countries [2]. Thus, low-cost alternative for phosphorus removal from wastewater would help maintaining good-quality supplies from wastewater receptors such as rivers, lakes, and reservoirs.

Nutrients removal through adsorption on variety of media is a widely known option in water and wastewater industry [5,6]. For this purpose, various materials have been tested like fly ash, red mud, slag, iron oxide tailing, activated alumina, ion-exchange resin, industrial by-products, alum sludge, etc. with varying degree of success [7–9]. When phosphorus is removed by calcium- and iron-containing products, they cause problems like increasing pH beyond desirable range and removal under aerobic conditions only while aluminum-containing products has the highest removal efficiency among all the materials [10–13].

Alum sludge is the left over waste material from drinking water treatment plants [14–17]. Water treatment plants utilize aluminum or iron salts with and without lime for coagulation of colloidal matter, color, and natural organic matter. Globally, over a million tons of alum sludge is produced by water treatment plants on daily basis [18]. The waste alum sludge is hard to dispose off without proper treatment.

Recently, plenty of studies have been conducted to determine the efficiency of alum sludge in removing phosphorus. A very little attention has been paid to natural distribution of phosphorus in wastewater and 95% of all the studies have only included soluble orthophosphorus (OP). In wastewater, phosphorus is broadly found in three groups: soluble OP, soluble Condensed phosphorus (CP), and particulate organic phosphorus [19,20].

The idea behind this research was to test and compare the effectiveness of alum sludge of two drinking water treatment plants for ortho and condensed phosphorous removal from synthetic and MWW. Adsorption studies were carried out to determine the efficacy of processed alum sludges for maximum adsorption capacity. Finally, optimized conditions were employed on MWW with great success.

## 2. Methods

### 2.1. Sludge processing and wastewater preparation

Alum sludge was collected from Simly and Rawal water treatment plants in Islamabad, Pakistan. Both water treatment plants use alum as sole coagulant. Simly alum sludge, here-in-after called sludge A, and Rawal alum sludge to be called as sludge B. Sludge from both sources was processed before experimental work. Sludge was processed involving air drying for 72 h at room temperature ( $25 \pm 3^\circ\text{C}$ ) followed by oven drying at  $103 \pm 2^\circ\text{C}$ . Dried sludge was ground with pestle and mortar manually, sieved through ASTM mesh # 10 to bring the particle size between 1.65 and 1.98 mm [10,12], and finally stored in a clean dry enclosure.

Elemental, physical, and chemical analyses of the sludges were carried out using BET single-point surface area analyzer, FTIR, and TOC analyzer. The qualitative analysis of dried sludge was carried out by XRF (Jeol 3202M Element Analyzer Na-U) and for quantifying the exact amount of elements present, ICP-OES-Inductively Coupled Plasma Optical Emission Spectroscopy was done (ICP-OES, Vista-Pro Axial, Varian Pty Ltd, Mulgrave, Australia). Leachability of the sludge A and B was tested using the extraction technique of the UK leach test and measuring the heavy metals concentration using flame atomic absorption spectrometry.

Phosphorus-rich wastewater (containing OP and CP) was prepared in the laboratory by dissolving 25 mg/L of potassium dihydrogen phosphate ( $\text{KH}_2\text{PO}_4$ ) and 15 mg/L of sodium hexa meta phosphate ( $\text{Na}_2\text{P}_3\text{O}_5 \cdot \text{H}_2\text{O}$ ) in distilled water. Phosphorus concentration was comparable with that generally found in typical MWW [6,18].

### 2.2. Phosphorus removal studies

Batch experiments were carried out to study the OP and CP removal by processed sludge. Effects of varying sludge dose on known concentrations of OP and CP were determined using jar testing apparatus (Phipps and Bird PB-700™). Sludge dose varying from 1 to 20 g/L for sludge A and 1 to 36 g/L for sludge B was added to the 1L OP- and CP-rich wastewater. The mixtures were then stirred at 200 rpm for specific contact time. After mixing, samples were allowed to settle for one hour. Settled samples were then filtered through 0.45  $\mu\text{m}$  cellulose filter to remove the sludge from wastewater. Reduction in phosphorous concentration from the solution was translated to be adsorbed on the processed alum sludge. The remaining OP and

CP concentrations were determined using ammonium molybdate as reagent with the help of HACH 2400 Spectrophotometer. In order to make CP to the readily available form, it was digested before determination.

Optimum phosphorous removal conditions were determined by using the optimized sludge dose and varying mixing time between 30 and 360 min. pH was also optimized within a pH range of 3.0–9.0 [15,16]. Keeping sludge dose and mixing time at constant optimum values, each experiment was carried out in triplicate to minimize experimental error.

Using the optimized conditions of sludge dose and contact time, the effect of variable OP and CP concentration was studied from 6 to 55 mg/L. The adsorption capacity at equilibrium was calculated by Eq. (1) [19,20].

$$q_e = \frac{C_i - C_s}{m} V \quad (1)$$

whereas,  $C_i$  (mg/L) is the initial concentrations of OP and CP,  $C_s$  (mg/L) is the final concentration for OP and CP,  $m$  (mg/L) is the adsorbent dose, and  $V$  is the volume of wastewater (L). These tests were performed both for sludge A and B.

Adsorption isotherm represents the quantitative relationship between adsorbent and the adsorbate at a fixed temperature under equilibrium conditions; furthermore, these represent the effect of initial concentration of the adsorbate as a function of adsorbent mass. In this study, Freundlich and Langmuir isotherms were applied.

The linear form of the Freundlich equation is:

$$\log q_e = \log K_F + \frac{1}{n} \log C_s \quad (2)$$

whereas,  $q_e$  = mass of P adsorbed on adsorbent at equilibrium (mg/g),  $C_s$  = equilibrium concentration of P solution (mg/L),  $K_F$  = Adsorption affinity (1/g), and  $n$  = deviation from linearity of the adsorption.  $n$  indicated the favorability of adsorption for Freundlich isotherm. If value of  $n = 1$ , linear adsorption, value of  $n > 1$ , favorable adsorption.

The linear form of the Langmuir isotherm equation is:

$$\frac{C_s}{q_e} = \frac{C_s}{Q_o} + \frac{1}{bQ_o} \quad (3)$$

whereas,  $q_e$  = mass of P adsorbed on adsorbent at equilibrium (mg/g),  $C_s$  = equilibrium concentration of P solution (mg/L),  $Q_o$  = adsorption capacity of

monolayer, and  $b$  = Langmuir constant (l/mg) attraction of the adsorbate for the adsorbent (affinity). The plot of  $C_s/q_e$  vs.  $C_s$  gives a straight line from which the isotherm parameters can be determined. The Langmuir dimensionless constant  $R_L$  called the equilibrium parameter indicates the type of isotherm.

$$R_L = \frac{1}{1 + bC_i} \quad (4)$$

whereas,  $C_i$  = initial phosphate concentration.

$R_L$  values indicate the favorability of isotherm for the experimental data. If  $R_L > 1$  isotherm is unfavorable, at  $R_L = 1$  linear,  $0 < R_L < 1$  favorable, and at  $R_L = 0$  isotherm becomes irreversible [20].

Finally, a 24 h composite sample of MWW was collected from the main drain of National University of Sciences and Technology (NUST). Batch tests were performed on this wastewater under the optimized condition to get the actual picture of efficiency of alum sludge in removing OP and CP.

### 3. Results and discussion

Qualitative analysis of the sludge A and B using X-ray fluorescence (XRF) revealed that four metals were prominently found in high concentrations in both sludge. These included aluminum (Al), calcium (Ca), magnesium (Mg), and iron (Fe). Later, another quantitative analysis carried out by inductively coupled plasma optical emission spectroscopy (ICP-OES) provided the exact concentrations of the metals present in each sludge. The results of ICP-OES are given in Table 1. pH level of both sludge A and B was found to be slightly basic. TOC tests results revealed higher total organic carbon in sludge B. Surface area calculated for both sludges under observation was very high i.e. 39.41 and 42.72 m<sup>2</sup>/g, indicating their high adsorption potential.

Table 1  
General characteristics and elemental composition of sludges A and B

Parameters	Sludge A	Sludge B
pH	7.1	7.5
Particle density (g/cm <sup>3</sup> )	1.81	2.11
TOC	1,298	1,486
Surface area (g/m <sup>2</sup> )	39.41	42.76
Al (mg/g)	174.60	108.00
Ca (mg/g)	214.00	240.00
Fe (mg/g)	17.31	14.70
Mg (mg/g)	48.05	44.04

Although elemental analysis illustrated a few elements in sludge profile, heavy presence of metals can have significant environmental impact and are hazardous to human health in the long term. So, heavy metal content of sludges, using UK Leach Test, was performed with distilled water as the leaching eluent. Heavy metal ions profile is presented in Table 2. Although the presence of few heavy metals like Fe and Al was detected in the chemical composition of the both sludges, the metals were not leached out significantly into solution form. Traces of some heavy metals were observed in the sludges; however, their concentrations were below threshold levels provided by DWAF guidelines [21].

### 3.1. Optimization of independent variables

Series of batch experiments were performed to evaluate alum sludge capacity for adsorption of phosphorus from the wastewater. Residual phosphorus showed good performance of alum sludges at equilibrium stage. Effect of sludge dose on pH, turbidity, and phosphorus removal are shown in Fig. 1(a).

As results indicate, there was potential decrease both in OP and in CP with rise in sludge dose up to a certain limit. Beyond 12 g/L of sludge A dose, further reduction in phosphorus was comparatively negligible. So, the maximum removal i.e. 79% for OP and 86% for CP was achieved at 12 g/L of sludge A after four hours of mixing. No significant change was observed in pH and turbidity, with increasing sludge dose. Sludge B also exhibited good removal i.e. 92% of OP and 89% of CP at 30 g/L after four hours of mixing. Again, no significant effect on pH and/or turbidity could be noticed.

In a similar study, Yang et al. [11] confirmed that adsorption potential of sludge depends upon its concentration. Higher amount results in larger percent removal of phosphorus. They obtained maximum removal of 90% at 5.0 g/L of sludge. Mohammad and

Rashid [10] reported that removal of phosphorus depended upon the availability of adsorption sites. They used sludge dose from 5 to 50 g/L and achieved 85% removal of phosphorus at 50 g/L of sludge dose.

### 3.2. Effects of equilibrium time

To determine the time required for the adsorption process to reach the equilibrium stage, the adsorption of phosphorus species onto the sludge was studied as a function of mixing time. Fig. 1(b) shows the profile of the remaining phosphorus in the wastewater after reaction with optimum alum sludge dosages at various mixing times.

As evident from Fig. 1(b), remaining concentration of OP and CP decreased rapidly in first 50 min of mixing. It's clear from the graphs that, after 90 min for sludge A and 8 min for sludge B, there was hardly any subsequent phosphorous removal. So the time optimized for further experiments was 90 min for sludge A and 80 min for sludge B. These results correspond to those obtained by Yang et al. [11] where P concentration decreased significantly for first 2 h. Xiaohong [7] however, used fresh alum sludge and achieved equilibrium within 30 min of contact time using only OP as P specie and obtained 90% removal.

### 3.3. Effect of pH

Reaction between the phosphate in wastewater and alum sludge is highly pH dependent [12]. Fig. 1(c) illustrates a trend that higher phosphorous removal can be accomplished in the acidic pH range. In case of sludge A, removal of OP and CP was almost the same within a pH range of 4.0–6.0. In case of sludge B, removal of CP varied from 90 to 83% when pH was varied from 4.0 to 5.0.

Higher removal of OP and CP was achieved when pH of the solution was in the acidic range i.e. pH 4.0–6.0 (Fig. 1(c)). When the pH of the wastewater dropped below 4.0,  $\text{AlPO}_4$  was formed. This  $\text{AlPO}_4$  molecule became soluble below pH 4.0 due to its chemical characteristics [7]. Now, due to the dissolution of  $\text{AlPO}_4$  into the solution matrix, adsorbed  $\text{PO}_4^{3-}$  got re-dissolved into the solution, thus reducing percentage removal of phosphates.

As the pH increased,  $\text{OH}^-$  ions increased in solution. These  $\text{OH}^-$  ions surround aluminum ion and repel phosphate ions. With the increment of the pH, the net surface charge on the alum sludge shifted from positive to negative [20]. This net negative charge on the surface of alum sludge also repelled the phosphates ions present in the solution thus phosphate

Table 2  
Heavy metal leached from sludges A and B samples

Metals (mg/kg)	Sludge A	Sludge B
Fe	N.D	N.D
Cr	0.13	0.08
Ni	0.21	N.D
Cd	N.D	0.38
Pb	3.63	2.74
Al	1.63	3.17
Cu	0.03	0.16
Zn	0.06	N.D

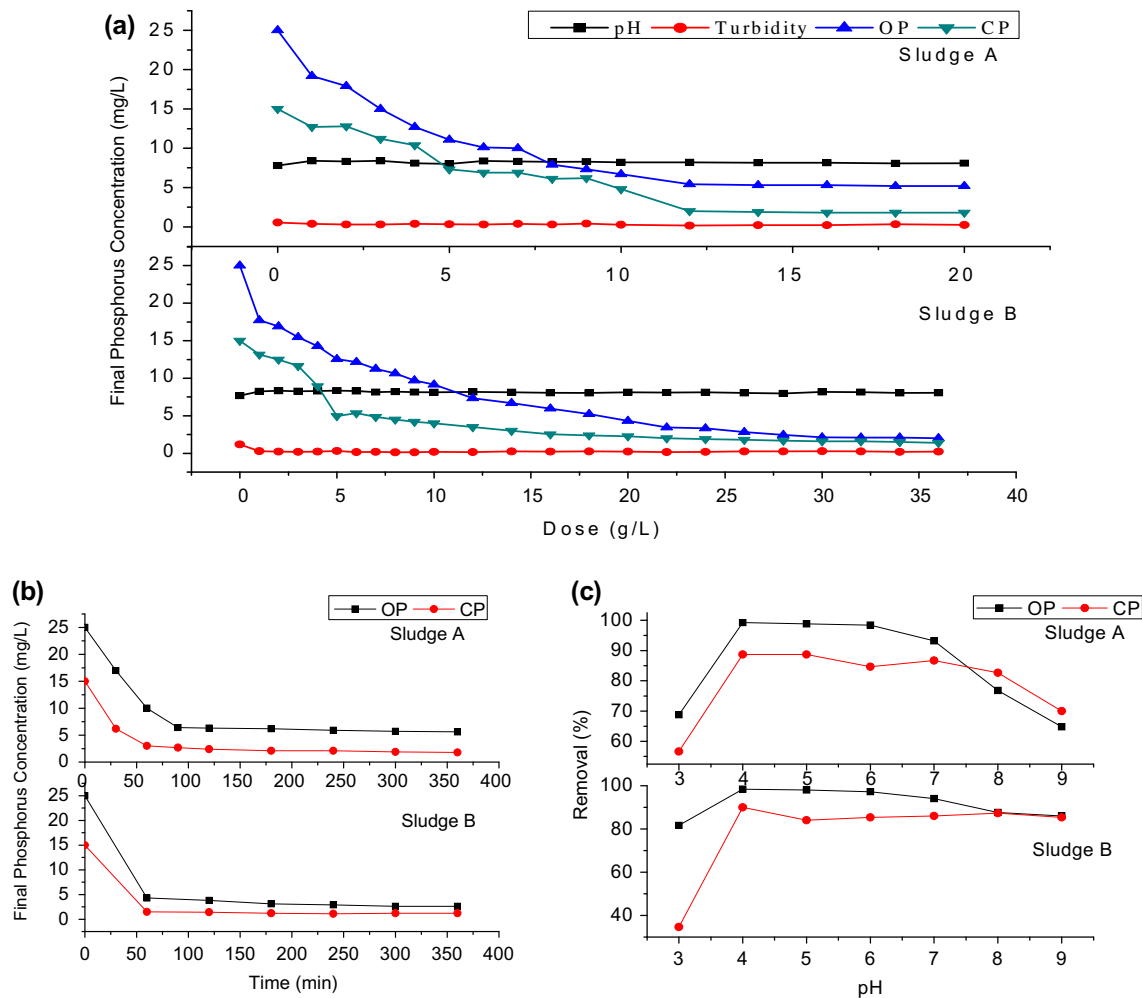


Fig. 1. (a) Effect of sludge dose on pH, turbidity, OP and CP, (b) effect of contact time on effluent phosphorus concentration (mg/L), and (c) effect of pH on percent OP/CP removal %.

ions did not get adsorbed on alum sludge surface. These results are in the cognition of the results obtained by other researchers [12,17], they reported that acidic environment is favorable for the adsorption of OP and CP on alum sludge, and adsorption of OP and CP on alum sludge was maximum at pH 4.0.

### 3.4. Adsorption study

Phosphorus adsorption is considered as a kinetically biphasic process [17]. That is, it has a rapid initial reaction, than a slow later reaction that can last for longer. The rapid initial phase represents anion exchange and ligand exchange on mineral edges or by amorphous oxides and carbonates. The slow reaction involves precipitation or polymerization

on mineral surfaces or diffusion of adsorbed P into the interior of solid phases [7,11]. The effect of increasing concentration  $C_s$  of OP and CP on the adsorption capacity ( $q$ ) of sludge A and sludge B is shown in Fig. 2.

Results as depicted in Fig. 2 showed that adsorption capacity of alum sludge increased up to a specific level until equilibrium was reached. After achieving equilibrium, further rise in initial concentration of OP and CP did not affect the results. The maximum adsorption capacity of sludge A for OP was 3.58 mg/g and for CP was 2.83 mg/g at initial concentration of 55 mg/L. The maximum adsorption capacity of sludge B for OP was 1.36 mg/g and CP was 1.27 mg/g at initial concentration of 55 mg/L.



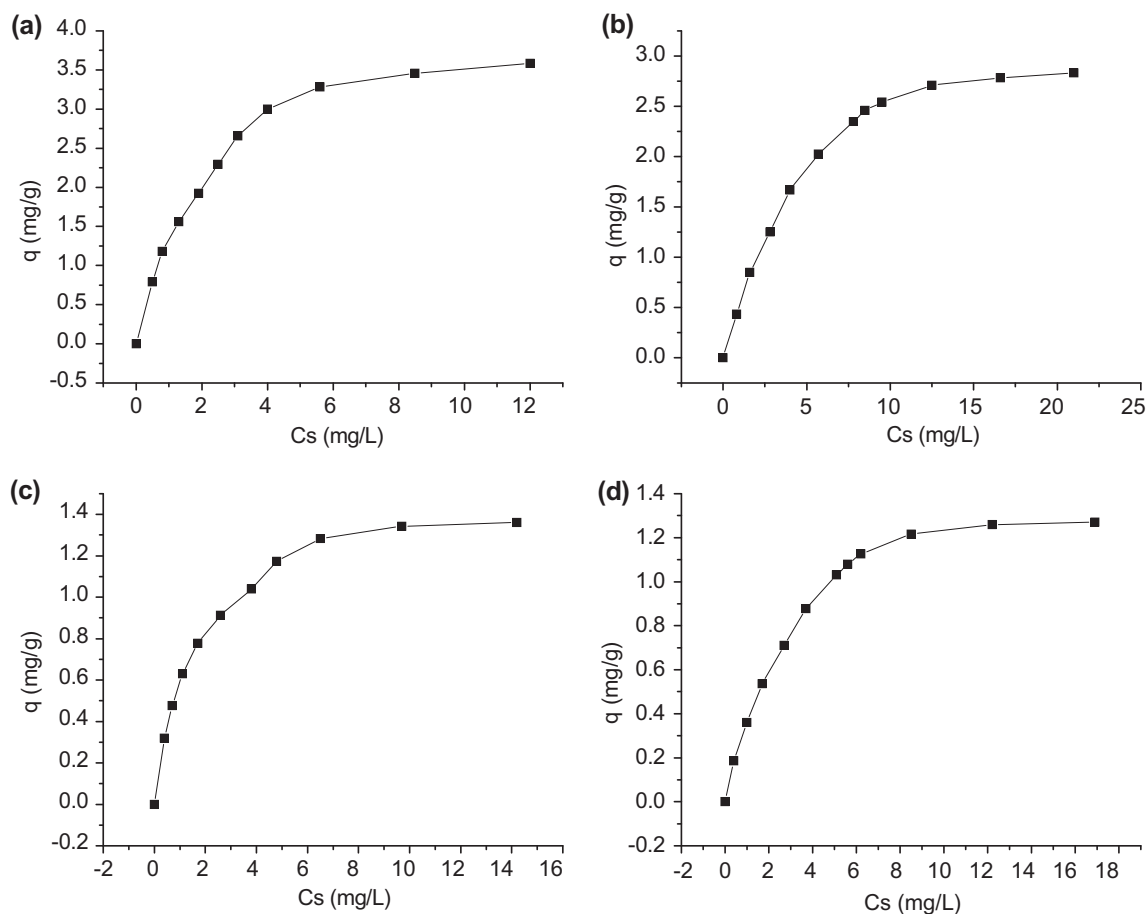


Fig. 2. Final concentration ( $C_s$ ) vs. adsorption capacity ( $q$ ) of OP for sludge A (a), CP for sludge A (b), OP for sludge B (c), and CP for sludge B (d) at  $25 \pm 2^\circ\text{C}$ .

## 4. Isotherm studies

### 4.1. Freundlich isotherm

Freundlich isotherm (Eq. (2)) describes the heterogeneity of the adsorbent.  $K_F$  and  $n$  are the factors affecting adsorption process i.e. adsorption affinity and nonlinearity. Higher  $K_F$  values show that higher is the adsorption capacity.  $n$  shows the deviation of data from linearity and decreases as the concentration of the pollutant (Phosphate) increases in the solution. The values of constants ( $K_F$ ,  $n$ , and  $R^2$ ) are given in Table 3. Fig. 3 shows the linear form of Freundlich isotherm.

The value of  $K_F$  and  $n$  are higher for OP than CP in case of sludge A. This indicates that OP has higher adsorption affinity as compared to CP.  $K_F$  value for OP is almost double of CP in sludge A. Thus, it can be concluded that tendency of sludge A to adsorb OP is higher than sludge B. In case of sludge B,  $K_F$  values

are higher for CP than OP but this difference is not significant.

In case of this study,  $n$  value falls within the permissible range of  $n > 1$  as previously described in material and methods.  $n$  values show that the adsorption is favorable.  $R^2$  values also show that data fits well on Freundlich isotherm but less favorably when compared to Langmuir isotherm when compared to linear regression ( $R^2$ ) values [20].

### 4.2. Langmuir isotherm

The Langmuir adsorption model is the most common model used to quantify the amount of adsorbate adsorbed on an adsorbent as a function of concentration at a given temperature. This isotherm advocates that effective adsorption is monolayer in nature. In Langmuir adsorption isotherm (Eq. (3)),  $Q_o$  represents the maximum adsorption capacity and  $b$  is

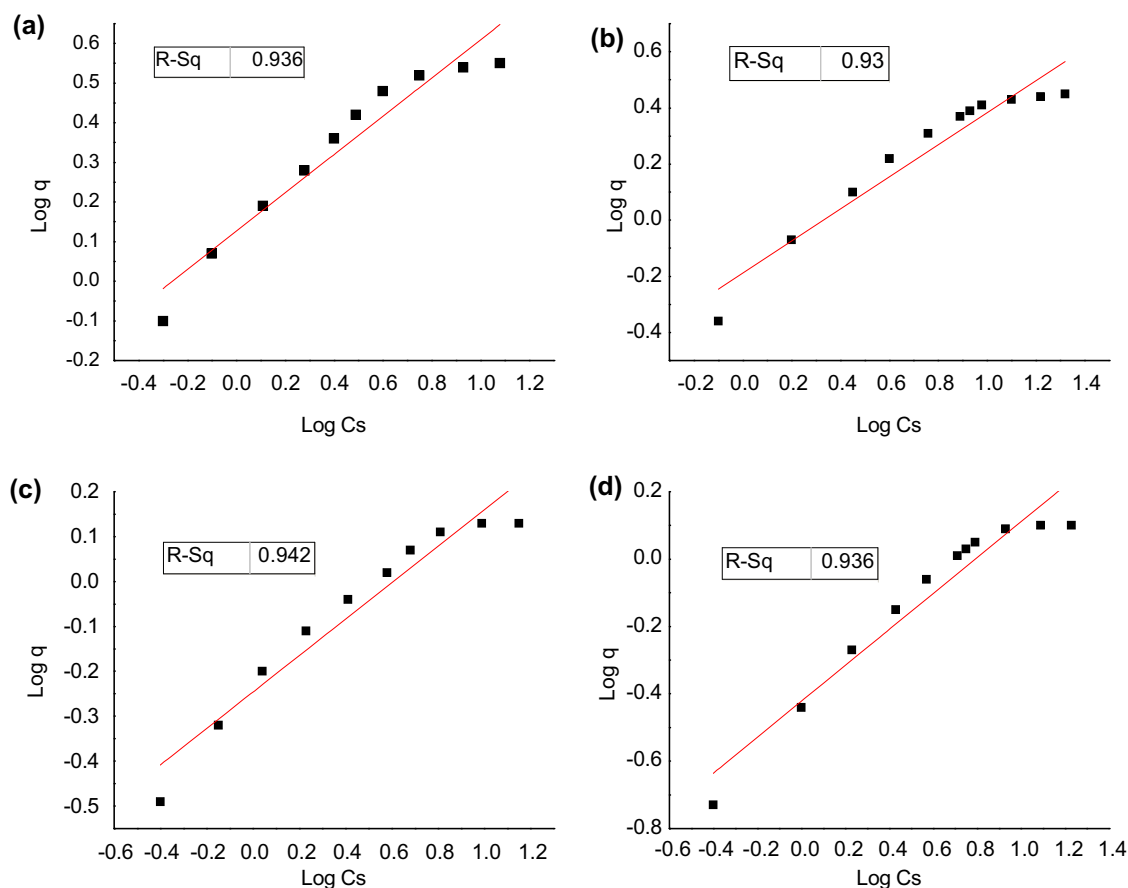


Fig. 3. Freundlich isotherm for OP (a), CP (b) for sludge A and OP (c), and CP (d) for sludge B.

the adsorption affinity. The values of constants ( $Q_0$ ,  $b$ , and  $R^2$ ) obtained from trend lines (Fig. 4) are given in Table 1.

As given in Table 3, adsorption capacity,  $Q_0$  of sludge A was 2–3 times higher both for OP when compared with sludge B. The highest adsorption capacity of sludge A was 4.86 mg/g for OP and 4.21 mg/g for CP. In case of sludge B,  $Q_0$  for both OP and CP was 1.58 and 4.71 mg/g respectively. The favorability of Langmuir isotherm depends upon  $R_L$  factor known as equilibrium parameter (Eq. (4)). Langmuir isotherm fitted the data fairly well because the value of  $R_L$  ranged between 0.03 and 0.32 i.e. within 0–1 indicating that the data laid in favorable mode as reported by Babatunde and Zhao [19]. Hence, experimental data were favorable for Langmuir isotherm. The coefficient of correlation ( $R^2$ ) also confirmed that Langmuir isotherm was applicable on the data. Hence, monolayer Langmuir isotherm was favored over Freundlich isotherm for the adsorption of OP and CP on both sludge A and B.

High diversity of the results in similar studies [6,11] could be attributed to the composition of the

alum sludge. Physical and chemical properties of alum sludge depend upon the type of alum used for coagulation and characteristics of suspension in raw water. The composition of alum sludge varies enormously from plant to plant. This difference leads to variations in their adsorption potential for phosphorus.

#### 4.3. Phosphorous removal from MWW

Real wastewater samples were collected from NUST main drainage and analyzed for OP and CP. It contained 24.5 mg/L of OP, 13.3 mg/L of CP, and 5.2 mg/L of Organic phosphates (Org. P). This high concentration of phosphorus was due to excessive use of detergents, soaps, and fecal materials. Optimized dose and contact time for sludge A and B were employed on real wastewater samples. About 85% of OP, 69% of CP, and 50% of Org. P was removed using 12 g/L of sludge A. About 88% of OP, 80% of CP, and 60% of Org. P was achieved using 30 g/L of sludge B.

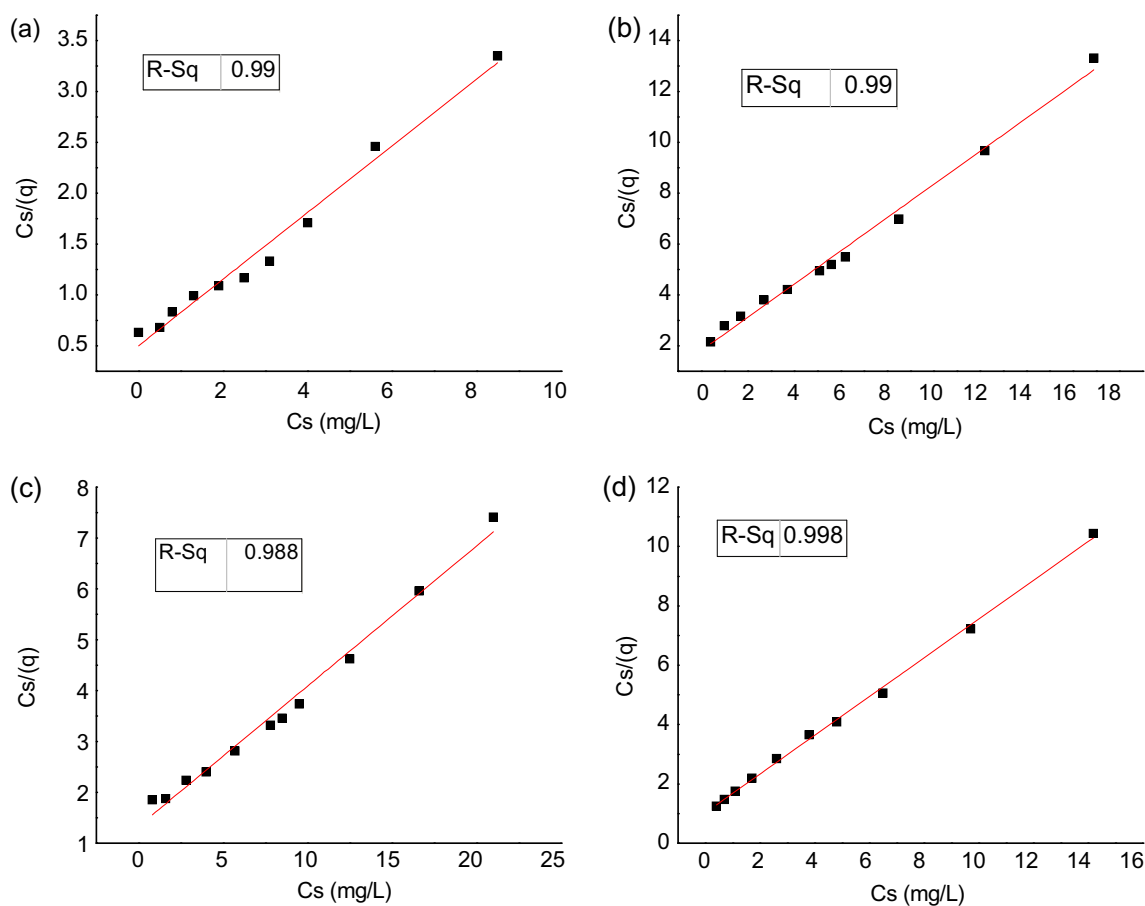


Fig. 4. Langmuir isotherm for OP (a), CP (b) for sludge A and OP (c), and CP (d) for sludge B (from left to right).

Table 3  
Constants of Freundlich and Langmuir isotherm for sludges A and B

Adsorbent	Freundlich constants					
	OP			CP		
	$K_f$	$n$	$R^2$	$K_f$	$n$	$R^2$
Sludge A	1.34	2.07	0.936	0.64	1.74	0.930
Sludge B	0.56	2.45	0.942	0.67	1.86	0.936
Adsorbent	Langmuir constants					
	OP			CP		
	$b$	$Q_o$ (mg/g)	$R^2$	$b$	$Q_o$ (mg/g)	$R^2$
Sludge A	0.43	4.86	0.993	0.21	4.21	0.988
Sludge B	0.64	1.58	0.998	0.36	4.71	0.992

### 5. Conclusions

This study concludes that waste alum sludge provides a relatively high phosphorous adsorption capacity due to small particulate materials, which provides

its high surface area for interparticulate bonding. Moreover, Leach Tests indicated that traces of Pb, Fe, Ni, Al, and Cr were leached out by distilled water from the sludges in permitted levels, so these sludges



can safely be used as a great resource for phosphorous removal. Both models proved to be favorable for sludge A and B. However, on the basis of  $R^2$  value, Langmuir adsorption isotherm fitted better on the adsorption data generated in this study, compared to Freundlich isotherm. This showed that on processed sludge, a monolayer adsorption was favored over multilayer. Using optimized dose, pH and mixing conditions, highest removal efficiency achieved for sludge A for OP and CP was 99 and 89%, respectively. Similarly, under optimized condition for sludge B, maximum OP removal of 98% and CP removal of 90% was achieved.

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