



# Removal of Phosphate from Aqueous Solution Using Anion Exchange Resin: Equilibrium Isotherms and Kinetics

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## Abstract

Phosphate removal before discharging wastewater into the receiving environment is important since eutrophication has become an environmental problem on a global scale. In this study, phosphate ion removal from aqueous solutions was investigated using a strong anion exchange resin, Purolite A200E. The effects of initial phosphate ion concentration, initial pH value of the solution, resin dosage, stirring speed, presence of some anions in solution, and temperature on the ion exchange process were researched. Resin dosage, stirring speed, and temperature increases were determined to increase the phosphate removal rate. The highest phosphate removal efficiency was observed in the pH interval 7–9. With initial phosphate concentration of 10 and 20 mg/L, 1.50 g/500 mL resin dosage fully removed phosphate at the end of 120 min. However, at phosphate concentrations higher than 20 mg/L, increasing phosphate concentrations caused a reduction in removal efficiency. The competitive anion with the most effect on phosphate removal was sulfate while adding bicarbonate did not affect phosphate removal. Pseudo-first-order (PFO) and pseudo-second-order (PSO) kinetic models were used to assess experimental data. Kinetic studies revealed that the ion exchange process can be explained better by the PFO kinetic model. Equilibrium isotherm data were analyzed with the Freundlich and Langmuir equations, and the Freundlich isotherm model fitted the equilibrium data better.

**Keywords** Eutrophication · Phosphate removal · Ion exchange · Purolite A200E

## 1 Introduction

Phosphorus is a source of energy and basic nutrients for all organisms. It is an element commonly used in agricultural, industrial, and daily products. Phosphorus release has significantly increased with agricultural waste, urban wastewater, industrial wastewater, burning of fossil fuels, and use of fertilizers [1–3]. Phosphorus forms found in aqueous media include orthophosphates, polyphosphates, and organic phosphates. The greatest disadvantage of excessive release of phosphorus compounds into aqueous media is that it causes eutrophication in rivers, lakes, and seas [4–6]. Eutrophication causes consumption of excessive amounts of oxygen in aqueous media, which endangers living organisms [7]. At the same time, it causes negative effects, such as disruption

of water quality, bad appearance, odor problems, taste variations, increases in toxic reduced matter levels, and resulting difficulty in treatment processes. For this reason, researchers paid attention to the importance of phosphorus removal to control eutrophication [8–10]. The surface water quality management regulation in Türkiye states that phosphorus removal is mandatory for discharge in natural and artificial water areas which are eutrophic or at risk of eutrophication with the aim of protecting water. The total phosphorus limit value was determined as 0.03 mg/L to control eutrophication [11]. Some countries in the world implement similar regulations [1, 12].

With the aim of controlling eutrophication, it is necessary to remove phosphorus immediately before discharging wastewater into the receiving aqueous environment. Many methods are effectively applied for phosphorus removal. These methods may be listed as biological treatment [13], chemical precipitation [14], electrocoagulation [15], membrane filtration [16], and ion exchange [17]. The biological treatment process has some disadvantages due to requiring continuous control of several parameters, such as

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temperature, carbon source, alkalinity, oxygen concentration, and processing difficulties; in addition, it is not suitable for the removal of phosphorus at trace levels [18–21]. When the chemical precipitation process is not optimized, it may create additional pollution due to chemical materials used and additionally sludge forms linked to the chemical materials [19, 20, 22].

A variety of chemicals can be effectively removed from polluted natural waters and wastewaters using the ion exchange process. For example, removal of anionic matter such as nitrate, nitrite, phosphate, uranium, and phenols can be performed with anion exchange resins [23–27]. In addition, the removal of ammonium, iron, copper, and zinc from liquid solutions were tested using different cation exchange resins [28–30]. Synthetic polymeric ion exchangers are accepted as one of the best technologies for phosphorus removal due to good mechanical strength, different structures, and rapid regeneration capacity [7, 31]. The ion exchange method has the ability to remove shock loads; in addition, it ensures removal of trace levels of phosphorus and can operate in a broad temperature interval [18, 32]. Furthermore, the ion exchange process can be used not only for phosphorus removal, but also for phosphorus recycling [33]. However, the production costs of these synthetic polymeric materials and the effect of other anions present in the water limit their broad application [21, 34]. For this reason, laboratory studies were performed with the aim of determining removal performances of resins sold commercially.

The aim of the study was to research phosphate removal from aqueous solutions using Purolite A200E anion exchange resin. In line with this aim, a batch reactor was used and the effects of initial pH, resin dosage, stirring speed, initial phosphate concentration, competitive ions, and temperature on phosphate removal were researched. Furthermore, the phosphate removal kinetics were investigated using the PFO and PSO kinetic models. Finally, experiments were completed with the aim of determining the best isotherm model (Langmuir, Freundlich) to describe the phosphate equilibrium between fluid phase and resin.

## 2 Experiment

The study used the strong anion exchange resin, Purolite A200E, as ion exchange resin. The properties of the resin used are given in Table 1. For preparation of solutions containing phosphate ions,  $\text{KH}_2\text{PO}_4$  was used. To set the pH values of the solutions, HCl and NaOH solutions were used. Setting of the pH used a WTW Multi 3620 IDS portable multiparameter meter.

The solution volume of the jacketed reactor used in the experiments was 500 mL, with a mechanical stirrer used for mixing. The temperature in the reactor was fixed to the

**Table 1** Typical chemical and physical characteristics of Purolite A200E

Property	Description
Polymer Structure	Gel polystyrene crosslinked with divinylbenzene
Functional Group	Type 2 Quaternary Ammonium
Appearance	Spherical beads
Ionic Form	$\text{Cl}^-$
Capacity	1.30 eq/L $\text{Cl}^-$
Mean Size Typical	0.60–0.85 mm
pH Limits	0–14 (stability)
Temperature Limit	85 °C
Moisture Retention	45–51%

desired temperature using a heating–cooling circulator. Samples taken linked to time in each experiment had phosphate concentrations determined using a Merck Millipore 114,848 test kit with a spectrophotometer. The phosphate percentage removed ( $R$ ), time-linked ion exchange capacity, and ion exchange capacity at equilibrium were calculated using Eqs. (1)–(3):

$$R(\%) = (1 - C_t/C_0) \times 100 \quad (1)$$

$$Q_t = [(C_0 - C_t) \times V] / m \quad (2)$$

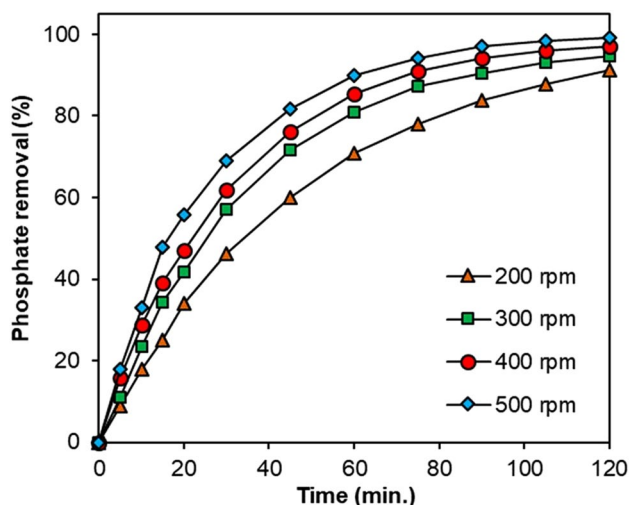
$$Q_e = [(C_0 - C_e) \times V] / m \quad (3)$$

where  $C_0$  is initial phosphate concentration,  $C_t$  is phosphate concentration at time  $t$ ,  $C_e$  is phosphate concentration at equilibrium,  $V$  is the reactor volume,  $m$  is the resin amount,  $Q_e$  is the phosphate exchange capacity of the ion exchange resin at equilibrium, and  $Q_t$  is the phosphate exchange capacity of the ion exchange resin at time  $t$ .

## 3 Results and Discussion

### 3.1 Effect of Stirring Speed

Mixing is an important factor for ion exchange processes in a batch-operated fully mixed reactor [35]. With the aim of determining the effect of stirring speed, the resin dosage was set to 0.5 g/500 mL, temperature 20 °C, initial pH 7, and initial phosphate concentration 10 mg/L and the effects of different stirring speeds from 200 to 500 rpm were researched. The results are given in Fig. 1. Results show that the increase in stirring speed had a positive effect on phosphate removal. At the end of 120 min, phosphate removal was 91%, 95%, 97%, and 99% at 200, 300, 400, and 500 rpm stirring speeds,

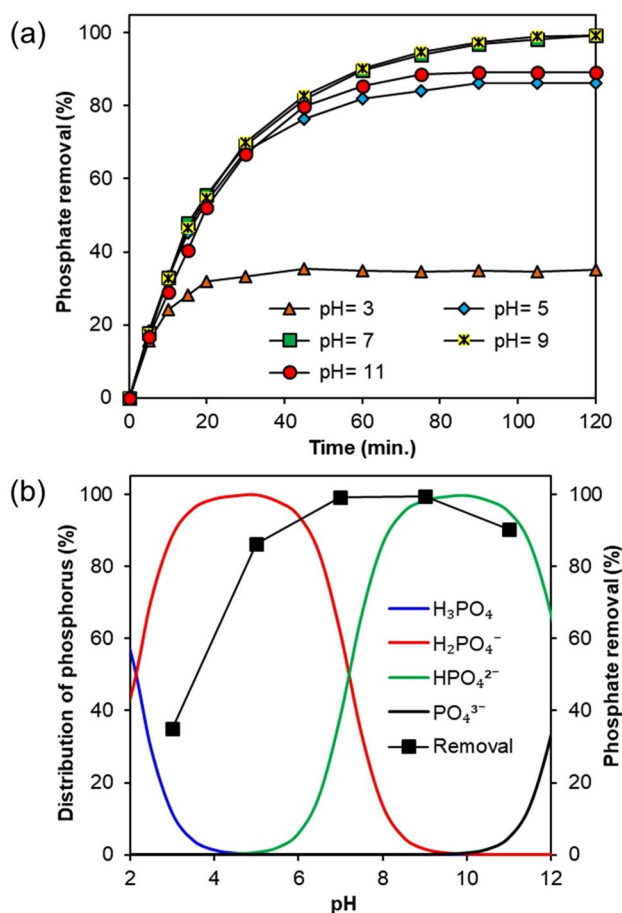


**Fig. 1** Effect of stirring speed on phosphate removal (Resin dosage = 0.5 g/500 mL, initial pH = 7,  $C_0 = 10$  mg/L,  $T = 20$  °C)

respectively. The increase in the stirring speed ensured better interaction between the resin and phosphate ions and the highest phosphate removal was obtained at 500 rpm. This situation may be linked to the reduction in fluid film thickness with the increase in stirring speed [36, 37]. For this reason, the transfer of phosphate ions to the resin surface accelerates. As a result, the optimum stirring speed of 500 rpm was chosen for later experiments.

### 3.2 Effect of Solution pH

The distribution of phosphate ions in solution is linked to the pH of the solution; for this reason, solution pH is an important control parameter for the ion exchange process [6]. To determine the effect of pH value on phosphate removal, phosphate removal was investigated by performing serial experiments with the initial phosphate concentration fixed to 10 mg/L, stirring speed 500 rpm, resin dosage 0.5 g/500 mL, and temperature 20 °C, while the pH values were varied from 3 to 11 (Fig. 2). The results show that pH is significant for phosphate removal. As can be seen in Fig. 2, the phosphate removal was 35% at the end of 120 min at pH 3. At pH 5, the phosphate removal was nearly 86%, while at pH 7 and 9, more than 99% phosphate removal was provided. When the solution pH rose to 11, the phosphate removal reduced to 90%. The variation in phosphate removal linked to pH may be explained by the different ionic species of phosphate ion in solution linked to the pH value (Fig. 2b). At pH 3, the phosphate forms are  $H_3PO_4$  and  $H_2PO_4^-$  and ion exchange of these two forms are more difficult than chloride ions. In addition, as the solution pH was set with hydrochloric acid or sodium hydroxide, addition of chloride ions to the solution may prevent ion exchange between phosphate and

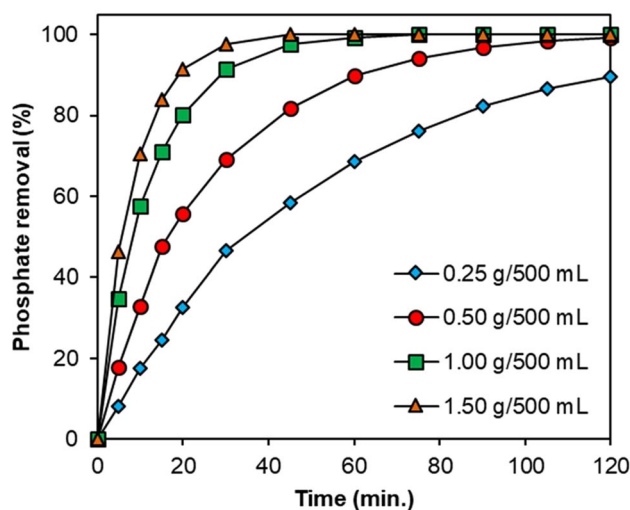


**Fig. 2** Effect of solution pH on phosphate removal (stirring rate = 500 rpm, resin dosage = 0.5 g/500 mL,  $C_0 = 10$  mg/L,  $T = 20$  °C)

chloride. At values of solution pH from 5 to 11, the dominant ionic species are  $H_2PO_4^-$  and  $HPO_4^{2-}$ . From the results, the ion exchange capacity for  $HPO_4^{2-}$  on the resin surface can be said to be higher than for  $H_2PO_4^-$ . However, at pH 11, the phosphate removal reduces due to competition between the phosphate ions and hydroxyl ions in the solution [38] or to formation of  $PO_4^{3-}$ . For this reason, pH 7 was chosen as the optimum value for later experiments.

### 3.3 Effect of Resin Dosage

Considering the economic factor, using less resin to ensure maximum phosphate removal is greatly important. Full exposure of phosphate ions to ion exchange sites with the addition of low amounts of resin makes it easier for a resin to reach maximum ion exchange capacity [18]. For this reason, experiments were performed with resin dosages of 0.25, 0.5, 1, and 1.5 g/500 mL to determine the effect of resin dosage on phosphate removal efficiency. In these experiments, values were set to initial phosphate concentration 10 mg/L,

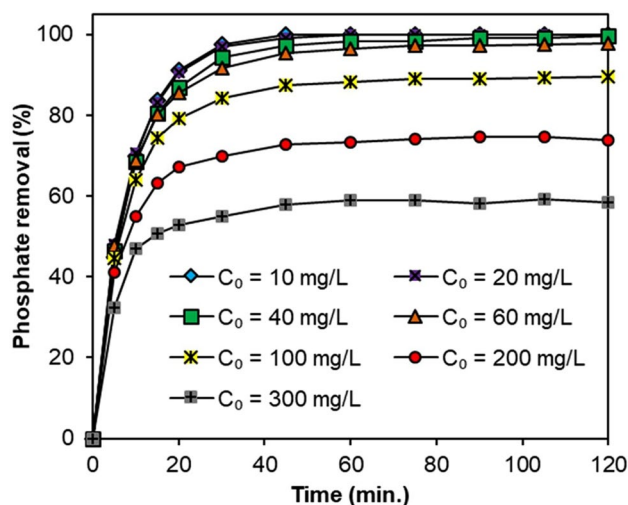


**Fig. 3** Effect of resin dosage on phosphate removal (stirring rate = 500 rpm, initial pH = 7,  $C_0 = 10$  mg/L,  $T = 20$  °C)

initial pH 7, stirring rate 500 rpm, and temperature 20 °C and the results are given in Fig. 3. The increase in resin dosage from 0.25 to 1.5 g ensured the phosphate removal increased from nearly 47–98% at the end of 30 min contact duration. At the end of 120 min, the phosphate removal with 0.25 g resin dosage was 90%, while the phosphate removal was more than 99% for resin dosages of 0.5, 1.0, and 1.5 g. As the ion exchange process occurs on the resin surface, the surface area was increased with the increase in resin dosage and for this reason, there was an increase in phosphate removal efficiency. As understood from the results, changing the dosage ensured both rapid and efficient removal due to sufficient contact between resin and phosphate and optimization of interaction.

### 3.4 Effect of Initial Phosphate Concentrations

With the aim of determining the effect of initial phosphate concentration, experiments were performed with other parameters fixed, while initial phosphate concentrations varied from 10 to 200 mg/L to investigate phosphate removal. Experiments used resin dosage 1.5 g/500 mL, initial pH 7, stirring speed 500 rpm and temperature 20 °C. Experimental results are given in Fig. 4. Figure 4 shows that the increase in initial phosphate concentration reduced the phosphate removal efficiency. With initial phosphate concentrations of 10 and 20 mg/L, nearly all phosphate was removed at the end of 120 min. At higher concentrations, phosphate removal reduced. With stable amounts of resin, the number of sites, where phosphate ions can be exchanged on the resin surface are fixed so the increase in initial phosphate concentration reduces removal efficiency. For example, there was 98% phosphate removal at 60 mg/L initial phosphate

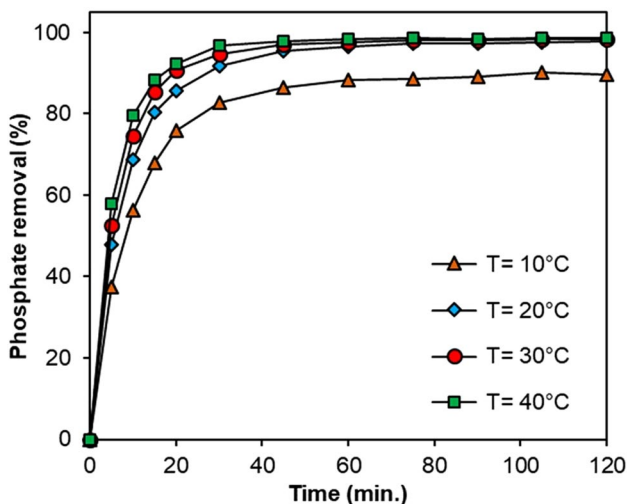


**Fig. 4** Effect of stirring speed on phosphate removal (Resin dosage = 1.5 g/500 mL, initial pH = 7, stirring rate = 500 rpm,  $T = 20$  °C)

concentration, while this fell to 74% for 200 mg/L phosphate concentration. However, the increase in phosphate concentration increased the phosphate exchange capacity of the ion exchange resin. The phosphate exchange capacity of the resin was 3.33, 6.67, 13.15, 19.69, 30.13, 50.00, and 55.87 mg/g for 10, 20, 40, 60, 100, 200, and 300 mg/L, respectively. A study using Duolite A171 anion exchange resin reported a similar trend [39].

### 3.5 Effect of Temperature

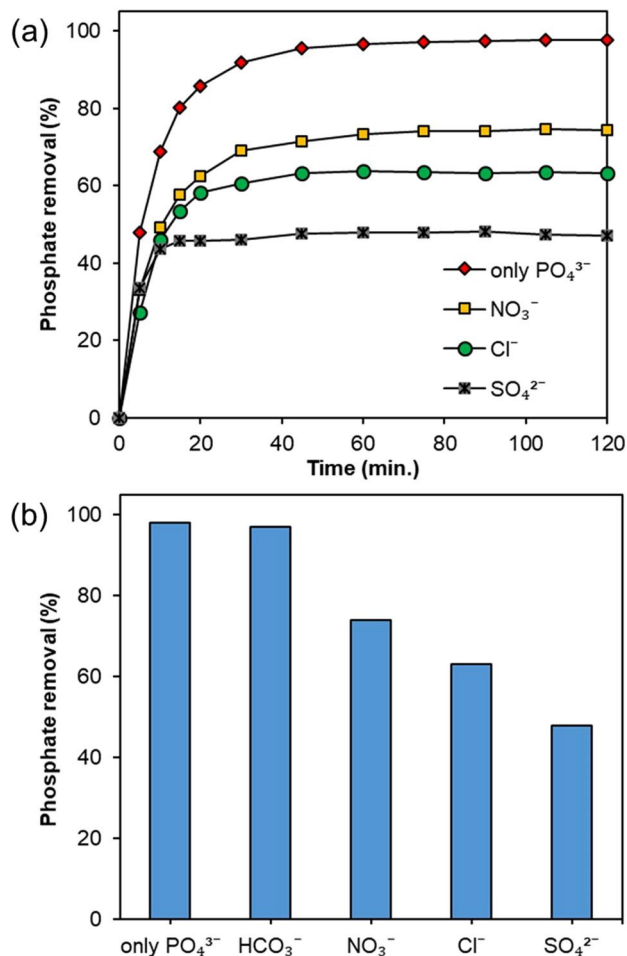
Resin pores may shrink at low temperatures and expand at high temperatures [37]. For this reason, the exchange capacity of ion exchange resins changes with temperature. The effect of temperature on the phosphate ion exchange capacity of Purolite A200E resin was studied at 10, 20, 30, and 40 °C. In experiments, resin dosage was 1.5 g/500 mL, initial pH 7, stirring speed 500 rpm, and initial phosphate concentration 60 mg/L. Experimental results are shown in Fig. 5. The increase in temperature was effective on phosphate removal efficiency and the necessary contact duration was observed to reduce. At the end of 20 min, phosphate removal was 76%, 86%, 91%, and 93% at 10, 20, 30, and 40 °C, respectively. At the end of 120 min, 90% phosphate removal was provided at 10 °C, while 98% phosphate removal was provided at 20, 30, and 40 °C. When removal rates are investigated, the increase in temperature increased phosphate removal rates. This situation may be linked to the increase in pore diffusion rate with the increase in temperature [40]. For this reason, high temperatures should be chosen to accelerate the phosphate exchange mechanism using Purolite A200E anion exchange resin.



**Fig. 5** Effect of temperature on phosphate removal (Resin dosage=1.5 g/500 mL, initial pH=7, stirring rate=500 rpm,  $C_0=60$  mg/L)

### 3.6 Effect of Competing Anions

Selectivity of ion exchange resin is an important factor affecting phosphate removal. In natural water or wastewater, anions such as sulfate, nitrate, and chloride are mostly found with phosphate and these anions are expected to compete with phosphate ions. For this reason, the effects of bicarbonate, sulfate, nitrate, and chloride ions on phosphate removal were researched. Each of the competing anions were separately added at 60 mg/L concentration and the effect of these anions on phosphate removal was investigated. The experimental results are given in Fig. 6. During the ion exchange process, the concentrations of other anions could not be measured, because the focal point of the experiments was the efficiency of phosphate removal. The phosphate amounts removed by Purolite A200E resin were reduced in the presence of competing anions. The anion which affected phosphate removal the most was sulfate. Addition of sulfate ions reduced phosphate removal at 120 min from 98 to 48%. Addition of chloride and nitrate ions lowered phosphate removal to 74% and 63%, respectively. Addition of bicarbonate had almost no effect on phosphate removal. A study using a commercial strong basic anion exchange resin (Varion ATM) for phosphorus removal investigated the effects of nitrate and chloride ions on phosphorus removal and reported that nitrate had higher effect on phosphorus removal efficiency compared to chloride ions [41]. A different study using a macro-porous strong basic anion exchange resin (Dowex Maraton MSA) found that increasing sulfate concentration reduced phosphate removal and lowered it to almost zero. With the aim of selectively removing phosphate, modification of the resin with nano-scale zirconium



**Fig. 6** Effect of other ions (Resin dosage=1.5 g/500 mL, initial pH=7, stirring rate=500 rpm,  $C_0=60$  mg/L, competing anion concentration=60 mg/L)

molybdate was reported to improve the removal efficiency in the presence of sulfate ions [10].

### 3.7 Ion-Exchange Kinetics

Ion exchange on resins is a mass transfer process between the liquid phase and solid phase. This phenomenon is characterized by equilibrium and kinetic rates that provide data to develop models for the process [42]. The PFO [43] and PSO [44] kinetic models are used to define the ion exchange capacity obtained under equilibrium and non-equilibrium conditions for phosphate exchange (adsorption) rate of the ion exchange resin. The PFO kinetic model is given by Eq. (4), while the PSO kinetic model is shown in Eq. (5):

$$Q_t = Q_e (1 - e^{-k_1 t}) \tag{4}$$

$$Q_t = (k_2 Q_e^2 t) / (1 + k_2 Q_e t) \tag{5}$$

where  $k_1$  is the rate constant for the PFO kinetic model ( $\text{min}^{-1}$ );  $k_2$  is the rate constant for the PSO kinetic model ( $\text{g/mg/min}$ ); and  $Q_t$  and  $Q_e$  represent the phosphate exchange capacity ( $\text{mg/g}$ ) at time  $t$  and at equilibrium, respectively.

Erroneous results and hence mistaken decisions are stated to be caused linked to the data used in the linear forms of these kinetic models [45]. In addition, the non-linear forms are stated to provide better results for fit to experimental data than linear forms in the literature [45–47]. As a result, maximum (at equilibrium) ion exchange capacity and rate constant values were obtained using the non-linear method and are given in Table 2. When Table 2 is investigated, it was concluded that the PFO kinetic model may be used to define the ion exchange kinetics. In nearly all experimental conditions, the PFO kinetic model had higher  $R^2$  values. However, suitable  $R^2$  values may not always be sufficient to define the appropriate kinetic model. Another important factor is the selection of an appropriate  $Q_e$  value. When the ion exchange capacities at equilibrium are investigated, the values predicted by the PFO kinetic model are almost the same as the experimental results. The  $Q_e$  values obtained with the PSO kinetic model are always higher than the values obtained from experimental results. For the effect of stirring speed on ion exchange capacity at equilibrium, especially, the data obtained with the PSO kinetic model is much higher than the experimental data. For this reason, the PFO kinetic model was used to fit the predicted values with the experimental values for phosphate removal with the A200E ion exchange resin. Stated differently, the PFO kinetic model may be said to better describe the ion exchange of phosphate with A200E resin.

### 3.8 Ion-Exchange Isotherms

Adsorption isotherms are used to show the interaction between adsorbate and adsorbent [48, 49]. The correlation between the initial phosphate concentration and equilibrium adsorption capacity was investigated using the adsorption isotherm models. To research the interaction between anion exchange resin and phosphate, results obtained from batch

experiments and adsorption equilibrium studies were performed. The common Langmuir and Freundlich isotherm models were used to interpret the adsorption isotherm.

The Langmuir isotherm model assumes that adsorption occurs on a homogeneous adsorbent surface and there is no interaction between adsorbate molecules. According to this model, there is single-layer adsorption only on the adsorbent [50–52]. In other words, the equilibrium distribution of phosphate between solid and liquid phases is in a single layer. Adsorption does not occur after this single layer has formed on the adsorbent. This type of single-layer adsorption process occurs on the adsorbent surface with limited numbers of similar sites. The non-linear form of the Langmuir isotherm model is expressed by the following equation:

$$Q_e = (Q_m K_L C_e) / (1 + K_L C_e) \quad (6)$$

where  $C_e$  is the liquid phase equilibrium concentration ( $\text{mg/L}$ ),  $Q_e$  is the solid phase equilibrium concentration ( $\text{mg/g}$ ),  $Q_m$  ( $\text{mg/g}$ ), and  $K_L$  ( $\text{L/mg}$ ) is the Langmuir constant related to the adsorption capacity and adsorption energy, respectively (affinity between adsorbent and adsorbate).

The Freundlich isotherm model is one of the oldest empirical equations used to describe equilibrium data and adsorption characteristics for a heterogeneous surface. The Freundlich isotherm model assumes adsorption occurs on a heterogeneous surface with sites which have different adsorption energy [50]. The non-linear form of the Freundlich isotherm model can be expressed with the following equation:

$$Q_e = K_F C_e^n \quad (7)$$

where  $C_e$  is the liquid phase equilibrium concentration ( $\text{mg/L}$ ),  $Q_e$  is the solid phase equilibrium concentration ( $\text{mg/g}$ ),  $K_F$  is the Freundlich constant, and  $n$  (dimensionless) is the Freundlich intensity parameter, which indicates the magnitude of the adsorption driving force or the surface heterogeneity. According to the Freundlich theory, the adsorption isotherm becomes linear when  $n = 1$ , favorable when  $n < 1$ , and unfavorable when  $n > 1$ .

**Table 2** Calculated parameters of PFO and PSO kinetic model

Initial conc (mg/L)	$Q_{e, exp}$ (mg/g)	PFO model			PSO model		
		$k_1$ ( $\text{min}^{-1}$ )	$Q_{e, cal}$ (mg/g)	$R^2$	$k_2$ (g/mg/min)	$Q_{e, cal}$ (mg/g)	$R^2$
10	3.33	0.1229	3.33	0.9999	0.0835	3.33	0.9599
20	6.67	0.1372	6.67	0.9906	0.0514	6.67	0.9367
40	13.15	0.1188	12.97	0.9983	0.0177	13.33	0.9813
60	19.69	0.1243	19.40	0.9968	0.0123	20.00	0.9856
100	30.13	0.1288	29.69	0.9971	0.0064	32.05	0.9946
200	50.00	0.1451	49.48	0.9941	0.0046	53.01	0.9967
300	55.87	0.1560	55.37	0.9937	0.0045	59.17	0.9938

**Table 3** Ion-exchange isotherm parameters of Langmuir and Freundlich

Models	Parameters	Values
Langmuir	$Q_m$ (mg/g)	53.045
	$K_L$	0.2678
	$R^2$	0.8215
Freundlich	$K_F$ ((mg/g)/(mg/L) <sup>n</sup> )	18.439
	$n$	0.2365
	$R^2$	0.9894

The Langmuir and Freundlich constants were calculated and results are shown in Table 3. When the fit of the equilibrium adsorption capacities calculated at equilibrium concentration are compared, the high determination coefficient ( $R^2$ ) indicates that the Freundlich isotherm model has better fit for phosphate adsorption compared to the Langmuir isotherm model. The results show that the phosphate exchange capacity of the ion exchange resin increases with the increase in initial phosphate concentration; however, phosphate removal efficiency reduces.

## 4 Conclusion

The effects of initial pH, stirring speed, resin dosage, initial phosphate concentration, temperature, and common ions were systematically researched for phosphate removal using Purolite A200E ion exchange resin and phosphate removal was successfully completed. Phosphate removal is mainly linked to pH and the highest phosphate removal occurred in the pH interval 7–9. Phosphate removal rates increased with the increase in stirring speed, temperature, and resin dosage and reduced with the increase in initial phosphate concentration and the presence of competing ions. Sulfate content, especially, significantly reduces the phosphate removal efficiency of the resin. The effect of competitive anions on phosphate removal was determined to follow the order  $\text{SO}_4^{2-} > \text{Cl}^- > \text{NO}_3^- > \text{HCO}_3^-$ . Later, non-linear forms of the kinetic models were used and it was revealed the PFO kinetic model better reflected phosphate removal. Finally, the Freundlich isotherm better described the equilibrium data for phosphate ions on Purolite A200E anion exchange resin. All these results reveal that the anion exchange resin may be used for phosphate removal.

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**Data Availability** The authors declare that the data supporting the findings of this study are available within the paper.

## Declarations

**Conflict of Interest** The author(s) declared no potential conflicts of interest with respect to the research, authorship, and/or publication of this article.

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