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Removal of tripolyphospate from water by ion exchange resins

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ABSTRACT

In this paper, two different ion-exchange resins namely strongly basic Purolite A420S and hybrid anion exchange resin infused with iron oxide, Purolite FerrIX A33E, were evaluated for the removal of tripolyphosphate (TPP) from an aqueous medium. Resins were compared in terms of their TPP removal capacity, kinetics, and regeneration efficiency under batch conditions. The results of batch experiments show that the maximum adsorption capacities were 256.91 mg-TPP/g-resin for A420S and 222.32 mg-TPP/g-resin for FerrIX A33E resin. The sorption kinetics of TPP onto A420S is well governed by the pseudo-first-order kinetic model, and pseudo-second-order model for FerrIX A33E resin. Sorption isotherm data were well described by the Langmuir model for the resins. The regeneration studies were also examined for such resins and obtained results showed that FerrIX A33E resin can be fully regenerated with 1 M NaCl and A420S resin can be regenerated with 4 M HCl with 95% efficiency. It was also found that, the percentage removal of TPP was maintained at more than 85% as compared to the initial value, even after five sorption–regeneration cycles. Thermodynamic studies showed that the sorption of TPP onto ion exchange resins is an exothermic process. The negative values of free energy change (ΔG^0) indicate the spontaneous nature of the sorption process.

Keywords: Anion exchange resin; Hybrid anion ion exchange resin; Purolite A420S; Purolite FerrIX A33E; Tripolyphosphate

1. Introduction

Phosphates and condensed phosphates have been used widely in industrial fields such as detergents, chemical fertilizers, food additives, biomaterials, electric and electronic materials, glass ceramics, flame retardants, etc. [1]. A large amount of used phosphate finally reaches water environment as diluted waste, which often leads to pollution of the water environment [2]. Excessive amounts of phosphorous entering into lakes and streams trigger the growth of undesirable algae and other aquatic weeds, which results in the eutrophication problem [3]. Eutrophication can be defined as the enrichment of water by nutrients and the consequent deterioration of quality due to the luxuriant growth of plant life, and its repercussions on the ecological balance of the waters affected [4]. The phosphorus usually occurs in wastewater and surface water in the form of inorganic (ortho and condensed) and organic phosphates [5]. In particular, tripolyphosphate ($P_3O_{10}^{5-}$) is important species of phosphate that mainly comes from used detergents and causes the growth of aquatic plants and cyanobacteria because TPP is slowly degraded to orthophosphate and has a long lifetime. Therefore, TPP removal is necessary from the wastewaters [6]. The amount of phosphate in typical raw domestic sewage is also highly variable, but the approximate concentrations of the various forms have been estimated as orthophosphate (5 mg P/L), tripolyphosphate (3 mg P/L), pyrophosphate (1 mg P/L), and organic phosphates (1 mg P/L) [7]. In industrial wastewater, its concentration reaches to as 28 mg P/L [8].

Various treatment technologies such as precipitation [6], adsorption [8–11], coagulation [12] and membrane process [13] have been applied for TPP removal from water. The efficiencies of such methods vary from 80% to 100%.

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In this study, we successfully removed TPP from an aqueous solution. The two different ion exchange resins namely Purolite A420S (strongly basic anion exchange resin) and, Purolite FerrIX A33E (hybrid anion ion exchange resin infused with iron oxide) compared for TPP removal. The batch experimental parameters such as resin dosage, initial pH of solution, temperature as well as sorption isotherms and kinetic studies were carried out for clarify the TPP removal mechanism.

2. Experimental

2.1. Materials

This research was performed using the commercially available ion exchange resins, which were obtained from Purolite Int. Co., Italy. The typical physical and chemical characteristics of these resins were summarized in Table 1.

Tripolyphospahe solutions of different concentrations were prepared from analytical grade $Na_5P_3O_{10}$ (Alfa Aesar). The solutions utilized for pH control were prepared from HCl (Merck, Germany) and NaOH (Merck, Germany). pH meter (Mettler Toledo, FG2) was calibrated by using pH 4.0 and 7.0 buffers (Merck, Germany).

The Eriochrome Black T (EBT), $MgCl_2.6H_2O$, NH_3, NH_4Cl, methanol (all from Merck) used for TPP analysis.

2.2. Analysis of TPP

The analysis of TPP was performed spectrophotometrically by adaptation of magnesium (Mg^{2+}) analysis [14]. EBT react with Mg^{2+} in NH_3/NH_4^+ buffer (pH 10.1) form deep reddish complex and the free form of EBT is blue in the same pH. The TPP forms stronger complex with Mg^{2+} then EBT. Thus, when TPP was added to Mg-EBT complex solution, the color of solution turned from reddish to blue. The Mg-EBT complex solution`s absorption wave length is 520 nm. When the TPP was added to solution, absorption value decreased and from such decrement, TPP concentration calculated.

In a general procedure; aliquot amount of TPP solution transferred to volumetric flask then 1 mL of EBT solution (0.1 w/v in methanol), 5 mL of NH_3/NH_4^+ solution (pH 10.1) and 5 mL of Mg^{2+} solution (5.0 mg-Mg^{2+}/L) added and filled to 25 mL with distilled water. This solution stayed in dark for 1 h then measured at 520 nm against blank solution only contains EBT and buffer solution (without TPP and Mg^{2+}). Fig. 1 shows the obtained calibration graph for TPP analysis.

2.3. Batch sorption experiments

In order to find out the effects of resin dose on TPP removal, the batch experiments were carried out in 25 mL square plastic bottles at the temperature of 25°C. For each run, 0–0.05 g resin was added into 25 mL solutions of 100 mg-TPP/L (pH: 6), and shaken for 24 h. Afterwards, the resin was separated from the solution by decantation and the residual concentrations of TPP in solution was determined. The effects of initial solution pH on TPP removal were studied within the range of pH 1.0–8.0 for A420S resin and between pH of 4.0 to 8.0 for FerrIX A33E. The optimum resin amount (0.03 g) was added into 25 mL of TPP solution of 100 mg/L and shaken in a shaker. After equilibrium was reached, resin was removed and TPP content in the solution was determined.

Sorption isotherms were carried out by contacting 0.03 g of resins with 25 mL of TPP solutions with concentrations varied from 100 to 1000 mg/L (pH = 6). The samples were collected after shaking for 24 h to allow attainment of equilibrium. Kinetic tests were performed by putting 1.2 gram of resin in contact with 1.0 L of the TPP solution (100

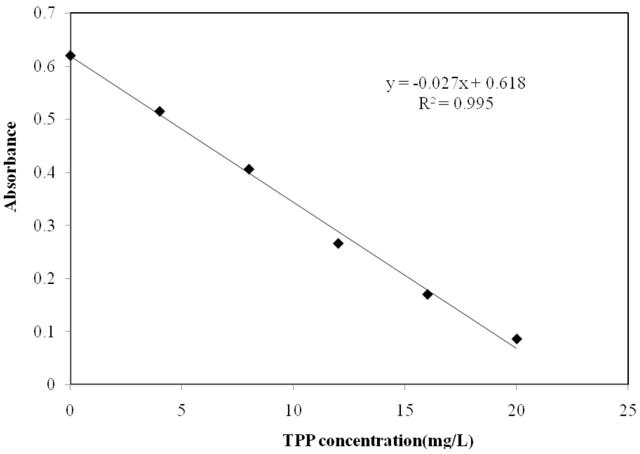

Fig. 1. Calibration graph for TPP analysis.

Table 1
Physical and chemical properties of ion-exchange resins

Properties	FerrIX A33E	A420S
Polymer structure	Polystyrene cross-linked with divinylbenzene	Gel polystyrene crosslinked with divinylbenzene
Appearance	Brown spherical beads	Spherical beads
Particle size range (μm)	300–1200	425–1200
Temperature Limit (°C)	80	60
pH limits, operating	4.5–8.5	0–14
Functional group	Hybrid anion ion exchange resin infused with iron oxide	Type I quaternary ammonium

mg/L, pH = 6). The solution was stirred with an overhead mixer. The TPP concentrations were monitored after taking samples at prescribed times. All experiments (except kinetic experiment) were replicated twice and mean of the obtained results were presented.

3. Results and discussion

3.1. Effect of resin dosage on TPP removal

The effect of resin dosage on the removal of TPP from aqueous solution is shown in Fig. 2. The percentage of TPP removal increases with the increasing sorbent dosage. This phenomenon could be explained by the increase in surface area and the available sorption sites with an increase in the sorbent dosage [15]. The removal of TPP reached maximum at dose 0.03 g for 25 mL of solution for such resins.

3.2. Effect of solution pH

The removal of TPP by ion exchange resins at different initial pH values is depicted in Fig. 3. This figure shows that the complete removal of TPP achieved in the pH range of 4–8. When the A420S resin was used and pH of solution decreased to pH = 1, removal of TPP was not possible. The possible causes are as follows:

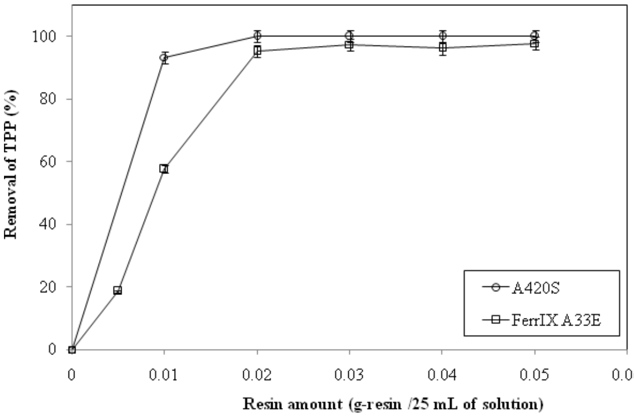

Fig. 2. Effect of resin amount on TPP removal.

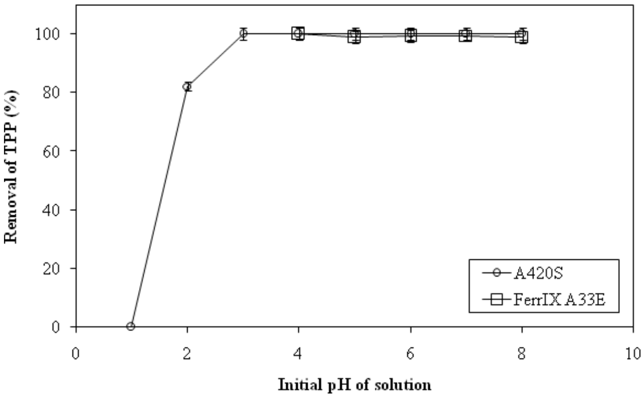

Fig. 3. Effect of solution pH on TPP removal.

i) *Nature of the exchanging ions:* Two aspects of the exchanging ions are important; the valence and the size of the hydrated ions. The diffusing ions are subject to coulombic attraction by the fixed ions of the resin. The greater the valence of the ion the greater is this retarding force [16]. The Triphosphoric acid ($H_5P_3O_{10}$) is a penta-acid and it's pKa values are $pK_a(1) \leq 0$; $pK_a(2) = 0.89$, $pK_a(3) = 4.09$; $pK_a(4) = 6.98$; $pK_a(5) = 9.93$ [17]. The dissociation of triphosphoric acid and equations for the calculation of TPP species are shown at following equations (Eqns. (1)–(10)).

$$H_5P_3O_{10} + H_2O \rightarrow H_3O^+ + H_4P_3O_{10}^- \tag{1}$$

$$H_4P_3O_{10}^- + H_2O \leftrightarrow H_3O^+ + H_3P_3O_{10}^{2-} \tag{2}$$

$$H_3P_3O_{10}^{2-} + H_2O \leftrightarrow H_3O^+ + H_2P_3O_{10}^{3-} \tag{3}$$

$$H_2P_3O_{10}^{3-} + H_2O \leftrightarrow H_3O^+ + HP_3O_{10}^{4-} \tag{4}$$

$$HP_3O_{10}^{4-} + H_2O \leftrightarrow H_3O^+ + P_3O_{10}^{5-} \tag{5}$$

$$\alpha_{H_4P_3O_{10}^-} = \frac{[H_3O^+]^4}{[H_3O^+]^4 + [H_3O^+]^3K_{a1} + +[H_3O^+]^2K_{a1}K_{a2} + [H_3O^+]K_{a1}K_{a2}K_{a3} + K_{a1}K_{a2}K_{a3}K_4} \tag{6}$$

$$\alpha_{H_3P_3O_{10}^{2-}} = \frac{[H_3O^+]^3K_{a1}}{[H_3O^+]^4 + [H_3O^+]^3K_{a1} + [H_3O^+]^2K_{a1}K_{a2} + [H_3O^+]K_{a1}K_{a2}K_{a3} + K_{a1}K_{a2}K_{a3}K_4} \tag{7}$$

$$\alpha_{H_3P_3O_{10}^{2-}} = \frac{[H_3O^+]^2K_{a1}K_{a2}}{[H_3O^+]^4 + [H_3O^+]^3K_{a1} + [H_3O^+]^2K_{a1}K_{a2} + [H_3O^+]K_{a1}K_{a2}K_{a3} + K_{a1}K_{a2}K_{a3}K_4} \tag{8}$$

$$\alpha_{HP_3O_{10}^{4-}} = \frac{[H_3O^+]K_{a1}K_{a2}K_{a3}}{[H_3O^+]^4 + [H_3O^+]^3K_{a1} + [H_3O^+]^2K_{a1}K_{a2} + [H_3O^+]K_{a1}K_{a2}K_{a3} + K_{a1}K_{a2}K_{a3}K_4} \tag{9}$$

$$\alpha_{P_3O_{10}^{5-}} = \frac{K_{a1}K_{a2}K_{a3}K_{a4}}{[H_3O^+]^4 + [H_3O^+]^3K_{a1} + [H_3O^+]^2K_{a1}K_{a2} + [H_3O^+]K_{a1}K_{a2}K_{a3} + K_{a1}K_{a2}K_{a3}K_{a4}} \tag{10}$$

The species of triphosphoric acid versus pH is shown in Fig. 4. As can be seen from Fig. 4 that, at pH 1; $H_4P_3O_{10}^-$ and $H_3P_3O_{10}^{2-}$ are the dominant species of TPP in the solution and Cl^- ions present in the solution (because of adding HCl for pH adjustment). Although $H_3P_3O_{10}^{2-}$ has higher valance

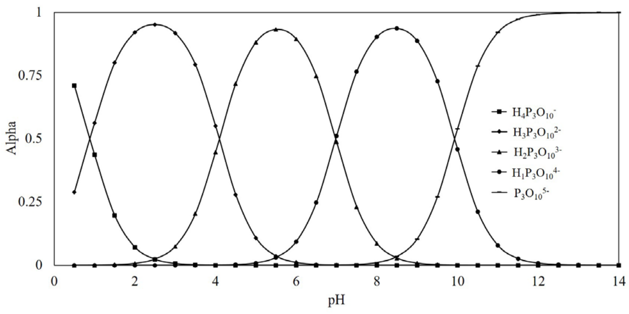

Fig. 4. Alpha values of TPP versus pH.

then Cl^- ions; it's hydrated radius larger than Cl^- ion thus removal of TPP affected by Cl^- ion.

ii) *Concentration of solution (or ion):* If the concentration is increased in a series of experiments where all the other conditions are kept unchanged, the rate of exchange increases linearly with concentration [16]. At pH = 1 and 2; chloride concentration is higher than TPP species thus removal of TPP may be affected by the Cl^- ions. On the other hand, when the pH of solution reached 4 and higher, chloride concentration in the solution decreased (because of less added amount of HCl for pH adjustment) and valance of TPP increased ($H_3P_3O_{10}^{2-}$ and $H_2P_3O_{10}^{3-}$ are the dominant species) thus removal of TPP increased.

Accordingly, in the hybrid sorbent two main mechanisms could be expected for the removal of TPP: (a) anion exchange with the quaternary ammonium; and (b) anion exchange or sorption with the Fe–OH groups of the iron oxide particle.

i) Anion exchange with the quaternary ammonium: The exchange of TPP with quaternary ammonium group is represented in Eq. (1) (at pH 4; $H_3P_3O_{10}^{2-}$ is an example for ion exchange reaction represented in Eq. (11))

$$\overline{2R-NH_4^+Cl} + H_3P_3O_{10}^{2-} \leftrightarrow \overline{(R-NH_4^+)_2H_3P_3O_{10}^{2-}} + 2Cl^- \qquad (11)$$

ii) *Anion exchange or sorption:* If the pH of solution is lower than point of zero charge pH (pH_{pzc}), surface of iron oxide has a positive charge and this positively charged surface catch the negatively charged TPP species. However, if the pH of solution is above the pH_{pzc}, the surface of iron oxide has a net negative charge and will electrostatically repel anionic TPP from the surface of resin. The mechanism and chemistry of surface already explained elsewhere [18–20]. The resin data sheet also recommends the working pH at the range of 4.5–8.5 so we did not try other pH range.

3.3 Kinetic of TPP removal

Kinetic tests were performed by contacting 1.2 g resin with 1.0 L of 100 mg-TPP/L (pH = 6) solution at 25°C. Solution was stirred with overhead mixer. TPP concentrations were monitored by obtaining the samples at prescribed times intervals, and such samples were analyzed and collected results are depicted in Fig. 5. It is clear from Fig. 5 that kinetic of FerrIX A33E is faster than A420S resin. This can be because of two removal mechanism for TPP removal (ion exchange and sorption by iron particles) in case of hybrid resin used. The experimental results were evaluated by using pseudo first order and second order reaction model. The linear form of pseudo first order rate equation was given by the following Eq.(12) [21].

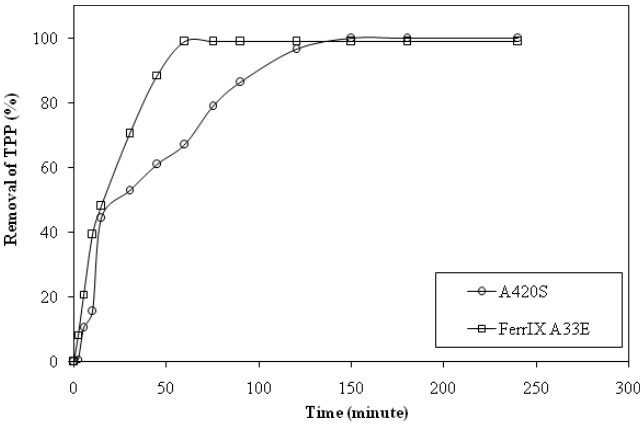

Fig. 5. Removal of TPP versus time for such resins.

Table 2
Calculated parameters of kinetic models

Kinetic model	Parameter	A420S	FerrIX A33E
Pseudo-first order	k_1 (min^{-1})	0.03	0.06
	q_e (mg/g)	79.18	92.21
	R^2	0.95	0.98
Pseudo-second order	k_2 (g/mg min)	1.37×10^{-4}	5.04×10^{-4}
	q_e (mg/g)	128.20	108.18
	h (mg/gmin)	2.25	5.90
	R^2	0.85	0.99

$$\log(q_e - q_t) = \log(q_e) - \frac{k_1 t}{2.303} \qquad (12)$$

Sorption rate constant k_1 (min^{-1}) can be calculated from the plot of log ($q_e - q_t$) versus time. If the rate of sorption is a second order mechanism, the pseudo-second order chemisorption kinetic rate equation is expressed as Eq. (13) [22].

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{1}{q_e} t \qquad (13)$$

If pseudo-second order kinetics is applicable, the plot of t/q_t against t of Eq. (13) should give a linear relationship, from which q_e and k_2 (g/mg-min) can be determined from the slope and intercept of the plot. Constant k_2 is used to calculate the initial sorption rate r; at $t \cdot 0$, as shown in Eq. (14) [23].

$$h = k_2 q_e^{\,2} \qquad (14)$$

The calculated parameters of kinetic models were summarized in Table 2. Removal of TPP by such resins showed good compatibility with pseudo-first order kinetic model for A420S resin and with pseudo second order kinetic model for FerrIX A33E resin.

The infinite solution volume models (ISV) and the unreacted core models (UCM) were also examined for the sorption mechanism. The explanations about such models can be found in [24,25]. The obtained results are summarized in Table 3.

According to the ISV model, film diffusion is the rate-controlling step for such resins and UCM model suggest that rate-controlling step is chemical reaction.

3.4. Sorption isotherm

In order to quantify the sorption capacity of such resins for the removal of TPP and to get an insight into the sorption mechanism; Langmuir and Freundlich equations [Eqs. (15) and (16)] were adopted to describe the TPP sorption on such ion exchange resins.

The Langmuir equation is valid for monolayer sorption onto a surface is given as follows in Eq. (15) [26]

$$\frac{C_e}{Q_e} = \frac{1}{bQ_0} + \frac{C_e}{Q_0} \tag{15}$$

C_e is equilibrium concentration (mg/L), Q_e is the amount adsorbed under equilibrium (mg/g), Q_0 is the theoretical maximum adsorption capacity, and b (L/mg) is a Langmuir constant, which indicates the affinity of the TPP toward the ion exchange resin. The values of Langmuir parameters Q and b were calculated from the slope and intercept of the linear plots of C_e/Q_e versus C_e, and the calculated parameter values and R^2 are presented in Table 4.

The Freundlich equation, indicative of surface heterogeneity of the sorbent, is given in Eq. (16) as follows [26,27]:

$$\log Q_e = \log K_F + \frac{1}{n}\log C_e \tag{16}$$

1/n and K_F are Freundlich constants, related to adsorption capacity and adsorption intensity (heterogeneity factor), respectively, Q_e is the amount of TPP sorbed per unit weight of the ion exchange resin (mg-TPP/g-resin), and C_e is the equilibrium concentration of TPP in solution (mg/L). The values of K_F and $1/n$ were obtained from the slope and intercept of the linear Freundlich plot of log Q_e vs. log C_e.

The R^2 values in Table 4 suggested that the Langmuir isotherm provides a good model of the sorption system. The sorption capacity, Q_0, found 256.91 mg-TPP/g for A420S resin and 222.32 mg-TPP/for FerrIX A33E resin.

3.5. Thermodynamic study

The effect of the temperature on the removal of TPP by ion-exchange resins was investigated by varying the temperature in the range of 30–50°C. The experiments were carried out with 0.03 g of resins contacting with 25 mL of TPP (100 mg-TPP/L, pH 6). Thermodynamic parameters, including Gibbs free energy change (ΔG°), enthalpy change (ΔH°), and entropy change (ΔS°) were used to decide whether the ion-exchange process is spontaneous or not. ΔG° were calculated from the following equations [28,29].

$$\Delta G = -RT\ln K_C \tag{17}$$

$$\ln K_C = \frac{\Delta S^0}{R} - \frac{\Delta H^0}{RT} \tag{18}$$

$$\Delta G^\circ = \Delta H^\circ - T\Delta S^\circ \tag{19}$$

In these equations, K_c is the equilibrium constant was calculated by Q_e/C_e, R is the universal gas constant (8.314 J/mol K), and T is the temperature (K). Parameters change in enthalpy (ΔH^0) and change in entropy (ΔS^0) are calculated from the slope and the intercept of the linear plot of ln K_c vs. 1/T. The calculated values are summarized in Table 5.

Table 3
Linear regression analysis data related to diffusion models on sorption kinetics of TPP

Method	Equation	*k*	Rate-controlling step	R^2 value	
				A420S	FerrIX A33E
ISV	$-\ln(1-X) = k_{li}t$,	$k = 3DC/r_o\delta\, Cr$	Film diffusion	0.99	0.97
	$-\ln(1-X^2) = kt$,	$k = Dr\varpi^2/r_o^{\,2}$	Particle diffusion	0.98	0.95
UCM	X	$k = 3C_{Ao}K_{MA}/ar_oC_{So}$	Liquid film	0.83	0.88
	$3-3(1-X)^{2/3}-2X = kt$	$k = 6D_{eR}C_{Ao}/\ ar_o^{\,2}C_{So}$	Reacted layer	0.95	0.97
	$1-(1-X)^{1/3} = kt$	$k = K_sC_{Ao}/\ r_o$	Chemical reaction	0.96	0.98

Table 4
Isotherm constants for TPP sorption on the ion exchange resin

Resin	Langmuir isotherm constants			Freundlich isotherm constants		
	Q_0 (mg·g^{-1})	b (L/mg)	R^2	K_F (mg/g)	n	R^2
Purolite A420S	256.91	0.23	0.99	162.22	13.50	0.92
Purolite FerrIX A33E	222.32	0.18	0.99	203.92	90.33	0.78

Table 5
Thermodynamic parameters for TPP removal

	ΔS^0 (KJ/(mol K))	ΔH^0 (kJ/mol)	ΔG^0 (kJ/mol)
Purolite A420S	–0.1	–51.5	–17.9 (303 K)
			–16.8 (313 K)
			–15.7 (323 K)
Purolite FerrIX A33E	–0.16	–66.8	–17.6 (303 K)
			–16.0 (313 K)
			–14.3 (323 K)

The negative value of heat of adsorption (ΔH^0) suggests that the sorption of TPP onto ion exchange resins is an exothermic process. The negative values of Gibbs free energy change (ΔG^0) indicate the spontaneous nature of the ion-exchange reaction. The ΔS^0 value is negative for such resins, in this case, we can say that removal of TPP by ion-exchange resins is spontaneous at low temperature but it becomes nonspontaneous at high temperature [30].

3.6. Regeneration of ion exchange resins

2.0 g of resins were contacted 450 ml of TPP solution (1000 mg-TPP/L, pH 6) for 24 h. Later, the resins were separated from solution by decantation and washed with deionized water then dried in oven at 40°C. The TPP amount in feed and effluent was analyzed and loaded amount of TPP on ion exchange resins were calculated. A 0.1 g of dry resin was contacted with 25 mL regeneration solutions, and then the mixture was shaken at room temperature for 24 h and after this period TPP concentration in the solution was determined. Regeneration efficiency (RE, %) was calculated using Eq. (20) and obtained results are summarized in Table 6.

$$\text{RE}(\%) = \frac{\text{Amount of eluated TPP}}{\text{Amount of loadedTPP}} \times 100 \qquad (20)$$

As can be seen from Table 6, FerrIX A33E resin can be completely regenerated by 1 M NaCl, however, in case of A420Sresin, 4 M HCl is required for high regeneration efficiency. Because of iron impregnation, FerrIX A33E resin cannot be treated with HCl, otherwise, iron will be dissolved. Caustic treatment is also not recommended because it will change the intrinsic pH of the product depressing its sorption properties that is why only NaCl was used for FerrIX A33E resin regeneration. An additional goal was to monitor the recovery of ion-exchange capacity for TPP loading in subsequent exhaustion – regeneration cycles. The results depicted in Fig. 6 demonstrate that in each run the TPP capacity in the exhaustion mode was found to be affected and removal of TPP decreased to 85% for A420S resin and 90% for FerrIX A33E resin after 5th cycle.

Table 6
Percentage recovery of TPP by different regeneration solution

Regeneration solution	Regeneration efficiency
1 M HCl (for A420S)	64
2 M HCl (for A420S)	78
4 M HCl (for A420S)	95
1 M NaCl (for FerrIX A33E)	100
2 M NaCl (for FerrIX A33E)	100

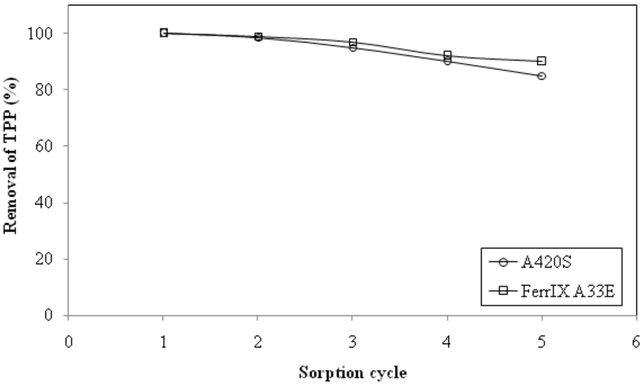

Fig. 6. Recycle use of resins for TPP removal.

4. Conclusion

The removal of TPP by strongly basic anion exchange resin and iron oxide infused hybrid anion exchange resin was studied in a batch system with respect to the resin dosage, initial solution pH, and temperature. When the A420S resin was used, initial solution pH affected the removal performance of resin. At low pH values, removal of TPP was low but increasing the solution pH to 3 or higher, increased the removal of TPP up to 100%. Because of pH limitation for FerrIX A33E resin, only 4–8 pH range was studied and in such pH range, complete removal of TPP was achieved.

The Langmuir sorption model was used to represent the experimental data and equilibrium data fitted very well to the Langmuir isotherm model. The maximum uptake capacity of A420S resin for TPP (256.91 mg-TPP/g-resin) was slightly higher (222.32 mg-TPP/g-resin) than the FerrIX A33E resin. The kinetic performance of FerrIX A33E resin is better than A420S resin. In 60 min, complete removal of TPP obtained however, 90 min required for A420S resin.

The negative value of ΔG° showed that the removal of TPP by such resins is feasible and negative value of ΔH° confirmed the exothermic nature of ion-exchange process.

The regeneration performance of resins is different from each other. The FerrIX A33E resin provided better regeneration performance than the A420S resin. 1 M NaCl completely regenerated the FerrIX A33E resin but 95% of sorbed TPP recovered from A420S resin by 4M HCl.

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