# Removal of iron from wet-process phosphoric acid using titanium silicate-polymer composite

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## ABSTRACT

Removal of iron from wet process phosphoric acid solution was studied using synthesized poly (acrylamide - acrylic acid)- titanium silicate (P (AM-AA)-TS) composite adsorbent. The polymer composite was prepared through gamma radiation-induced template polymerization of acrylic acid (AA) on polyacrylamide (PAM) as template polymer in presence of titanium silicate and crosslinker. The prepared composite was characterized via FTIR and SEM. P(AM-AA)-TS was studied for iron removal from phosphoric acid solution, different parameters affecting the adsorption reaction were investigated (pH, contact time, metal ion concentration, adsorbent dose and temperature). Removal percentage reached more than 70% using P(AM-AA)-TS adsorbent. The adsorption isotherms were studied to evaluate the maximum sorption capacity of adsorbent. The isotherm studies revealed that the experimental results could fit well with Langmuir model, with adsorption capacity of 2 mg/g. P(AM-AA)-TS adsorbent was applied for removal of iron from crude wet process phosphoric acid through batch, and 46% iron removal was achieved.    © 2015 Trade Science Inc. - INDIA

## KEYWORDS

Removal;
Iron;
Phosphoric acid;
Titanium silicate;
Polymer composite.

## INTRODUCTION

Phosphoric acid is one of the highly applicable acids and the second highly produced acid after sulfuric acid. It is used in detergent production as raw material, food products, and alimentary supplies for cattle, toothpastes and fertilizers[1-3]. There are two known procedures for production of commercial phosphoric acid $H_3PO_4$; thermal and wet processes. In thermal process, $H_3PO_4$ is firstly produced by reduction of phosphate rock, followed by oxidation and hydration[4], while in wet process; the phosphate rocks are treated with $H_2SO_4$ or other mineral acid. Consequently, phospho-

ric acid produced by wet process, contains many undesirable impurities as fluoride, iron, copper and other metal ions originally present in the phosphate rocks which affect the grade of wet process phosphoric acid for applications in different fields. Some of these impurities are Fe(III) and U(VI). The presence of these impurities affects the quality of the product. Consequently, the majority of phosphoric acid produced by wet process (about 95%) is used as fertilizers and excluded from the use in non-fertilizer applications[5]. The usage of phosphoric acid in some industrial and food grade phosphate derivatives (e.g. food beverage, toothpaste and cleaning markets) requires application of high puri-

fication technique which enables the removal of the above mentioned impurities[6].

Different procedures were evaluated for phosphoric acid purification as; precipitation, ion-exchange, liquid–liquid extraction, adsorption on activated carbon, membrane technologies (electrodialysis (ED), reverse osmosis and nanofiltration)[7-15]. The application of these techniques were limited due to some disadvantages such as: limited efficiency, high costs of organic solvents, difficulty in recovering all the solvent from the purified acid and increasing environmental pollution through introduction of some by-products[16]. Removal of heavy metals from phosphoric acid was studied by precipitation as sulfides[17]. Other cationic impurities were removed from wet process phosphoric acid by precipitation as; Fe, Al, Mg, and Ca. Another way for removing these cations is the precipitation through increasing the pH of phosphoric acid. The content of $Fe_2O_3$ in fertilizer grade phosphoric acid (50% $P_2O_5$) should be less than <1.5% $Fe_2O_3$[18]. High iron content decreases the solubility of fertilizer $P_2O_5$ in water[19]. The Egyptian phosphate concentrates when leached with $H_2SO_4$ or recycled $H_3PO_4$, the produced phosphoric acid (50% $P_2O_5$) contains 4 or 2.7% $Fe_2O_3$, respectively. Decreasing iron content in wet phosphoric acid was studied using different precipitating reagents as sodium silicate, silicon dioxide, potassium sulfate, (silicon dioxide + sodium carbonate + potassium sulfate) [0.8885:1.5672:4.0000], calcium sulfate and potassium amyl xanthate (0.01–6.00 g/100 ml $H_3PO_4$)[19].

Organometallic materials have recently attracted much interest to provide new advances in iron ions capturing since they can provide high surface area activity as well as reasonable chemical stability in the acidic media. In line with this trend, a new composite material based on titanium silicate and acrylamide co-polymer is introduced through this study. Titanium silicate is a highly insoluble thermally stable and suitable for several applications. The base of unit of a silicate mineral is the [SiO4]$^{4-}$ tetrahedron. In the vast majority of cases, silicon is in four-fold or tetrahedral coordination with oxygen. These silica tetrahedra are then polymerized to some degree to create various structures, such as one-dimensional chains, two-dimensional sheets, and three-dimensional frameworks. The basic silicate mineral

where no polymerization of the tetrahedra has occurred requires other elements to balance out the base 4-charge. These characteristics in turn reflects their high capability for adsorption and ion exchange properties[20,21].

'The present work deals with preparation and characterization of poly (acrylamide-acrylic acid)- titanium silicate composite adsorbent (P(AM-AA)-TS). The prepared composite adsorbent studied for the removal of iron from wet phosphoric acid produced by leaching of phosphate ore with nitric acid in previous work[22].

## EXPERIMENTAL

### Materials

Wet phosphoric acid produced through leaching of Sebaiya phosphate ore with nitric acid solution (in previous study) was used which has the constituents mentioned in TABLE 1.

**TABLE 1: Constituents of wet process phosphoric acid produced from nitric acid leaching of phosphate ore**

| | Phosphoric Acid | |
|---|---|---|
| | Concentrated | Recycled |
| **Constituent, %** | | |
| $P_2O_5$ | 42.0 | 20.0 |
| CaO | 0.10 | 0.25 |
| $Fe_2O_3$ | 0.45 | 0.25 |
| F | 0.19 | 0.10 |
| Cl | 0.20 | 0.14 |
| $SO_4^{2-}$ | 0.21 | 0.18 |
| $SiO_2$ | 0.05 | 0.02 |
| **Constituent, mg** | | |
| Mn | 6.7 | 2.4 |
| Cd | < 0.3 | < 0.3 |
| As | 3.3 | 2.0 |
| Pb | < 1.0 | < 1.0 |
| Zn | 4.1 | 1.2 |
| U | 4.0 | < 1 |

All chemicals and reagents were of A.R. grade and used without further purification. Fe and other constituents of $H_3PO_4$ were determined using atomic absorption spectroscopy (AAS). The fluoride content was determined by ion selective electrode, a Genway model 3330 pH-meter supplied with Orion model 94-09 BN

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fluoride electrode. $P_2O_5$ was determined spectrometrically using spectrophotometer.

## Preparation of titanium silicate polymer composite:

Titanium silicate was prepared by reaction of sodium silicate with titanium chloride with equivalent stoichiometry with vigorous stirring. The pH of the mixture was adjusted at 7.5 - 7.9 using dil HCl and NaOH. After filtration, the obtained gel was washed with deionized water and dried at 70 °C for 24 h.

Poly(acrylamide-acrylic acid)-titanium silicate (P(AM AA)TS) was prepared by gamma radiation induced template polymerization of acrylic acid (AA) on polyacrylamide P(AM) as a template polymer in the presence of the prepared titanium silicate and methylene bisacrylamide (MBA) as crosslinker[24]. The content of titanium silicate in final composite was adjusted to be 50% weight percentage.

## Adsorption of iron (Fe (III)) from phosphoric acid solution:

The removal of iron from wet phosphoric acid solution was studied as a function of different parameters (pH, time, sorbent concentration and iron concentration). A known volume of acid solution containing Fe (III) was mixed with the appropriate amount of solid composite adsorbent and left with shaking to appropriate time. After shaking, the acid solution was separated from the solid adsorbent and the concentration of iron was measured using atomic absorption spectroscopy (AAS). The removal percentage (R%) was calculated as:

$$R \% = \frac{C_o - C_e}{C_o} \times 100 \qquad (1)$$

where $C_o$ and $C_e$ are the initial and equilibrium iron concentration in solution (mg/L), respectively.
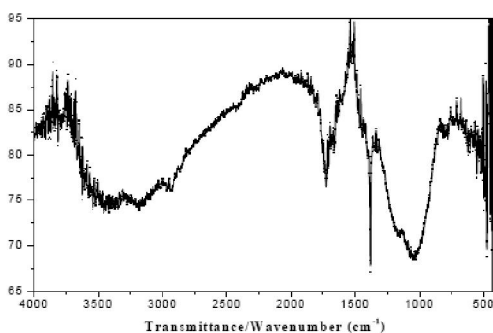
The distribution coefficient ($K_d$) were calculated using the equation:

$$K_d = \frac{(C_o - C_e)V}{C_e \, m} \quad (mL/g) \qquad (2)$$

where V is the volume of the solution (mL) and m is the mass of adsorbent (g).

## Sorption of iron from crude phosphoric acid

The composite sorbent was applied for removal of iron from crude wet process phosphoric acid (produced from through leaching of Sebaiya phosphate ore with nitric acid in previous work) in batch experiment. In this experiment appropriate amount of adsorbent was mixed with appropriate volume of crude phosphoric acid for 24 hour at room temperature. The concentration of iron was measured before and after experiment using atomic absorption spectroscopy.

# RESULTS AND DISCUSSIONS

## Characterization of the prepared composite adsorbent

### Fourier Transform Infrared (FTIR)

The FT-IR spectra of polymeric composite before and after iron adsorption are presented in Figures (1a and 1b). The spectrum of P(AM-AA)-TS showed peaks at 2900, 1750, 1500 and 1050 cm-1, while P(AM-AA)-TS-Fe has peaks at 3000, 1750, 1500, 1300 and 1030 cm-1. Both the spectra exhibit bands for the carboxylic C=O, carboxylic –OH and Si-O-Si at 1750, about 3000 and 1030 - 1050 cm-1, respectively.

### Morphological characterization
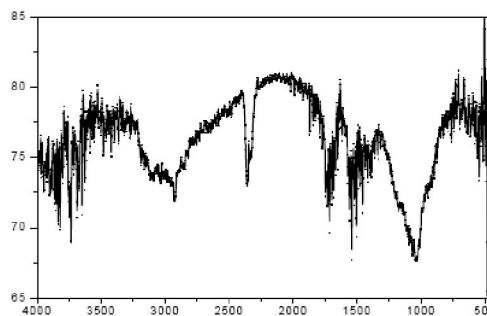
Scanning electron micrograph of P(AM-AA)-TS



**Figure 1a : FTIR spectrum of P(AM-AA)-TS; Figure 1b : FTIR spectrum of P(AM-AA)-TS-Fe.**
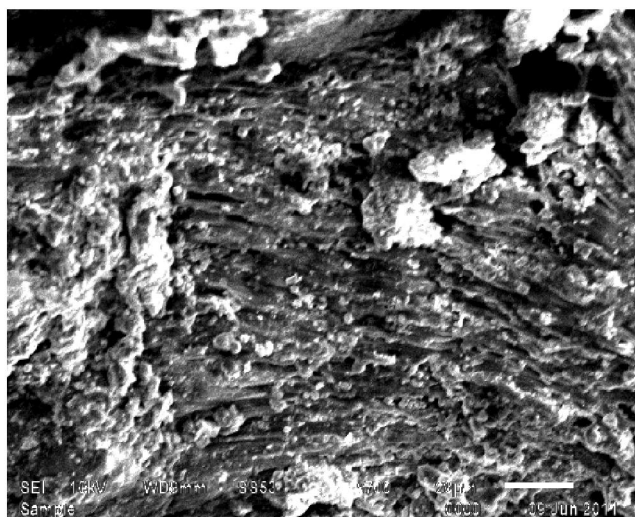
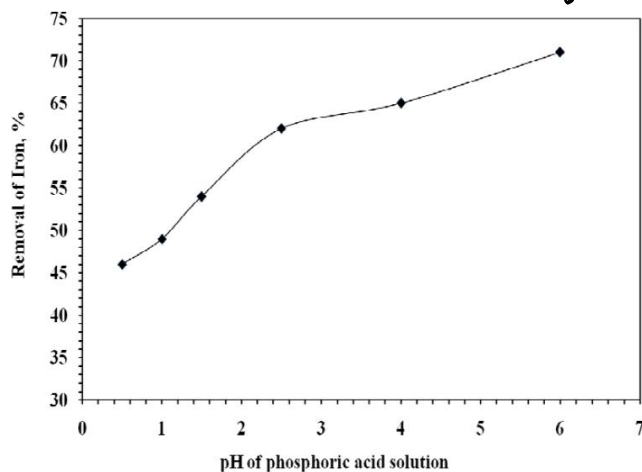**Figure 2 : SEM for P(AM-AA)-TS composite adsorbent**



**Figure 3 : Adsorption of Fe(III) from phosphoric acid solutions of different pH using P(AM-AA)-TS; adsorbent dose 50 g/L, iron concentration 50 mg/L, time 24 h.**

composite shown in Figure 2, confirms the formation of the composite material. The image in the figure shows the surface

Characteristics and the surface morphology of the composite material, in which the polymer layers and TS surface are attached together generating the porous composite adsorbent. This structure was formed in the course of polymerization process, as a result of polymer growth on the surface of TS particles. The composite structure could be defined depending on the nature and properties of inorganic material. It is generally proved that inorganic materials exhibit essential stability owing to their structures and polymerization growth efficiency. The stability and other physicochemical properties of the produced composite enhance the specific applications.

**Sorption of Fe(III) from phosphoric solution:**

**Effect of Ph**

The speciation of metal ions depends strongly on the pH, which affect the adsorption reaction. Within acid to neutral pH conditions, Fe(III) may form a number of soluble species including Fe(III), $Fe(OH)^{2+}$ and $Fe(OH)_2^+$. Dimerized species (eg. $Fe_2(OH)_2^{+4}$) could be formed depending on solution concentration. The removal of Fe(III) from phosphoric acid solution of variable pH was studied using poly(acrylamide-acrylic acid)-titanium silicate composite adsorbent (P(AM-AA)-TS) and the obtained results are represented in Figure 3.

The results showed a dependence of Fe(III) adsorption on pH. At low pH, the protons compete with Fe(III) and the predominant species in solution is $Fe^{3+}$. As the pH increased (0.5 - 2.5) the adsorption sites go deprotonated and the adsorption increased (the residual Fe(III) concentration decreased). At pH higher than 2.5, soluble hydrolyzed species ($Fe(OH)^{2+}$ and $Fe(OH)_2^+$) go formed which inhibit the adsorption of Fe(III) onto the adsorbent. Higher pH causes complete hydrolysis and precipitation which renders the residual Fe(III) concentration in solution highly decreased (due to precipitation not because adsorption)[25,26].

**Effect of contact time**

The contact time for complete sorption is important to give insight knowledge about the kinetics of the sorption process. Also, it gives information on the minimum time required for considerable adsorption and the possible diffusion control mechanism. The required time for equilibrium could give information about the movement of Fe(III) from the bulk solution towards the adsorbent surface. The adsorption of Fe(III) from wet process phosphoric acid onto P(AM-AA)-TS was studied at different time periods keeping the other parameters constant (ion concentration, adsorbent concentration and pH).

The results in Figure 4 show an initial rapid raise of iron removal, due to the availability of adsorbent active groups. Generally, the adsorption kinetics depends on: (i) the adsorbent surface area, (ii) the nature and concentration of the surface active groups present on the adsorbent which could react with Fe(III) ions. The re-
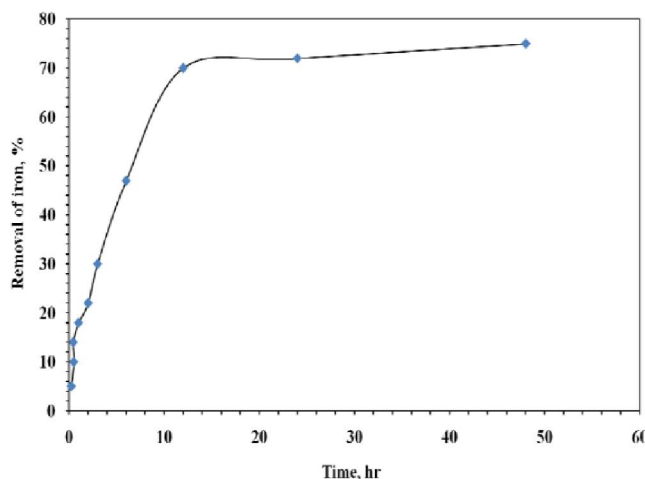
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**Figure 4 : Effect of contact time on the adsorption of Fe(III) from wet process phosphoric acid onto P(AM-AA)-TS composite; adsorbent dose 50 g/L, iron concentration 50 mg/L, pH 2.5.**

moval of Fe(III) reaches maximum after 24 h, after that time, no significant increase in the removal of Fe(III) from phosphoric acid solution. This is due to the decrease of the number of active sites.

## Effect of adsorbent doses

Different weights of the polymer- titanium silicate composite adsorbnet, were used for iron removal at 25 °C within dose range of 5 - 50 g/L while the other parameters were kept constant. Wet process phosphoric acid with 50 ppm Fe(III) was used to optimize the required amount of adsorbent under prescribed conditions for maximum uptake. The results in Figure 5 show that the adsorption of Fe(III) increases with increasing the adsorbent dose due to the high dose of adsorbent in the solution, the greater availability of exchangeable sites for the ions, i.e. more active sites are available for binding of Fe(III) ions.

A significant increase in the iron removal was observed within the dose range (5 - 40 g/L). It is clear in the figure that the maximum removal for Fe(III) of 70 % is observed with the dose of 40 g/L. Further increase in adsorbent dose produce insignificant increase in iron removal. This finding could be explained as a result of cluster formation by adsorbent particles resulting in decrease of surface area and surface active sites on the adsorbent[27,28].

## Effect of initial metal ion concentration

Batch experiments are conducted to study the effect of varying initial Fe(III) concentration on the ad-
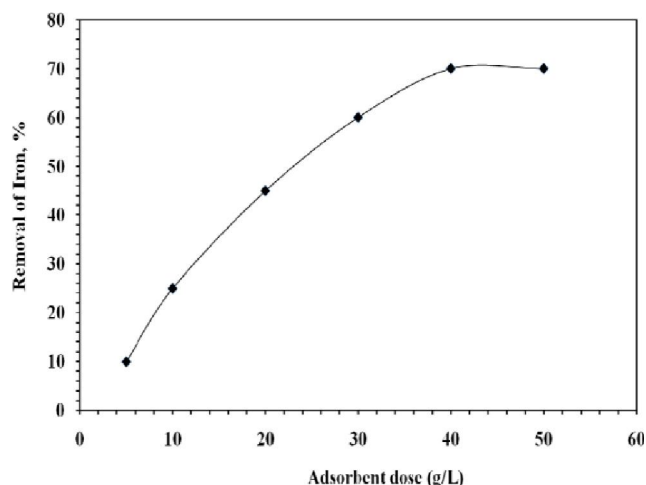


**Figure 5 : Effect of adsorbent dose (g/L) on the adsorption of Fe(III) from phosphoric acid solution onto P(AM-AA)-TS; pH 2.5, time 24 h, [Fe(III)] 50 mg/L).**

sorption (removal % and distribution coefficient) of Fe(III) from wet phosphoric acid by (P(AM-AA)-TS) adsorbent. The experiment was performed using 40 g/L adsorbent with different Fe(III) concentrations within range 20- 200 mg/L. The concentration of Fe(III) is

Represented against both the removal percentage and the distribution coefficient in Figure 6. It could be obviously deduced that at the same adsorbent dose, there is a decrease in removal% and increase in distribution coefficient with increasing initial Fe(III) concentration. This finding could be explained as at lower initial metal concentration, sufficient adsorption sites were available for the sorption of metal ions. However, at higher concentration, the adsorption sites were rela-
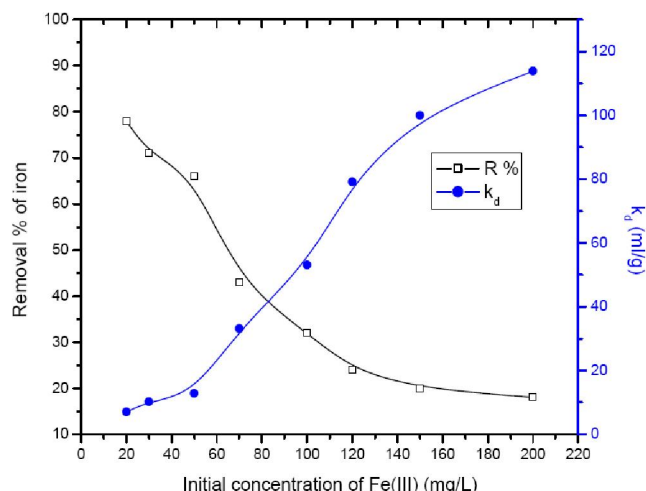


**Figure 6 : Effect of initial metal ion concentration on the adsorption of Fe(III) from phosphoric acid solution onto P(AM-AA)-TS; adsorbent dose 40 g/L, pH 2.5, time 24 h).**

tively low. The results indicate that energetically less favorable sites became involved

with increasing metal ion concentration and attributable to competition which cause blocking to available adsorption sites on adsorbent[29].

## Effect of temperature

The removal of iron from wet phosphoric acid solution was studied at different reaction temperature (25 - 50 °C). The experiment was performed at reaction time 24h, adsorbent dose 40 g/L, iron concentration 50 mg/L and pH 2.5. The results in Figure 7 indicate that the removal % of iron was affected by the increase in temperature from 25 to 50 °C where, the removal of iron decreases from about 69 to 50%. This behavior indicates that the iron adsorption from wet phosphoric acid solution by P(AM-AA)-TS is an exothermic process. The room temperature represents the preferred reaction temperature. This finding may be due to the increase in the escaping tendency of the metal ions from the solid adsorbent to the bulk phase; or due to the weakness of adsorptive forces between the active sites of the adsorbents and the adsorbate species and between the adjacent molecules of adsorbed phase[16].

## Thermodynamics studies

The adsorption thermodynamic equilibrium constant K was determined as the equation: $K = q_e/C_e$, where $q_e$ is the amount of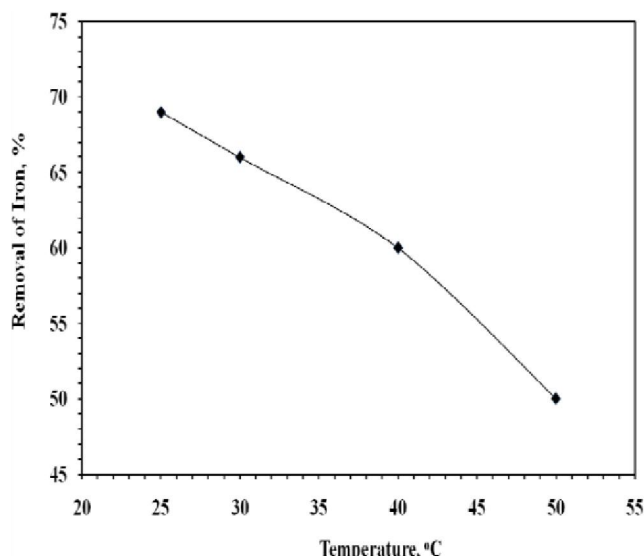 metal ions on the composite adsorbent and $C_e$ is the equilibrium metal ion concentration in solution (mol/L). The standard free energy change ($\Delta G_{æ\%}$ in kJ mol⁻¹), enthalpy ($\Delta H_{æ\%}$ in kJ mol⁻¹) and entropy change ($\Delta S°$ in kJ mol⁻¹) for the adsorption reaction were calculated using the following equations[30]:

$$\Delta G_o = -RT\ln K \tag{3}$$

where R is the gas constant (8.314 J/mol K) and T is the absolute temperature (K).

$$\ln K = (\Delta S_o/R) - (\Delta H°/RT) \tag{4}$$

The values of $\Delta H_{æ\%}$ and $\Delta S_{æ\%}$ could be calculated from the slope and intercept of the plot of lnK versus 1/T equation 4, Figure 8. The calculated values were given in TABLE 2. The negative value of enthalpy change $\Delta H_{æ\%}$ for the adsorption reaction reflects the exothermic nature of the process, while the negative entropy change refers to the affinity of composite adsorbent toward Fe(III) ions.

The negative values of free energy change reflect the spontaneous nature and feasibility of adsorption reaction.

## Adsorption isotherms

Adsorption isotherm was assayed by studying the adsorption of Fe(III) of wide concentration range upon the polymeric composite adsorbent. The amount adsorbed of iron onto adsorbent (mg/g) was represented against the equilibrium concentration of Fe(III) in phosphoric acid solution, Figure 9.
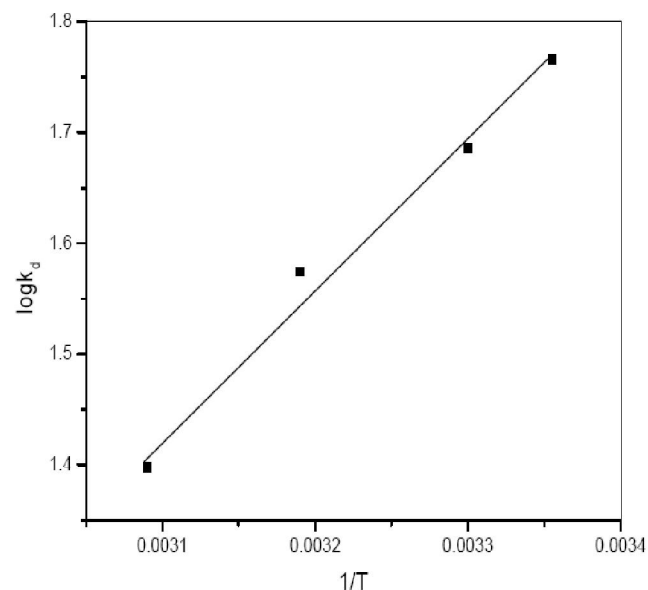


**Figure 7 : Effect of temperature on the adsorption of Fe(III) from phosphoric acid solution onto P(AM-AA)-TS; adsorbent dose**



**Figure 8 : plot of lnK against 1/T for removal of Fe(III) onto P(AM-AA)-TS**

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**TABLE 2: Thermodynamic parameters for removal of Fe(III) from wet-phosphoric acid solutions using P(AM-AA)-TS.**

| T(K) | $-\Delta G_o$ kJmol$^{-1}$ | $-\Delta H_o$ (kJmol$^{-1}$) | $-\Delta S_o$ (Jmol$^{-1}$K$^{-1}$) | S.D |
|------|------|------|------|------|
| 298 | 10.074 | | | |
| 303 | 9.779 | 11.171 | 22.772 | 0.02273 |
| 313 | 9.431 | | | |
| 323 | 8.644 | | | |

Langmuir isotherm model[31] is based on the assumption that maximum adsorption corresponds to a saturated monolayer of adsorbate molecules on the adsorbent surface that the energy of adsorption is constant and that there is no transmigration of adsorbate in the plane of the surface. Adsorption isotherm was obtained by shaking the adsorbent of fixed doses and the adosrbate solution containing varied concentrations of Fe (III) for 24 h.

Langmuir isotherm model represents the equilibrium distribution of metal ions between the solid and liquid phases. The following equation can be used to describe adsorption isotherm according to Langmuir:

$$1/q_e = (1/q°) + (1/bq°)(1/C_e) \qquad (5)$$

where $C_e$ is the equilibrium concentration of Fe(III) in solution (mg/L), $q_e$ is the amount adsorbed at equilibrium on adsorbent (mg/g), q° is the maximum metal ions uptake per unit mass of adsorbent (mg/g), which is related to adsorption capacity and b is Langmuir constant (L/mo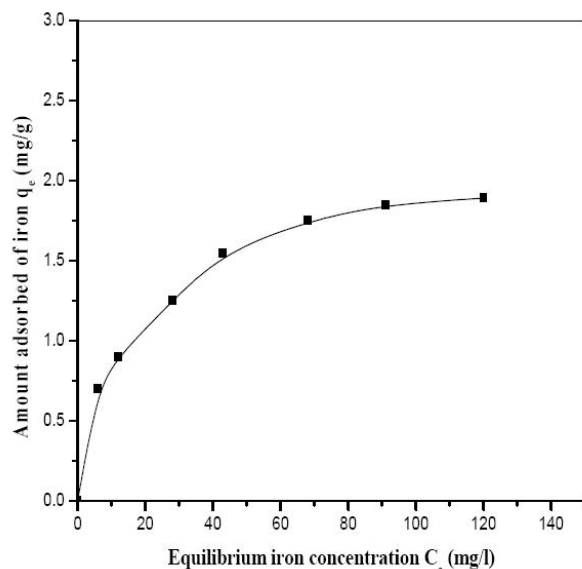l) which is exponentially proportional to the heat of adsorption and related to the adsorption intensity. Thus, a plot of $1/q_e$ vs. $1/C_e$ should be linear if Langmuir adsorption were applicable;

Consequently the Langmuir constants could be calculated. Figure 9 shows the equilibrium adsorption isotherm of Fe(III) in wet phosphoric acid using (P(AM AA)-TS) as an adsorbent. The isotherm rises sharply in the initial stages for low $C_e$ and $q_e$ values. This indicates that there are plenty of radial accessible sites. Eventually a plateau is reached, indicating that the adsorbent is saturated at this level. The decreases in the curvature of the isotherm are tending to a monolayer adsorption. Considerably increasing the $C_e$ values with slight increase in $q_e$, is possibly due to less active sites being available at the end of the adsorption process and/or the difficulty of the edge molecules in penetrating the adsorbent, while Fe(III) ions partially covering the surface sites. In order to optimize the design a sorption system to remove Fe(III) from commercial wet process phosphoric acid, it is important to establish the most appropriate correlation for the equilibrium curve. The linearzed Langmuir plot according to Eq. (5) is shown in Figure 10, and the Langmuir parameters were calculated and recorded in TABLE 3.

The favorable nature of adsorption can be expressed in terms of a dimensionless separation factor of equilibrium parameter, which is defined as:

$$R_L = 1/(1+bC_o) \qquad (6)$$

where b is the Langmuir constant and $C_o$ is the initial concentration of the adsorbate in solution[32]. $R_L$ was
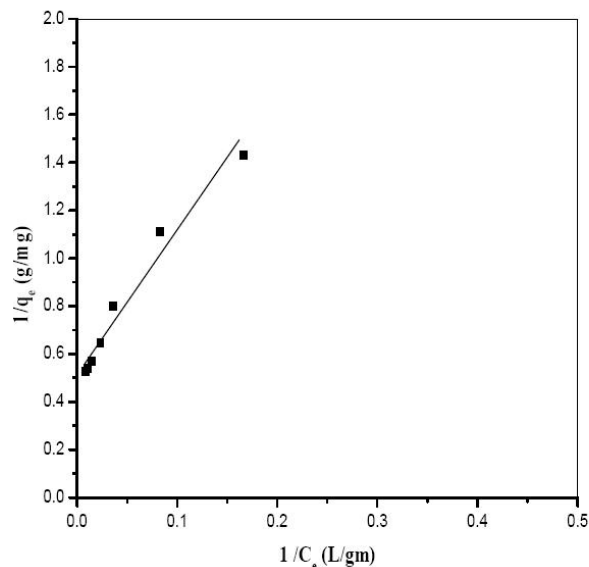


**Figure 9 : Adsorption isotherm for adsorption of Fe(III) onto P(AM-AA)-TS (adsorbent dose 40 g/L, time 24 h, pH 2.5).**



**Figure 10 : The linearzed Langmuir plot for the removal of Fe(III) from wet phosphoric acid solution.**

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**TABLE 3 : Langmuir constants for adsorption of Fe(III) onto P(AM-AA)-TS.**

| q° | b | R² |
|---|---|---|
| 1.99 | 0.0089 | 0.9822 |

less than one indicating favorable adsorption. The Langmuir constant values (q°) can be used to estimate the specific surface area, S, of sorbent[33] using the following equation

$$S = q^o NA /M \quad (7)$$

where S is the specific surface area, $(m^2/g)$ of adsorbent; q° is monolayer sorption capacity, gram metal per gram adsorbent; N is Avogadro number, $6.02 \times 10^{23}$; A is the cross-sectional area of metal ions, M is molecular weight of metal. The cross-sectional areas of Fe(III) has been determined to be 0.949 Å². The maximum specific surface area of polymeric composite adsorbent towards Fe(III) binding is 35.157 m²/g. The increased adsorbent surface area tends to increase the adsorbent removal efficiency[34]. The results indicate that (P(AM AA)-TS) could be potentially adsorb Fe(III).

The Freundlich model was chosen to estimate the adsorption intensity of the metal ions on the sorbent surface. The Freundlich equation is presented as[35]:

$$\log q_e = \log K_f + (\log C_e)/n \quad (8)$$

where $K_f$ (mg/g) and n are Freundlich constants incorporating all factors affecting the adsorption pro-
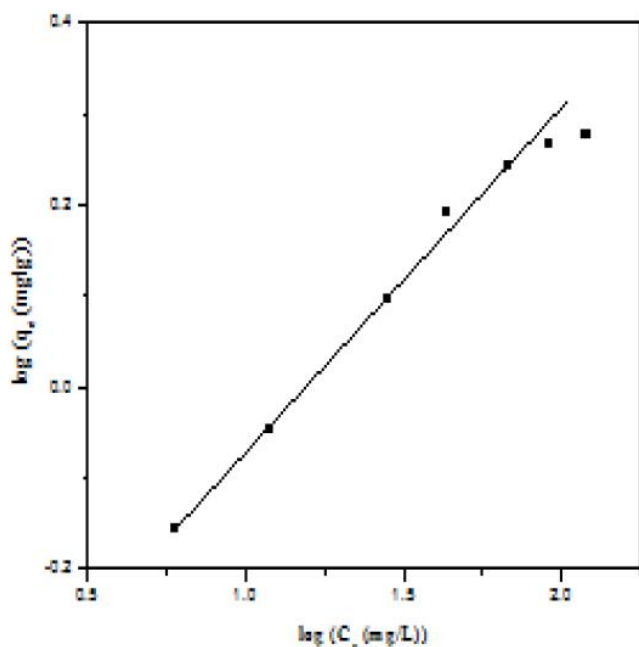


**Figure 11 : The linearzed Freundlich plot for the removal of Fe(III) from wet phosphoric acid solution**.

cess such as adsorption capacity and intensity of adsorption. These constants are determined from the intercept and slope of linear plot of log $q_e$ versus log $C_e$ (Figure 11) and listed in TABLE 4. Although the correlation coefficients are greater than 95%, they do not correlate the data as well the Langmuir isotherm, which has consistently higher correlation coefficients.

**TABLE 4 : Freundlish constants for removal of Fe(III) onto P(AM-AA)-TS**

| log K$_f$ | 1/n | R |
|---|---|---|
| -0.415 | 0.3498 | 0.9911 |

## Application of P(AM-AA)-TS for removal of iron from crude wet process phosphoric acid

The prepared P(AM-AA)-TS was studied for removal of iron from wet process phosphoric acid produced from Sebaiya phosphate ore through leaching with nitric acid. In this concern, 1 g of the composite was mixed with 25 ml phosphoric acid and left for 24 h with continuous shacking at room temperature. The iron concentration before and after experiments was measured using AAS, and the removal percentage was calculated and recorded. The loaded composite adsorbent was regenerated using 0.5 M HCl, where the adsorbent was shacked with 20ml solution of HCl 0.5M. The regenerated adsorbent was used in repeated sorption desorption regeneration cycles up to four cycles without significant decrease in sorption capacity; the results are presented in TABLE 5.

**TABLE 5 : The removal percentage and adsorbed amount of iron onto P(AM-AA)-TS in repeated sorption-desorption regeneration cycles.**

| Cycle No. | Removal % without |
|---|---|
| 1 | 46 |
| 2 | 42 |
| 3 | 40 |
| 4 | 36 |

## CONCLUSION

Titanium silicate as an adsorbent and ion exchange material could be supported within polymeric composite for enhancing their physicochemical properties.

P(acrylamide-acrylic acid)- titanium silicate was prepared, characterized and studied for removal of Fe(III) from wet-phosphoric acid solution.

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The pH has a notable effect on the removal of Fe(III) from phosphoric acid solutions, as it affects the iron speciation and adsorbent surface as well.

Studying the temperature effect revealed that the removal reaction is exothermic, spontaneous in nature.

The isotherm studies show that the experimental results could fit with Langmuir than Freundlish isotherm model, and the adsorption reaction is favored.

Application of P(AM-AA)-TS for removal of iron from crude phosphoric acid in batch experiment, showed that it could be potentially used in this purpose.

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