

Potentiality of Iron and Aluminum Extraction from Crude Phosphoric Acid using Modified Polyurethane Foam and Activated Carbon

Taha M.A. Razek¹, Mostafa M.H. Khalil², Awais M.S³ and Marwa M. Qayed^{4*}

¹Department of Environmental Basic Science, Ain Shams University, Egypt

²Department of Chemistry, Ain Shams University, Egypt

³Department of Chemistry, Nuclear Material Authority (NMA), Egypt

⁴Abu Zaabal Fertilizer Company (AZFC), Egypt

Abstract

The preparation of Iron (III) and Aluminum (III) adsorbents by impregnating two solvents namely, tri-octylamine (TOA) upon Polyurethane foam (PU) and charcoal (C) (charcoal) The relevant factors affecting solvents impregnation process including, solvents concentration, shaking time, volume/mass ratio, impregnation temperature and type of diluents were studied. Also, the adsorption characterization of the prepared adsorbents has studied. The optimum conditions affecting adsorption of Iron (III) and Aluminum (III) at pH of 3, temperature of 25°C for 1h and m/v ratio 10 g/L were the best conditions to remove Iron (III) and Aluminum (III). crude phosphoric acid (WPPA) used as a case study and showed a decrease in Iron and Aluminum from 8500 mg/L and 4234 mg/L to 5117 mg/L and 2012 mg/L.

Keywords: Heavy metals • Iron and aluminum recovery • TOA • Iron (III) and aluminum (III) sorption

Introduction

Environmental pollution has resulted from rapid industrial growth and a massive increase in the global population [1,2]. Because of their severe toxicity and proclivity for bioaccumulation in the food chain even at relatively low quantities, heavy metals in the environment are a major source of worry [3-6]. The US Environmental Protection Agency (USEPA) compiled a list of organic and inorganic chemicals discovered in wastewater that pose substantial health risks in 1978. Metal plating, electroplating, metal finishing, mining, fertilizers, paint, and paper production are only a few of the businesses that emit heavy metal-contaminated wastewater into the environment [7-11]. Heavy metal-containing wastewater must be cleaned before being discharged [12-15]. Growing interest in environmental protection, energy efficiency, process optimization, and fundamental chemistry progress has resulted in the development of innovative chemical separation techniques in recent years. From an ecological and economic standpoint, more specialized systems for dilute metal recovery have led to the development of new extractants, exchangers, and adsorbents [16]. Heavy metals, including precious metals, have been separated and recovered via solvent extraction extensively [17]. On the other hand, growing concern in user health, environmental protection, energy efficiency, and process optimization has resulted in a number of significant advancements in chemical separation medium. Solvent extraction, ion exchange, adsorption, membrane separation, photocatalytic degradation, biological treatment, and other methods can be used to selectively remove toxic components such as heavy metals, organic dyes, phenolic compounds

[18], biologically active compounds, and others from waste effluents and pre-concentrate different pollutants from aqueous solution. Two immiscible phases, one aqueous and the other organic, are involved in the solvent extraction technique, in particular [19]. Between the aqueous phase, which contains the pollutant, and the organic phase, which contains the solvent system with which pollutants form complexes and are separated [20,21], different components are distributed. The ability of the solvents to extract and interact with the solute causes the separation of these components [22]. When the concentration of the solute is quite high, as it is in the liquid effluents of numerous industrial processes, this separation technique may not be effective. In addition, there is a considerable amount of liquid to be processed. The ion exchange method [23] is an alternative to this technique for recovering the solute. The solute's ionic form is exchanged with counter (anionic and/or cationic) ions located on the polymeric matrix's surface. The method has many advantages over solvent extraction, and the polymeric resin's separation capability can be maintained across many cycles. Ion exchange resins have some disadvantages, such as the difficulty of generating a coordinating polymer, its expense, time-consuming preparation, and the inability to retain the selective qualities of created polymers. As a result, the solvent impregnated resin (SIR) concept was developed [24]. SIR is a mix of solvent extraction and ion exchange that can considerably enhance separation efficiency and selectivity for a wide range of substances to be eliminated. In a physical impregnation approach, a solvent or combination of solvents is introduced into the resin functioning as the support [25]. SIR could be utilized as a solid adsorbent with unique properties. Because of the solid phase's properties, porous resins impregnated with extracting solvents have given various advantages over liquid-liquid extraction [26-28]. They are made by soaking porous resins like polyurethane foam or charcoal in a volatile organic solvent containing extracting agents and then evaporating the solvent. In the preparation of solvent-impregnated resins, however, significant volumes of organic solvents are still used. We investigated two solvent-impregnated resins employing nonionic surfactants that are less hazardous, nonvolatile, and inflammable [29,30]. Polyurethane and charcoal were chosen because of their high surface area macroporosity, mechanical stability, and adsorption characteristics. SIR could be utilised as a solid adsorbent with unique properties. Because of the solid phase's properties, porous resins impregnated with extracting solvents have given various advantages over liquid-liquid extraction [26-28]. They are made by

*Address for Correspondence: Marwa M. Qayed, Abu Zaabal Fertilizer Company (AZFC), Egypt, Tel: +01096993701, E-mail: marwaqayed92@gmail.com

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soaking porous resins like polyurethane foam or charcoal in a volatile organic solvent containing extracting agents and then evaporating the solvent [29,31]. In the preparation of solvent-impregnated resins, however, significant volumes of organic solvents are still used. We investigated two solvent-impregnated resins employing nonionic surfactants that are less hazardous, nonvolatile, and inflammable. Polyurethane and charcoal were chosen because of their high surface area macroporosity, mechanical stability, and adsorption characteristics.

Materials

Iron (III) nitrate (Merck, Germany), Aluminum (III) (Merck, Germany), Phosphoric acid (Abu Zabaal Co., Egypt), N,N-dioctyloctan-1-amine (Acros Organics, Belgium), Polyurethane foam (Royal Foam Company, Egypt) and Charcoal (Adwic, Egypt), Benzene (Panreac Co.), toluene (Cornerstone Co.), acetone (Naser Co.), Butan-1-ol (Naser Co.), Kerosene (Misr-Petroleum Co., Egypt), Sodium Hydroxide (Merck, Germany) and Hydrochloric Acid (Merck, Germany).

Methods

Impregnation process

By cleaning the inert support for impregnation operations with distilled water, then squeezing to remove any dust, and finally washing with 2M HCl to activate the pores for impregnation processes as following:

Preparation of modified Resin occurred by using Polyurethane foam and charcoal Resin in order to remove inorganic impurities as well as monomeric material and activated macroporous via plug was squeezed in 2 M HCl in a batch extractor for 1 h, washed with distilled water until free of HCl, and again squeezed, and air dried overnight before using. The Polyurethane foam 0.5 cm in diameter and 0.50 cm long (average weight = 0.0500 ± 0.0050 g), were cut from a Polyurethane foam and charcoal used as granular shape with different size, different types of diluents were used for decreasing the viscosity and density of the organic solvent (TOA and D2HEPA) for increasing the modification process. The Polyurethane foam and charcoal were cleaned and dried plugs were modified on TOA and D2HEPA solution respectively and then diluents were evaporated. The extraction was carried out by a dynamic method in an automatic squeezing system. The heavy metals concentration was determined by Atomic absorption. The acids used were diluted to different concentrations by distilled water and standardized with sodium hydroxide solutions. Factors affecting the modification process [32], then washing several times with deionized water until chloride ion was not detected, and the fine floating particles were separated and discarded and finally dried at 60°C modified resins were prepared by using dry modification methods [33-34]. The modification procedure can be summarized as proper volume of modified solution placed in contact with 0.0.05 g dry macro-porous resin as polymeric support, and then the two phases were shaken in a mechanical shaker for different time intervals. The solvent modified resin was separated from the organic solution by filtration through a porous filter using a water pump for evacuation then the polymeric beads were washed several times with hot distilled water at 60°C followed by drying for resin beads at 60 °C.

Iron (III) and Aluminum (III) used as an example for heavy metal to examine the efficiency of impregnated resin. A synthetic solution of 100 ppm concentration of Iron and Aluminum was used.

Results and Discussion

Impregnation Trioctylamine (TOA) upon Charcoal (C) and Polyurethane (PU)

TOA concentration effect: Several impregnation experiments were carried out using identical circumstances at room temperature to investigate the effect of TOA concentration on impregnation results was 25 °C, time was

1h., m/v ratio was 5g/L and diluents type was kerosene with different TOA concentration was ranged from 0.02 up to 0.55 Mol/L. The results of the obtained impregnation graphically represented in Table 1 and Figure 1 which obtained that by increasing TOA amounts available in the solution, increasing impregnation until reaching a maximum at 0.45 Mol/L in the fifth trial, the curve became steady due to TOA saturating the porous surface of PU and C. So 0.45 Mol/L was the preferred TOA concentration.

Effect of shaking time: A series of impregnation experiments were performed under fixed conditions, including m/v ratio 5g/L, 0.45 Mol/L TOA concentration, temperature 25 °C, benzene as TOA diluents, and shaking time ranging from 0.50 to 24h, to investigate the effect of shaking time on the amounts of TOA impregnated on the dry PU and C working sample. The results were reported in Table 2 and Figure 2, which clearly show that the amount of TOA loaded onto PU and C increased as the impregnation time increased from 0.5 to 3 hours. Because all active pores of PU and C were loaded and saturated with TOA after 3 hours, there was no substantial increase in the amount of loaded TOA. As a result, 5 hours were recommended time due to give maximum % TOA impregnation for PU=94.55.

Table 1. Effect of TOA concentration impregnated onto the dry PU and C.

TOA concentration Mol/L	PU	C
0.02	9.22	8.6
0.05	10.27	8.86
0.11	31.1	20.78
0.23	48.81	39.03
0.45	81.69	64.38
0.5	81.69	64.35
0.55	81.69	64.35

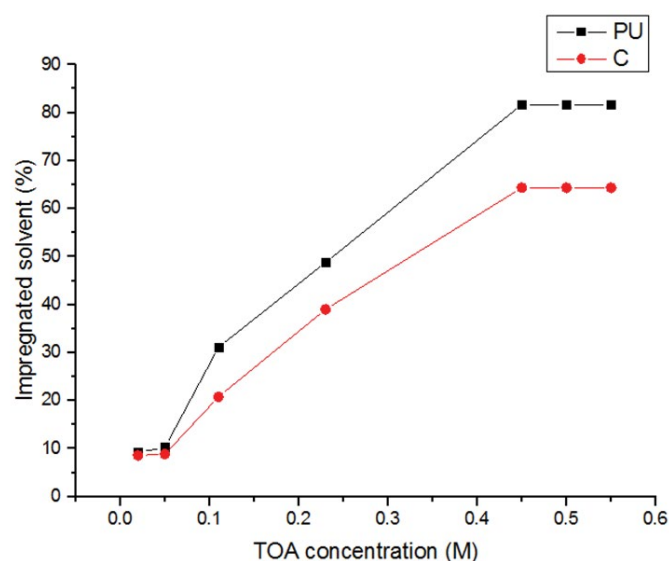


Figure 1. Effect of TOA concentration on the loaded amount onto PU and C (T=25°C, t=1h., m/v ratio=5g/L, diluents type=benzene. TOA concentration=0.02 upto 0.55 Mol/L).

Table 2. Effect of shaking time upon the loaded amount of TOA impregnated onto the dry the PU and C.

Shaking time ,h	PU	C
0.5	69.09	45.65
1	81.59	64.38
3	94.55	82.6
5	94.55	82.63
10	94.55	83
15	94.55	83
20	94.55	83
24	94.55	83

Effect of m/v ratio: A series of impregnation studies were carried out under fixed circumstances to investigate the influence of m/v ratio on the quantity of TOA. The experiments were carried out at 25°C for 3 hours with a TOA concentration of 0.45Mol/L, benzene as TOA diluents, and m/v ratios ranging from 1g/L to 10g/L. The obtained findings are reported in Table 3 and Figure 3, which show that the quantity of TOA loaded onto the PU and C grew as the impregnation solution volume rose, indicating that the amount of TOA contained in the solution increased. Because all active porous surfaces of PU and C are soaked with TOA solvent, there is no substantial increase in the quantity of loaded TOA beyond 5g/L for PU and 6g/L for C and become saturated after that decreasing in amount of impregnation of TOA solvent this due to reversibility where soluble pits of TOA solvent in diluents were used. So 5g/L for PU and 6g/L for C 6g/L as a prefer m/v ratio.

Effect of impregnation temperature: A series of impregnation experiments were performed to study the impregnation temperature effect under fixed conditions namely, TOA concentration of 0.45Mol/L for 3h., m/v ratio of 5g/L for PU and 6g/L for C 6g/L, benzene used as TOA diluents and temperature was ranged from 25 up to 70 °C. The obtained results shown in Table 4 and plotted in Figure 4 which clearly obvious that the amount of TOA loaded decreased with increasing of the temperature further than 25°C due to the PU and C surfaces change where become more flexible and begin to shrank. So 25°C a prefer temperature.

Effect of diluents type: To make the impregnation solution, diluents are needed to lower the solvent viscosity and density, increasing the solvent's extension on the dry PU and C surface and improving the TOA's capacity to access the inner PU and C pores. Several impregnation studies utilizing various types of diluents were carried out under controlled conditions to assess the diluents type effect. namely, benzene, toluene, acetone, Butan -1-ol, and Kerosene., at TOA concentration of 0.45 Mol/L for 3h, m/v ratio of 5g/L for PU and 6g/L for C 6g/L at 25 °C. The obtained results were summarized in Table 5 and Figure 5, which clearly obvious that benzene gives the best impregnation results due to give high impregnation and cheaper than other diluents were used.

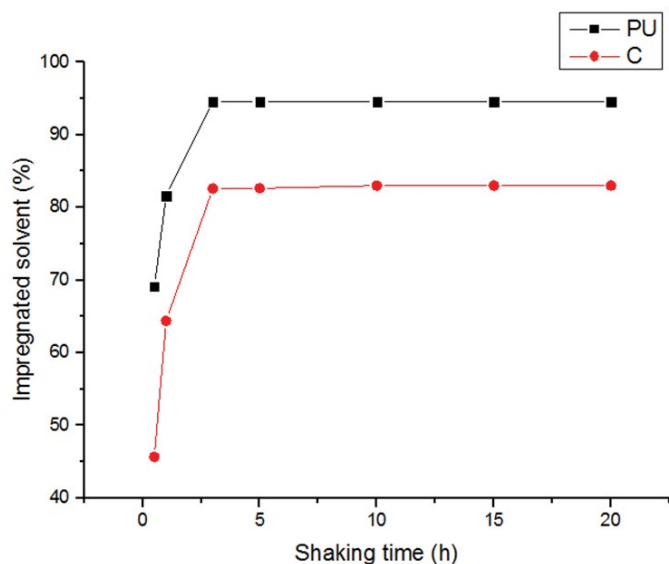


Figure 2. Effect of shaking time upon the loaded amount of TOA onto PU and C (TOA concentration=0.45 Mol/L, T= 25°C, m/v ratio=5g/L, diluents type=benzene and t=0.5 to 24 h).

Table 3. Effect of m/v ratio upon the loaded amount of solvent TOA impregnated upon the dry PU and C.

m/v ratio	PU	C
1g/L	30	20
2.5g/L	72.8	40.4
5g/L	94.2	82.8
6g/L	94.33	83.17
10g/L	94.8	83.3

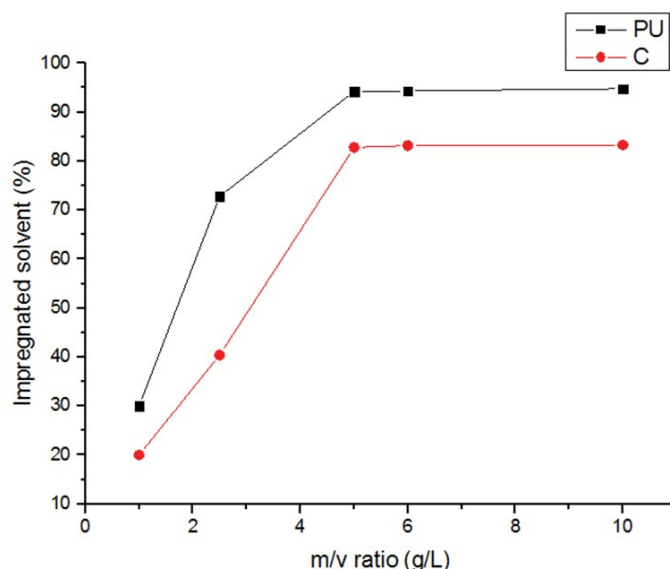


Figure 3. Effect of m/v ratio upon the loaded amount of TOA onto PU and C (T=25°C, t=3h., TOA concentration=0.45 Mol/L, diluents type =benzene and m/v ratio=1g/L up to 10g/L).

Table 4. Effect of temperature upon the loaded amount of TOA impregnated onto the dry PU and C.

Temperature, °C	PU	C
25	94.38	83.33
40	94.17	61.25
50	87.71	40.83
60	49.96	27.08
70	29.6	4.61

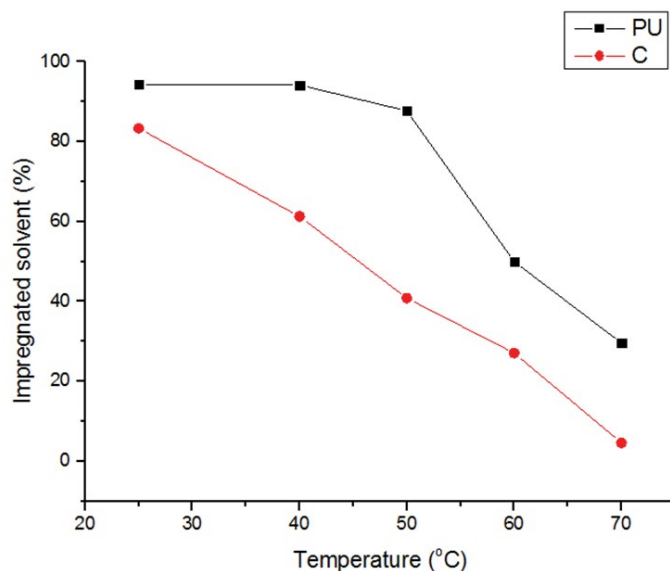


Figure 4. Effect of impregnation temperature upon the loaded amount of TOA onto the working PU and C (solvent concentration=0.45M, T=3h., v/m ratio=5g/L, solvent diluents= benzene and temperature=25 up to 70°C).

Choice of the prefer conditions: It is noticeable from the previous reported results of the relevant factors of the PU and C impregnation processes that different combinations of the relevant components of experimental circumstances might provide equivalent TOA solvent impregnated quantities. Economic concerns would drive the careful selection of these outcomes' preferred values. Given the considerations discussed, it appears cost-effective to use the following techniques for the TOA solvent impregnation process in Table 6 and plotted in Figure 6.

Table 5. Effect of type of diluents upon the loaded amount of solvent TOA impregnated onto the dry PU and C.

Diluents type	PU	C
Benzene	94.17	83.75
Kerosene	90.67	79.17
Acetone	80.17	70.83
Toluene	88.63	80
Butan- 1- ol	81.54	66.67

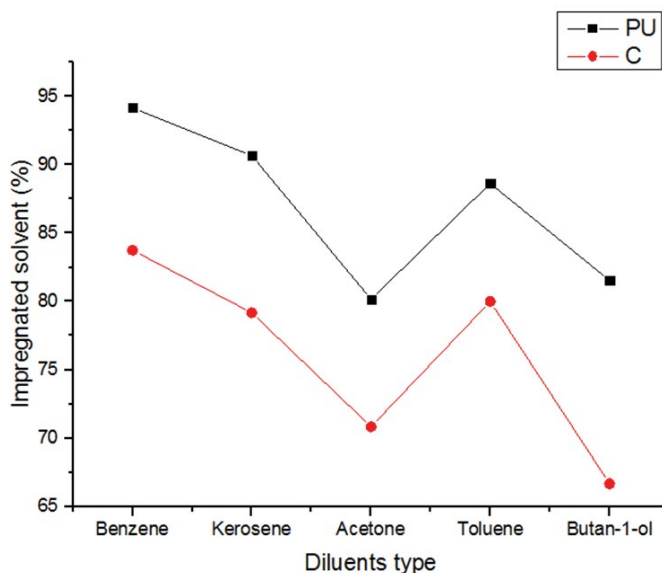


Figure 5. Effect of diluents type upon the loaded TOA amounts onto the dry PU and C (TOA concentration. =0.45M, T= 3h., m/v ratio=5g/L for PU and 6g/L for C and t= 25°C).

Table 6. The prefer values of impregnation TOA solvent onto the dry PU and C.

Factors	PU	C
Conc., Mol/L	0.45	0.45
shaking time, h	3	3
m/v ratio	5g/L	6g/L
Temp °C	25	25
Diluents type	Benzene	Benzene
% Impregnation	94.17	83.75

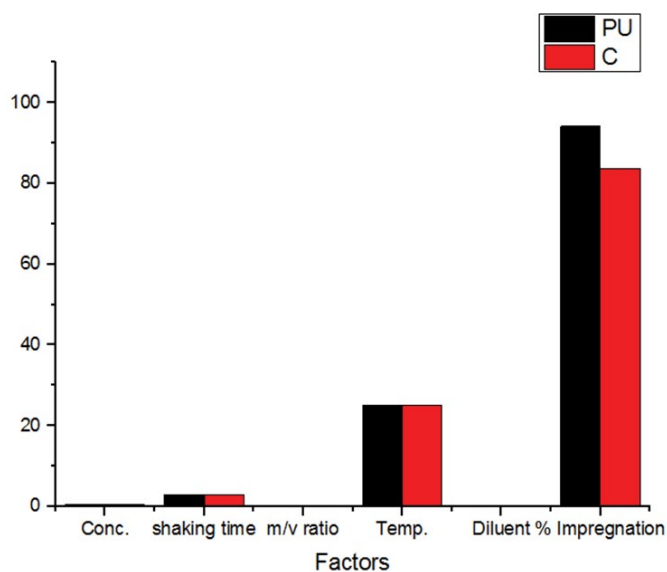


Figure 6. The prefer values of impregnation TOA solvent onto the dry PU and C.

According to the preferred values, percent Impregnation TOA on PU has the greatest value of 94.17, thus we recommend using Impregnation TOA on PU to remove Iron (III) from synthetic solutions.

Application of TOA upon PU on removal of Iron (III) and Aluminum (III)

Effect of pH: In order to study the effect of pH value of the nitrate solution upon Iron (III) and Aluminum (III) adsorption onto the prepared impregnated PU, a series of experiments were performed using different pH values were ranged from 0 up to 8. The experiments were performed under constant initial Iron (III) and Aluminum (III) concentration of 100 ppm at room temperature was 25°C for contact time was 1 hour. For this purpose, different aliquots of the Iron (III) and Aluminum (III) synthetic solution 100 ppm treated with HCl or NaOH solution to reach the required pH values. The results were recorded in Table 7 and plotted in Figure 7. From the obtained, it is clearly obvious that, there is a significant increasing of the adsorption efficiency from pH of 3 up to 8 further decreasing in pH value behind pH of 3 the adsorption effectiveness decreased significantly due to its highly acidic medium which affect strictly on the impregnated PU. It can thus be concluded that the pH value of 3 could be considered as the optimum acidity of the working solution. Sorption of the metal ions onto the modified polyurethane surface at a low H⁺ concentration in the solution (a high pH) was increased, due to the existence of low competition between the heavy metal and H⁺ ions for the sorption sites on the polyurethane.

Effect of contact time: To study the effect of the contact time upon Iron (III) and Aluminum (III) adsorption onto the prepared TOA impregnated PU, a series of adsorption experiments were performed m/v ratio 0.05g/10ml of PU with Iron (III) and Aluminum (III) solution of 100ppm at room temperature was 25°C, pH was 3 and time was ranged from 15 up to 300 min.

Table 7. Effect of pH upon Iron (III) and Aluminum (III) adsorption onto TOA impregnated PU.

pH	Iron removal %	Aluminum removal %
0	33.5	18.83
1	74	59.15
2	90.1	81.99
3	100	98
4	100	98
5	100	98
6	100	98
7	100	98
8	100	98

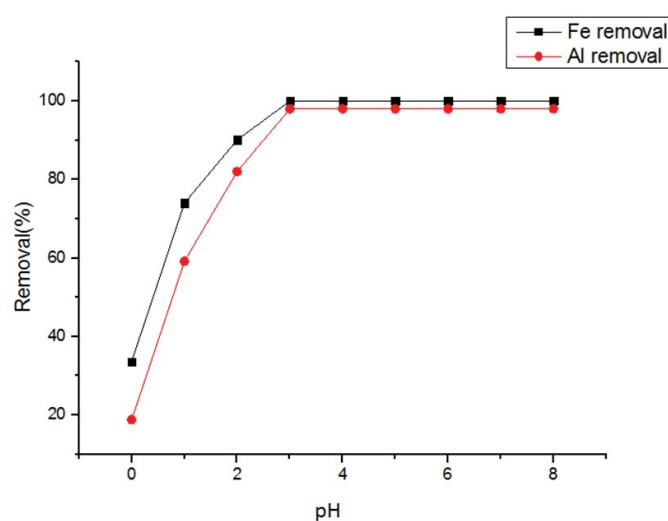


Figure 7. Effect of pH upon Iron (III) and Aluminum (III) adsorption onto TOA impregnated PU (T=25°C, t= 1h., m/v ratio=0.05gm/10ml and initial concentration=100ppm).

From the obtained results that recorded in Table 8 and plotted in Figure 8 which shows that Iron (III) and Aluminum (III) adsorption efficiency reach to its best was observed at the beginning of the adsorption operation. By increasing the contact time behind 15 min, adsorption efficiency increased at 60 minute shaking time was observed. Therefore, 60 minute was found to be appropriate for adsorption and was used in the subsequent experiments.

Effect of temperature: The Temperature effect on Iron (III) and Aluminum (III) adsorption onto TOA impregnated PU was studied through a series of adsorption including m/v ratio 0.05g/10ml of PU with Iron (III) and Aluminum (III) solution of 100ppm, pH was 3 and temperature was ranged from 20 oC to 80oC.

From the obtained results that recorded in Table 9 and plotted in Figure 9 which shows that Iron (III) and Aluminum (III) adsorption efficiency reach to its best was observed at room temperature. By increasing the temperature behind 20oC, adsorption efficiency decreased. Therefore, 20oC was found to be appropriate for adsorption and was used in the subsequent experiments.

Effect of m/v ratio: Under fixed conditions a series of experiments were performed to study the effect of m/v ratio upon Iron (III) and Aluminum (III) adsorption onto impregnated TOA with PU at pH of 3, temperature of 25°C for 1h, Iron (III) and Aluminum (III) concentration of 100 ppm and m/v ratio was ranged from 5g/L up to 25g/L.

The obtained results summarized in Table 10 and Figure 10 which clearly obvious that the efficiency of Iron (III) and Aluminum (III) removal decreased beyond 10g/L as the amount of solvent and the porous of the supporter was not enough to attract the heavy metal. So 10g/L used as a prefer m/v ratio.

Case Study on WPPA of Abu Zabaal for fertilizers Co

Wet-process phosphoric acid (WPPA) was produced in Abu Zaabal Company for Fertilizers and Chemicals, Egypt by Sulfuric Acid attack of Sebaiya phosphate rock followed by filtration. This technique provides a WPA with 26% of P₂O₅. WPA is diluted in the laboratory to reach 10% concentration of P₂O₅.

Table 8. Effect of contact time on Iron (III) and Aluminum (III) adsorption onto impregnated TOA with PU.

contact time, min.	Iron removal %	Aluminum removal %
15	47	50.1
30	74	80.18
60	100	97
120	100	97
180	100	97
240	100	97
300	100	97

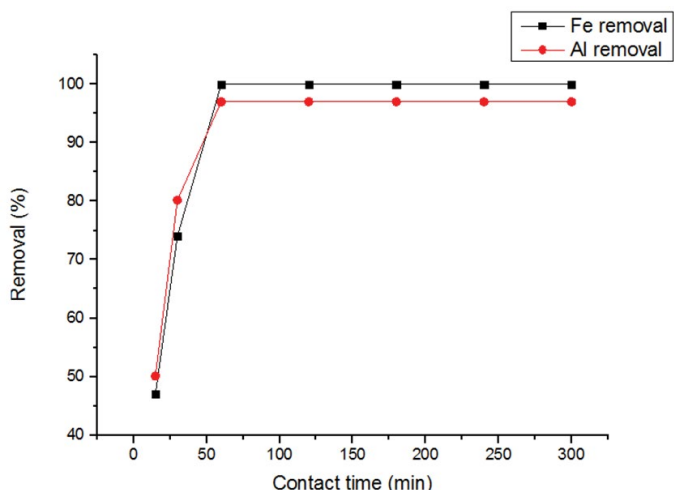


Figure 8. Effect of time upon Iron (III) and Aluminum (III) adsorption onto TOA impregnated PU (Ph=3, T=25°C, m/v ratio=0.05g/10ml and initial concentration=100ppm).

Table 9. Effect of temperature on Iron (III) and Aluminum (III) adsorption onto impregnated TOA with PU.

Temperature	Iron removal %	Aluminum removal %
20	91.4	87.2
35	86.31	80
50	65	61.81
65	40.5	39.11
80	32.1	40.1

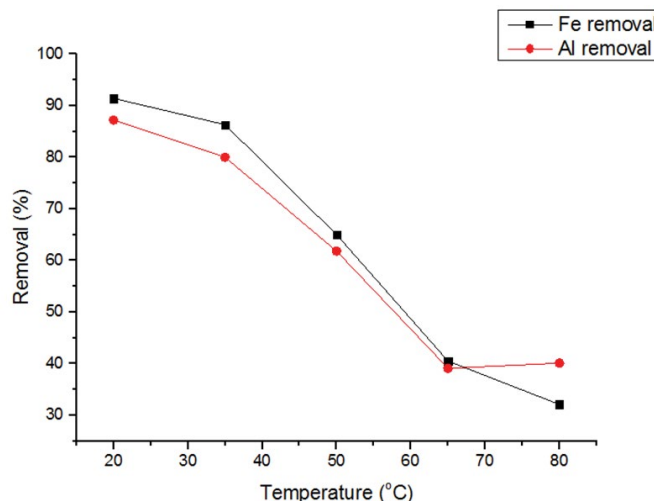


Figure 9. Effect of temperature upon Iron (III) and Aluminum (III) adsorption onto TOA impregnated PU (pH=3, t= 1h., m/v ratio=0.05gm/10ml and initial concentration=100ppm).

Table 10. Effect of m/v ratio on Iron (III) and Aluminum (III) adsorption onto impregnated TOA with PU.

m/v ratio	Iron removal %	Aluminum removal %
5	90.14	74.3
10	100	88
15	100	97.5
20	100	97.5
25	100	97.5

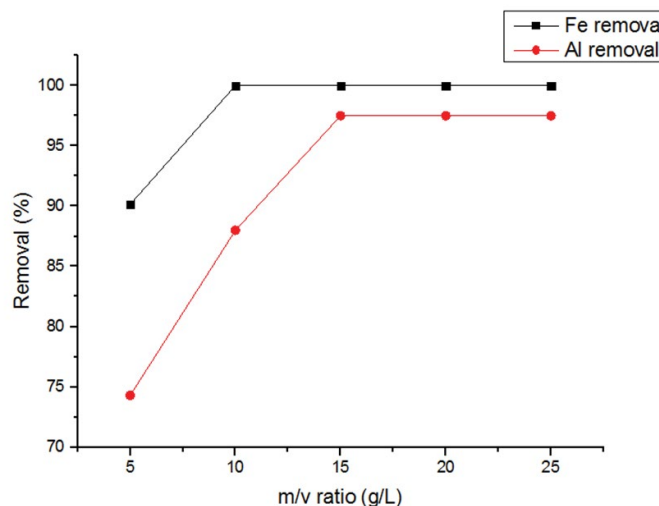


Figure 10. Effect of m/v ratio upon Iron (III) and Aluminum (III) adsorption onto TOA impregnated PU (pH=3, t= 1h., temp=25OC and initial concentration=100ppm).

By applying the above prefer results conditions that obtained on removal of Iron (III) and Aluminum (III) from WPPA in room temperature for 1 hr shaking time and 10g/L as a favour m/v ratio then determination the amount of Iron (III) and Aluminum (III) after and before adsorption in solution, the result showed a decrease in their concentration in WPPA (Table 11).

Table 11. Concentration of Iron (III) and Aluminum (III) before and after impregnation with TOA with PU.

Element	Fe		Al	
		mg/L		mg/L
Concentration	Before	8500		4234
	After	5117		2012

The present work deals with investigating the impregnation prefer conditions of TOA through removing of Iron (III) and Aluminum (III) from a synthetic solution as an example of a heavy metal. Using the extraction chromatography technique (solvent impregnated material), where this study discusses the preparation of two inert support (PU and C), each one of them impregnated by TOA through different processes. Choosing of PU as most preferable inert support for TOA which the % Impregnation values reached 94.17.

By using those conditions to be applied on removing of Iron (III) and Aluminum (III) as a study from a synthetic solution. pH, contact time, temperature and m/v ratio considerably affected the sorption capacity of modified polyurethane foam. pH of 3, shaking for 1 hr in room temperature besides using 10 g/l as m/v ratio, were the best conditions to remove both two metals.

The uniqueness of this work is applying impregnated TOA with PU, which has a significant promoted adsorption and purification ability, to WPPA that resulted a good removal of undesired heavy metals. Obtained results are promising in purification process which will reflect on protection of soil, water and environment in the future.

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