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The optimum conditions of Titanium Recovery process from the Iraqi Bauxite Ore

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A B S T R A C T

The present work is especially concerned with the Iraqi bauxite ore that includes 4.1% of titanium element (Ti). The goal of this study is to investigate the effect of various parameters (concentration of acid, ratio of solid: liquid, stirring speed, contact time, and temperature) in order to determine the best conditions for the leaching and extraction of titanium element. All tests of leaching were conducted with different acids (HNO_3, H_2SO_4, and H_3PO_4) with a ratio of solid: liquid from (1: 100) to (1: 8), the temperature of reaction from (25°C) to (50°C), period of time from (30 min) to (120 min), and speed of stirring from (400 rpm) to (1200 rpm). The best conditions were determined at (1: 100) solid: liquid ratio, (50°C) temperature, (4 M) HNO_3 concentration, and (900 rpm) stirring speed. While, the titanium extraction was from leached solution by (trioctylphosphine oxide) (TOPO), tri-n-butyl phosphate (TBP), and di-2-ethylhexyl phosphoric acid (D2EHPA) dissolved in kerosene) with an organic/aqueous ratio of (1/1, 1/2, 1/3, 2/1, and 3/1); temperature of reaction and period of time are correspondingly from (25°C) to (55°C) and from (8 min) to (15 min), and speed of stirring (400 rpm). The best conditions were obtained when using (55°C) temperature, (50%) TOPO concentration, (15 min) contact time, (400 rpm) speed of stirring and (1/3) O/A ratio.

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الشروط المثلى لاسترجاع التيتانيوم من خام البوكسايت العراقي

وليد تركي راشد قسم هندسة الانتاج والمعادن /الجامعة التكنولوجية/العراق – بغداد.
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الخلاصة

يتعلق العمل الحالي بشكل خاص بخام البوكسيت العراقي الذي يحتوي على 4.1٪ من عنصر التيتانيوم (Ti). الهدف من هذه الدراسة هو معرفة تأثير العوامل المختلفة (تركيز الحامض، نسبة المادة الصلبة: السائل، سرعة التحريك، زمن التلامس، ودرجة الحرارة) من أجل تحديد الظروف المثلى لترشيح واستخراج عنصر التيتانيوم. أجريت جميع تجارب الترشيح بأحماض مختلفة (HNO3، H2SO4، و H3PO4) بنسبة صلب: سائل من (1: 100) إلى (1: 8)، ودرجة حرارة التفاعل من (25 درجة مئوية) إلى (50) درجة مئوية)، والمدة الزمنية من (30 دقيقة) إلى (120 دقيقة)، وسرعة خلط من (400 دورة في الدقيقة) إلى (1200 دورة في الدقيقة). تم تحديد الظروف المثلى عند (1: 100) المادة الصلب: نسبة السائل، درجة الحرارة (50 درجة مئوية)، وبتركيز (4M) HNO_3، وسرعة خلط (900 دورة في الدقيقة). بينما، تم استخلاص التيتانيوم من محلول رشح بواسطة (TOPO، TBP، و D2EHPA مذاب في الكيروسين) بنسبة عضوية / مائية (1/1، 2/1، 3/1، 1/2، 1/3)؛ تتراوح درجة حرارة التفاعل من (25 درجة مئوية) إلى (55 درجة مئوية) والمدة الزمنية من (8 دقائق) إلى (15 دقيقة) وسرعة خلط (400 دورة في الدقيقة). تم الحصول على الظروف المثلى عند استخدام درجة حرارة (55 درجة مئوية)، (50٪) تركيز TOPO، (15)) دقيقة وقت التلامس، (400 دورة في الدقيقة) سرعة الخلط و (3/1) O / A.

الكلمات الدالة: تيتانيوم، ترشيح، خام البوكسيت، استرجاع.

1. INTRODUCTION

Bauxite is defined as a mix of minerals consisting chiefly of aluminum oxide that is bonded to one or more molecules of water (hydrated Al oxide). They're gibbsite, boehmite, and diaspore, [1], with a slight quantity of impurities, like K_2O, SiO_2, Fe_2O_3, Na_2O, CaO, TiO_2, and MgO [1]. Bauxite is the principal origin for the industrial manufacture of Al and Alumina via the Bayer technique. It's also utilized to manufacture refractory bricks; Through the Bauxite firing beneath (1200°C), and its structure is converted into dense granules containing chiefly Corundum ($\propto$-Al_2O_3). At the temperatures in the range (1250–1350°C), the phase of Mullite is developed a consequence of the reaction between the alumina and the silica [1]. Hydrargillite or Gibbsite lost its hydrate at the range (290–340°C) and convert into Boehmite, and at lower than the range (1200–1300°C), it's converted into corundum [1]. The wet bauxite remainder with slurry pH is around (12), owing to the existence of remaining NaOH. The bauxite remainder is stowed in discarding locations characteristically extending above numerous square kilometers and that postures an important danger. Such discarding location also aids in dewatering the bauxite remainder. Presently, the favored discarding technique is lagoon Ing as a marine discarding has been stopped, and as the bauxite remainder discarding in the sea may unfavorably influence the marine environmental equilibrium. The long-term storage of the bauxite remainder is a main topic because the bauxite remainder discarding locations aren't merely occupying enormous regions of land, which could else be utilized for agriculture, but the bauxite remainder also can result in grave contamination of the nearby groundwater, soil, and air. This environmental pollution displays that there's a requirement for the bauxite residue stockpiles remediation and a used solution of the stowed (legacy locations) and new bauxite remainder. Nevertheless, there're presently no uses of bauxite remainder in addition to slight use in the ceramic and cement manufacture [2, 3]. The Bauxite remainder has been stated to possess numerous likely uses [4], in the contamination governing area (the treatment of wastewater, the adsorption, and the acid leftover gases purification), as catalyst, coagulant, and adsorbent in paints and pigments, in the manufacture of ceramic, for the amendment of soil, the recovery of metal, and in the materials of construction. Nevertheless, none of such uses has been traditionally exploited upon a manufacturing scale up till now [5]. The Bauxite remainder is a polymetallic substance and a prospective origin of numerous metals [6]. Metals extraction from the bauxite remainder could get economically possible on condition that the appropriate methods of extraction being obtainable. The oxides of iron are the chief component of bauxite remainder and it is able to make till (60%) of the bauxite remainder mass. Actually, the red color bauxite remainders resulted via iron (III) oxides (frequently Hematite, Fe_2O_3). The Bauxite remainder also includes the Al, Ti, Si, and certain oxides valued metals, like the Rare Earth Elements (REEs). The aim of this work is the removal and recovery of Ti element from the Iraqi bauxite ore. The obtained element from the ores is considered as a significant opportunity for using them in some technological and industrial applications (Iraqi metals sources).

2. EXPERIMENTAL WORK

2.1. Materials and apparatus

Samples of bauxite were provided via the Iraqi Geological Survey from the Al-Hussainyat region in the Anbar province in the western of Iraq. Acid solutions and trioctyl phosphine oxide (TOPO), and kerosene were provided by El-Amin Chemicals Company, Iraq. The taken samples of the Bauxite ore rock being commented via the lab jaw crusher (Type Retch-BB100) as well as the ball mill (Type Retch-DM 200). This has been performed in the lab of Minerals Processing in the Production Engineering and Metallurgy Department at the University of Technology/Baghdad-Iraq. The sample being sieved for (10 min) in a lab sieve shaker (Type Retsch-AD60-01) employing a group of sieves having the sizes (250, 212, 150, 105, 75 and 53 µm), and the particle having a size from (-150 µm) to (105 µm) being utilized for the leaching experimentations. The analysis of the X-Ray fluorescence (XRF) of the Bauxite ore was accomplished via (model Spectro Xepos) in the Geology Department at the Baghdad University, Iraqi - Germany Lab, as depicted in Table1. The flow chart for procedures, including the recovery of Ti element from the bauxite ore is shown in Fig.1.

Table1.
The XRF analysis of the Iraqi Bauxite powder

Elements	Concentration (%)
Al_2O_3	42.2
SiO_2	12.41
P_2O_5	0.68
CaO	0.2
TiO_2	4.41
ZrO_2	0.24
Fe_2O_3	1.81
L.O.I	0.3

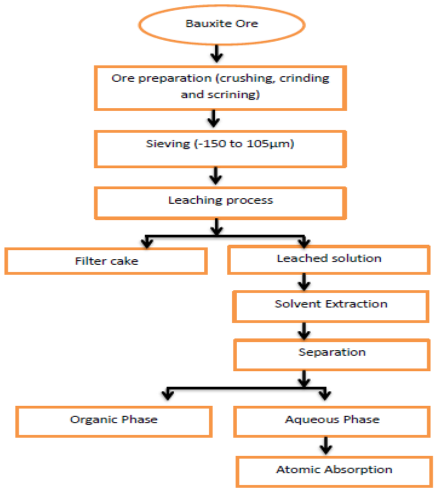

Fig.1. Flow chart for recovery of Ti element from Bauxite ore.

2.2. Leaching Process of Bauxite Ore

The leaching of titanium element from bauxite ore by acid (HNO_3) was performed in a (500 ml) necked round bottom reaction glass vessel shown in the Fig.2. The reaction vessel was heated using the magnetic stirrer hot plate. The stirring of the mixture was done by the magnetic stirring coated by Teflon. The ore particles size of phosphate samples was kept constant at (from -150 µm to +105 µm) throughout the experiments. The leaching efficiency (L%) can be calculated using the following equation [7]:

$$L\% = \frac{(C1\ \text{Ã—}\ V)}{(Co\ \text{Ã—}\ W)}\ \text{Ã—}\ 100 \qquad (1)$$

Where:
L %: The leaching efficiency (%)
$C1$: The metal concentration in solution in (g/l)
Co: The metal concentration in solid (wt%)
V: The leaching solution volume (l)
W: The solid sample weight (gm).

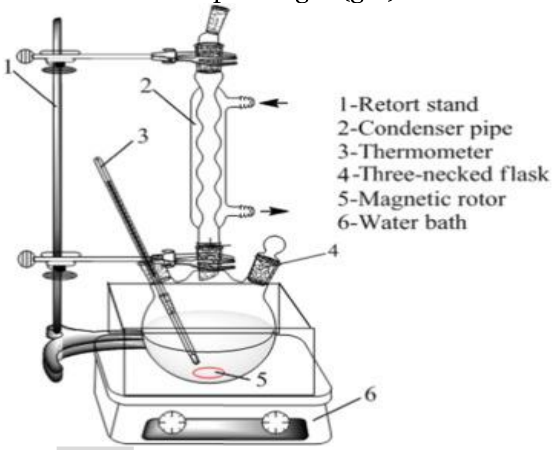

Fig.2. Leaching system (glass vessel).

2.3. Extraction process of titanium element

The solvent extraction technique was employed for the separation of titanium element transferred from the aqueous to the organic phase. The extraction percentage (E%) reflects the separation and extraction degrees of elements. An organic phase of the mixture (TOPO) was used as an extraction agent dissolved in the diluent. The present compounds were diluted using kerosene. The extraction percentage (E%) and the ratio of distribution (D) were calculated by the following equations [8, 9]:

$$D=[Me]org/[Me]aq \quad \textbf{(2)}$$

$$E\%=[D/D+Va/Vo]x100 \quad \textbf{(3)}$$

Where, [Me]org and [Me]aq are the concentrations of metal ions in the organic and aqueous phases, correspondingly. E is the percentage of extraction, D is the ratio of distribution, and (Vo, Va) are the volume of the organic and aqueous phases, correspondingly.

3. RESULTS AND DISCUSSION

Recovery Ti element process involves firstly the leaching of bauxite ore using nitric acid, and then titanium was extracted using TOPO from the leached solution.

3.1. Leaching of Ti element

The leaching process involves the extraction of titanium from the bauxite ore using acid, and the particles size was (105-150 µm). The results were obtained when the Iraqi bauxite ore was subjected to leaching with the acid solution under the following parameters: type of acid, acid concentration, stirring speed, reaction time, reaction temperature, solid to liquid ratio, and sold to liquid ratio.

3.1.1. Influence of acid type on leaching process

Influence of the type of acid (HNO_3, H_2SO_4, and H_3PO_4) with concentration (1 M) on the Ti element leaching was studied with contact time 30 min, temperature 25°C, solid/liquid (S/L) ratio (1/100 g/ml) and stirring speed 400 rpm. The obtained results are presented graphically as a relation between total leached percentage (L%) and acid type in Fig.3. HNO_3 has a more significant effect than other acids, and this is probably because the nitric acid is a strong acid and has a relatively small molecular weight, and this might be anticipated for having a fast diffusion rate in comparison to other acids in the leaching process. Thus, nitric acid has been chosen for the upcoming experiments.

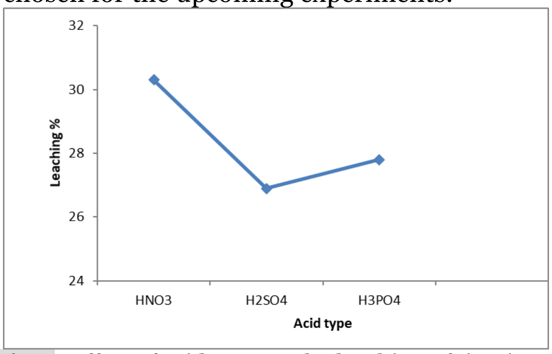

Fig.3. Effect of acid type on the leaching of titanium.

3.1.2. Influence of the concentration of acid upon the leaching

Influence of the concentration of NHO_3 upon the leaching procedure of Bauxite ore was investigated at various concentrations (1-4 M) at speed (400 rpm), time (30 min), temperature (25°C), solid to liquid (1/100 g/ml), and fraction of particle size (105-150 µm). The investigational outcomes elucidated that, when the concentration of acid was raised from (1 M) to (4 M), the Ti element leaching increased from (30.3%) to (67.2%). This might be ascribed to that increasing the acid concentration increased the acid attack on the structure which results in more broken bonds, and this is also attributed to the dominated hydrogen ions and the diffusion rate of hydrogen ions [10]. Therefore, more Ti element has been leached from the bauxite ore, as it can be seen in Fig.4. Thus, the (4 M) acid concentration has been chosen for the upcoming experiments.

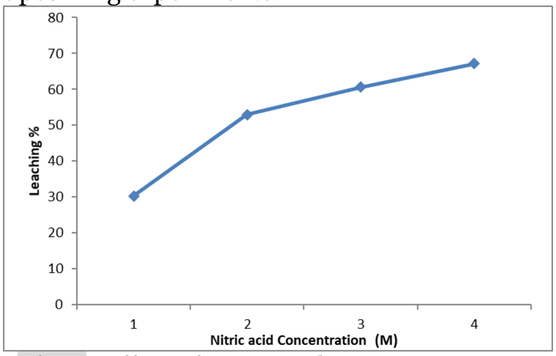

Fig.4. Effect of nitric acid concentrations on the leaching of titanium.

3.1.3. Influence of the stirring speed upon leaching

The impact of stirring speed on the bauxite ore dissolution through the nitric acid was examined at stirring speeds of 400, 600, 900, and 1200 rpm. Furthermore, the other parameters were fixed at 25°C temperature, 30 min reaction time, and (1/100 g/ml) S/L, as well as 4 M nitric acid concentration. Experimental results are provided in the Fig.5. specifying that the leaching of titanium element increases gradually at speeds between 400 to 900 rpm. However, if the stirring speed is raised for higher than (900 rpm), the efficiency of leaching starts to decrease. During the leaching process, the heterogeneous reaction occurs at the interface, which is between the phases of solid and liquid, at the boundary between two phases, and the layer of diffusion was created. Regarding the solid into the aqueous state, such layer includes a fixed aqueous layer, and the layer of diffusion might be thinned through the excessive speed of stirring, yet not totally eliminated, in which the leaching effectively elevated with the increase in stirring speed for 900 rpm. While the noted lower efficiency of leaching with a higher stirring rate of more than 1200 rpm might be ascribed to the aggregation of the granules as a result of the collision that has resulted from the high speed, this reduced the dispersion of the granules in the solution [11]. Thus, the stirring speed (1200 rpm) is not suggested for the leaching related to the elements under study. Therefore, a stirring speed of (900 rpm) was selected for the later experiments.

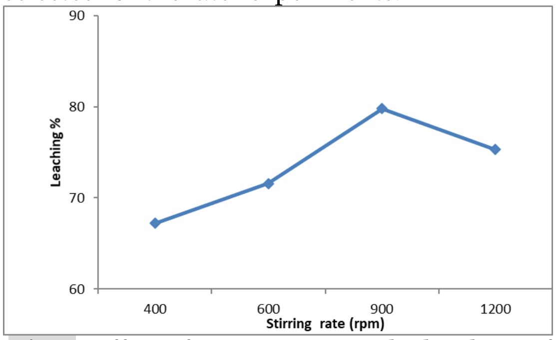

Fig.5. Effect of stirring rate on the leaching of titanium.

3.1.4. Influence of the Time of Contact Upon Leaching

Fig.6 manifests the influence of the time of contact upon the leaching percentage of the Ti element. The investigated periods are (30, 60, 90 and 120 min) at 4 M concentration, 25°C temperature, 1/100 S/L, and 900 rpm stirring speed. The best percentage of leaching is (83.2%) at the contact time (120 min). The cause for rising the percentage of leaching with the leaching period increment is owing to that the increment in time raises the solution solubility via contacting the solids and providing enough time for the solution diffusion into the solids, therefore raising the rate of reaction [12].

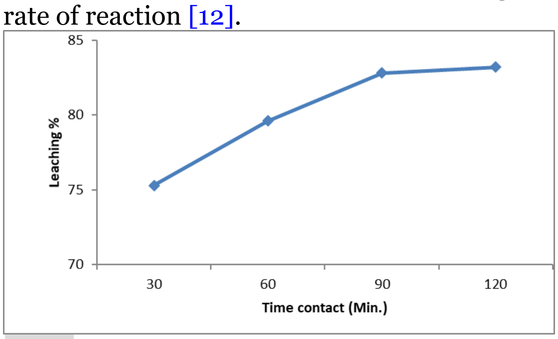

Fig.6. Effect of time contact on the leaching of titanium.

3.1.5. Influence of the Solid/Liquid Ratio Upon Leaching

The optimum ratio for S/L (1/100 g/ml) was selected from many experiments that were performed (1/100, 2/100, 4/100, 8/100, and 20/100 g/ml). The concentration of 4 M nitric acid, temperature 25°C, stirring speed 900 rpm, and contact time 120 minutes were selected. Fig.7 depicts the association between the S/L ratio and the leaching that was related to Ti element from the bauxite ore. With a decrease in the ratio of solid/liquid, the leaching efficiency of the ore sample increased [13]. High liquid to solid ratio was anticipated for reducing the slurry's viscosity through achieving better mixing, contributing to the decrease in the diffusional mass transfer resistance. On the other hand, the increased solid percentages led to increased viscosity, hindering the stirring that is related to reactants, thus decreasing the reaction rate. Besides, a high ratio of liquid-to-solid indicated more solution with hydrogen ions, which accelerated the leaching reaction [14]. According to that, a (1/100 g/ml) solid to liquid ratio was selected for the later experiments.

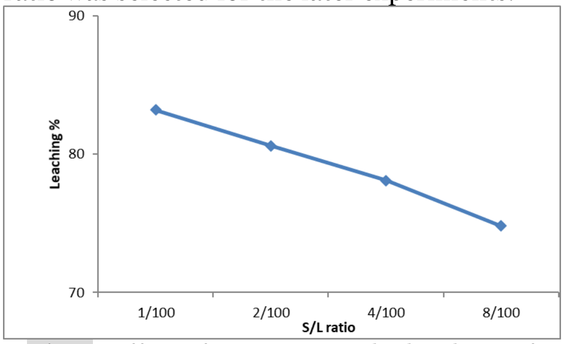

Fig.7. Effect of S/L ratio on the leaching of titanium.

3.1.6. Influence of Temperature Upon Leaching

To study the influence of temperature ranging from 25 to 50±1°C on the leaching of Ti element has been investigated. The other parameters have been fixed at 120 min reaction time, 900 rpm, stirring speed, 4 M nitric acid concentration, and (1/100 g/ml) S/L ratio. The obtained results were presented graphically in Fig.8 as a relation between leaching efficiency and temperature. From this figure, it is clear that the Ti dissolution efficiency (%) was slightly increased by increasing the reaction temperature from 25 to 50°C. Therefore, the room temperature represents the preferred temperature for the other factors of experiments. The temperature has an essential effect on the dissolution kinetics, because the leaching of elements is a diffusion dependent process. Therefore, increasing the reaction temperature also increases entropy, including the speed of molecular motion, and will increase the collisions between H+ and minerals [15]. Therefore, the rise in temperature allows the dissolution rate to increase. This result agrees with the results that have been provided by [16, 17], which mentioned that the increased temperature leads to increase the leaching efficiency.

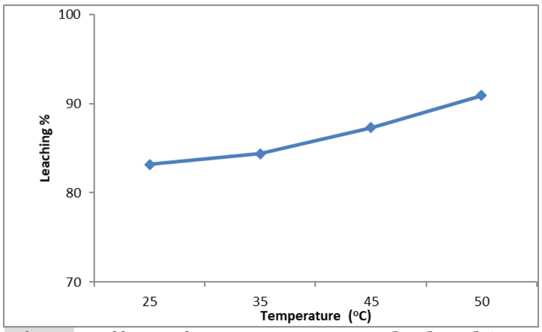

Fig.8. Effect of temperature on the leaching of titanium.

3.2. Extraction of Ti Element

Extraction of Ti element after the leaching of bauxite ore by nitric acid using the best selected leaching parameters. The extraction parameters are type of extraction agent, concentrations of extraction agents, contact time, and phase ratio (organic/aqueous).

3.2.1. Influence of extraction agent type

Three different reagents (TOPO, TBP, and D2EHPA dissolved in kerosene) were tested separately in the extraction of titanium from the bauxite ore. Using 30% from every solvent, the procedures being conducted at (25°C), 30 min time of contact, (A: O = 1: 1) aqueous: organic ratio, and 400 rpm stirring rate. In general, the best titanium extracting agent is TOPO. The results are shown in Fig.9. TOPO has been suggested as the extraction agent, which has been the optimum regarding the later experiments.

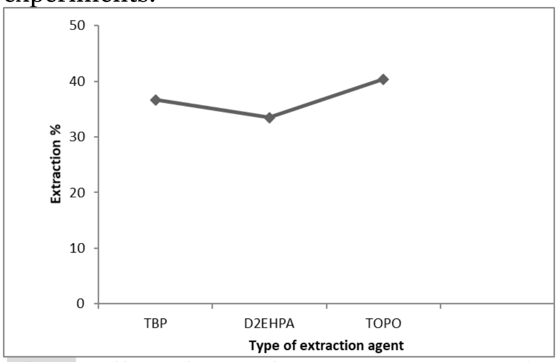

Fig.9. Effect of type of extraction agent on the extraction of titanium.

3.2.2. Influence of TOPO concentration

The influence of 10-60% TBP concentrations was tested on the extraction efficiency of the element The mixture was subjected to agitation for 10 min at temperature 25°C, aqueous: organic ratio (A: O = 1: 1), and 400 rpm stirring speed. The results are evinced in Fig.10. The TOPO concentration values in kerosene are considered an influencing factor in the extraction of Ti element. There was an increase in the extraction effectiveness of Ti element with concentration to reach a maximal value of 50%. Also, there was a decrease in the extraction effectiveness after such percentage. The increment in the concentration beyond 50% decreased the dilution related to TOPO in kerosene that reduced its capability for the extraction of elements [18].

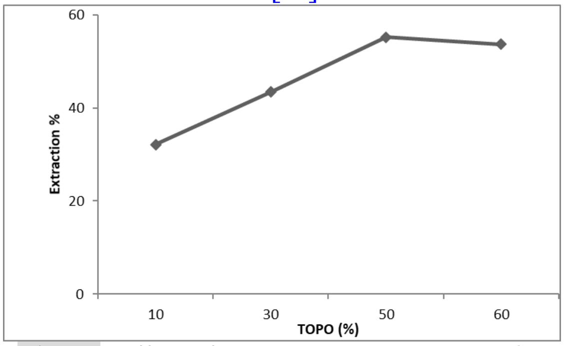

Fig.10. Effect of TOPO concentration on the extraction of titanium.

3.2.3. Influence of The Ratio of Organic to Aqueous (O/A)

Experiments were carried out at the O/A ratios (1/1, 1/2, 1/3, 2/1, and 3/1), the optimum value reached 64.6% at 1/3(O/A) ratio, concentration is (50% TOPO +50% kerosene), and 10 min time of contact. The organic to aqueous (O/A) ratio effect upon the Ti extraction% is displayed in Table 2.

Table4.

The Ti extraction% at various O/A ratios

No.	O\A	Extraction%
1	1\1	55.2
2	1\2	61.3
3	1\3	64.6
4	2\1	60.8
5	3\1	58.4

3.2.4. Influence of the time of contact

Titanium extraction was achieved at various times (8, 10, 12, and 15 min) of contact. The best outcome reached (70%) at (15 min) time of contact. The influence of the time of contact upon the Ti extraction via TOPO is portrayed in Fig.11. The TOPO concentration as well as the ratio of O/A was kept constant at 50% and 1/3, correspondingly. Also, this figure illustrates that there's an increment in the efficiency of the Ti element extraction through the first 10 min due to the availability of sufficient time for the extraction process. However, no major differences in the further rise in contact time from 12 to 15 min have been observed. According to this result, the reaction between aqueous medium and organic extracting has reached the equilibrium at 12 min [19,20].

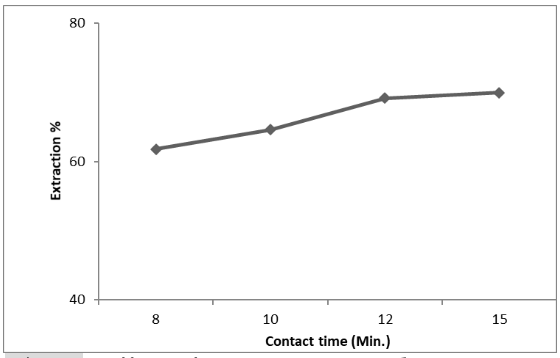

Fig.11. Effect of contact time on the extraction of titanium.

3.2.5. Influence of temperature

To study the influence of temperature on the extraction process, which is related to Ti, the experimentations were carried out with 12 min contact time at temperature (25-55°C). Also, the extraction study was conducted with using 400 rpm stirring speed, 50% TOPO/Kerosene at an A/O 3:1 phase ratio and the data being schemed between the temperature and the Ti element E% in Fig.12. This data exhibit that there's a notable increment in the Ti element E% occurred at 55°C temperature. In such regard, the procedure of extraction has to be conducted at elevated temperatures, since by temperature rise, viscosity of the solvent as well as the extract usually reduces. From the other side, the extract solubility into solvent raises [21]. Therefore, the temperature (55°C) is appropriate for giving the best percentage of the Ti element extraction.

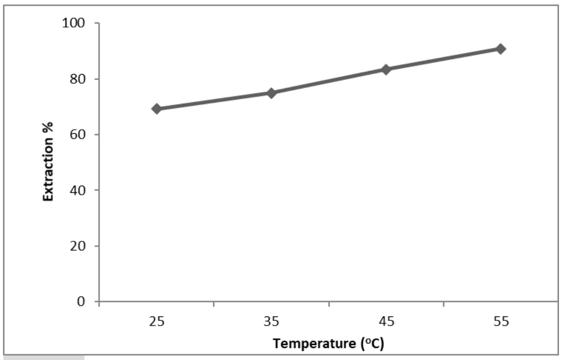

Fig.12. Effect of temperature on the extraction of titanium.

4. CONCLUSIONS

The obtained conditions of leaching and extraction of Ti element (91%) and (90.6%) respectively can be summarized:

The best leaching process of Ti element from the bauxite ore was obtained with nitric acid (HNO_3) compared with other leaching agents (H_2SO_4 and H_3PO_4) which is obtained at 50°C temperature, 4 M HNO_3 concentration, 90 min time of contact, (1/100 g/ml) S/L ratio, and 900 rpm stirring speed. The highest extraction percentage for Ti element was when using 50% TOPO-kerosene, at a temperature of 55°C, time of 12 min, organic/aqueous ratio 1/3, and stirring speed of 400 rpm. The TOPO is more efficient than TBP and D2EHPA as an agent extraction.

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