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Research Paper

Extraction and Separation of zirconium using Aliquat 336 from Egyptian black sand

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ABSTRACT:
A solvent extraction method has been employed to extract and separate zirconium from the nitric acid medium using 6% tri-caprylmethylammonium chloride (Aliquat 336) $[CH_3(CH_2)_7]_3N^+(CH_3)\ Cl^-$ dissolved in kerosene as diluent. The relevant factors controlling the extraction process of zirconium using Aliquat 336 were studied. These factors include Aliquat 336 concentration, A:O phase ratio, contact time and diluent type. More than 99.6% of Zr was extracted by 6% Aliquat 336 at contact time 10 minutes, phase ratio O:A (v: v) 2:1 and the diluent were kerosene. Third phase formation was studied and dissolving it by using decanol as modifier was also done. Stripping of zirconium from loaded Aliquat 336 has been carried out using 4M H_2SO_4 as an effective stripping agent. The feasibility of using Aliquat 336 for separation of zirconium was assisted by stripping studies. The loaded zirconium onto Aliquat 336 has been stripped by stripping efficiency 94.18% when using 4M H_2SO_4 as an efficient eluting agent at 10 minutes contact time and phase ratio O:A (v: v) 1:1.

KEYWORDS: *Solvent extraction; Zirconium; Aliquat 336; Stripping.*

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I. INTRODUCTION

Zirconium (with symbol; Zr) is a soft, malleable, lustrous, greyish white and ductile transition metal which becomes brittle, hard at lower purities and solid at room temperature [1-2]. It is a strong transition metal which resembles to Hafnium (Hf) to a greater extent and, to a lesser extent Titanium (Ti). The silicate mineral, zircon ($ZrSiO_4$) is the chief source of zirconium [1]. Zircon has been produced as the by-product from the mining and processing where rutile and ilmenite are being mined [3]. Zirconium has a wide range of coordination complexes and inorganic compounds which are colorless and diamagnetic solids [4]. In those complexes, Zr possesses the oxidation state +4. Till today, fewer compounds are found where Zr has +3 oxidation state and Zr (II) is very rare. Zirconia, also known as Zirconium dioxide (ZrO_2) is the most common oxide which is solid and colorless compound. Zirconia in its cubic form has chemical resistance and exceptional fracture toughness [5] which is also used as the thermal barrier coating [6].

Zirconium as metal finds application in production of parts of nuclear reactor, as it is highly resistant to corrosion, has a very low absorption cross-section of thermal neutrons and high melting point [7]. Zirconium has many industrial applications such as the ceramic industry, enamels, refractory materials, glazes, foundry mold and abrasive grits dye pigments and catalyst in organic reactions [8]. Traditional system for separating Zr (IV) by liquid extraction consists of 60% TBP (tri-n-butyl phosphate) solution in kerosene and 6 M HNO_3 MIBK (methyl isobutyl ketone)/NaSCN/HNO3 and TBP/HCl solvent extraction processes are used for separation of Zr, but the downstream contain high concentration of ammonium, cyanide and by-products of organic [9-10]. The development of an effective separation method still attracts the attention of many separation chemists; solvent extraction [11-12], solid/liquid extraction [13–15].

In this study, solvent extraction technique was used for the extraction of zirconium from as-prepared Zirconium nitrate solution resulted by alkali fusion of Egyptian Rosetta zircon. The extraction process was done by using Aliquat 336 and Factors affecting on the extraction process were evaluated. These factors include O: A phase ratio (v: v), Aliquat 336 concentration, contact time and diluent type. In addition, the third phase formation during the extraction process was also studied. The factors affecting on stripping of zirconium from

loaded Aliquat 336 like A:O phase ratio, contact time, type of stripping agent and stripping agent concentration, were studied to determine the optimum conditions for extraction and separation of zirconium using Aliquat 336.

II. EXPERIMENTAL

Materials and methods:

All chemicals used in the experiments were purchased and used as received without further purification. Materials used in this work alizarin red S, Zirconyl chloride octahydrate ($Cl_2OZr.8H_2O$, 98%), ethanol, butanol, pentanol, octanol, decanol, Hydrochloric acid, sodium hydroxide and nitric acid, Aliquat 336 were from Sigma–Aldrich. Egyptian Rosetta zircon mineral (97%) with chemical composition that ZrO_2+HfO_2 (66.42%), SiO_2 (32.23%), Fe_2O_3 (0.14%), TiO_2 (0.22), ThO_2 (0.02%), U_3O_8 (0.04%), MgO (0.02%), CaO (0.01%), RE Oxides (0.07%), Al_2O_3 0.06%), K_2O (<0.01%), Na_2O (<0.01%) and P_2O_5 (0.13%) by (ICP.Ms).

Preparation of zirconium nitrate solution

A sample of 250g of Egyptian Rosetta zircon was mixed with 312g of sodium hydroxide pellets and charged to a stainless steel crucible. The crucible was then fed to the electric furnace where the fusion reaction took place. The reaction temperature was adjusted at 650 °C for 2h. The fusion product was mixed with (4 times of its weight) distilled water at 60 °C and stirred for 2 hrs to remove sodium silicate solution. The residue ($Na_2ZrO_3.H_2SiO_3$) was dissolved in concentrated HCl at 90 °C with stirring for 2 hrs then immediately filtrated giving residue which mainly composed of unreacted zircon, TiO_2, Al_2O_3, ThO_2 and H_2SiO_3. The filtrate was crystallized at low temperature giving zirconyl chloride hydrate crystals which then filtrated and dissolved in 1M hydrochloric acid and pH was adjusted to 9 using ammonia solution to precipitate Zr $(OH)_4$[16]. The obtained zirconium hydroxide was dissolved in certain volume of nitric acid to form of zirconium nitrate solution($Zr(NO_3)_4$).

Extraction of zirconium using Aliquat 336

A mixture of 10 ml of as-prepared Zr $(NO_3)_4$ solution (4.11 g/l Zr^{+4}) and volumes of diluted Aliquat 336 (keeping the required phase ratio) was stirred vigorously in 50 ml beaker using magnetic stirrer at room temperature for certain contact time. The mixture then transferred to a separating funnel and allowed to be settled down for 20 minutes. The aqueous and organic phases were separated and the aqueous samples were analyzed. The factors which control the extraction process were studied in details like; phase ratio (O: A) v: v, solvent concentration, contact time and the effect of diluents used. In addition, the third phase formation during the extraction process was also studied and the usage of alcohols for solving this problem was done by using different types of alcohols.

Stripping of zirconium from loaded Aliquat 336

All the experiments were performed on the pregnant organic solution Aliquat 336, a mixture of 10 ml of the loaded organic (3.35 g/l of Zr^{+4}) and certain volume of stripping agent (keeping the required phase ratio) was stirred vigorously in 50 ml beaker using magnetic stirrer and at room temperature for certain contact time. The mixture then transferred to a separating funnel and allowed to be settled down for 20 minutes. The aqueous and organic phases were separated and the aqueous samples were analyzed. The factors controlling the stripping process were studied as, the effect of the type of stripping agent, the concentration of the stripping agent, the contact time and the effect of phase ratio (A: O) v: v.

Chemical analysis of Zirconium

Zirconium was analyzed in all aqueous phases using alizarin red S method [17-18]. Absorbance of the formed zirconium alizarin complex was measured at 520nm against proper standard solutions using A double beam UV–Visible spectrophotometer of high-resolution power model Jasco V-530.

Characterization of ZrO2 product

For characterization of the produced ZrO_2 was done using UV-visible absorbance spectra were recorded using spectrophotometer and ESEM-EDX analysis.

III. RESULTS AND DISCUSSION

Effect of organic solvent (O): Aqueous solution (A) ratio

The effect of organic solvent (O) to aqueous solution ratio was evaluated at different O: A ratio (from 3:1 to 1:5) using 2% Aliquat 336 in kerosene and contact time 5 minutes. Figure (1) shows that the extraction efficiency of zirconium slowly increased from O: A 1:5 till 1:2 (E% increased from 6.9 to 17.2%) but after 1:2 the extraction efficiency highly increased from O: A 1:2 till 2:1 (E% increased from 17.2 to 58.66%), then slowly increased

after 2:1, so that the best phase ratio for the extraction of zirconium from nitric acid medium using Aliquat 336 was found to be 2:1 (O: A) (v: v).

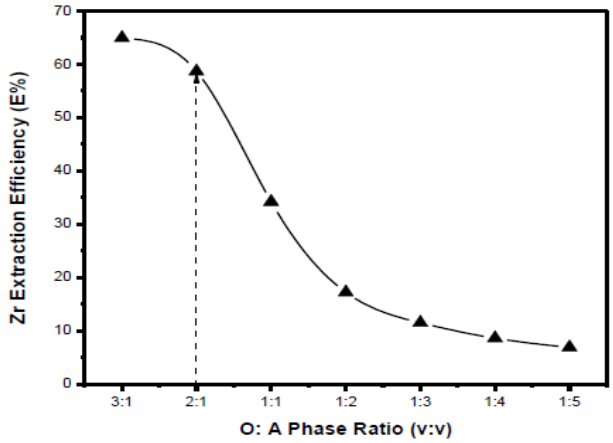

Figure (1): Effect of O: A phase ratio on the extraction of zirconium from nitrate medium using Aliquat 336.

Effect of organic solvent concentration

Experiments have been done to study the effect of organic solvent concentration on the extraction of zirconium using Aliquat 336 organic solvents while the other variables were kept constant thus O: A phase ration is 2:1, diluent is kerosene and contact time 5 minutes. The obtained results Table (1) and Figure (2) show that the extraction efficiency of zirconium was highly increased with increasing the concentration of Aliquat 336 till concentration 6% (E% increased from 58.66 to 96.46%). But after the concentration 6% the extraction efficiency became stable, so that the best organic solvent concentration is 6%.

Table (1): Effect of organic solvent concentration on the extraction of zirconium using Aliquat 336.

Aliquat 336 Conc., (%)	Zr(A), g/l	Zr(O), g/l	D(Zr)	Extraction efficiency, %
2	1.70	1.21	0.71	58.66
4	0.97	1.57	1.61	76.27
6	0.14	1.98	14.14	96.46
8	0.060	2.02	33.66	98.5
10	0.051	2.03	39.80	98.75

Zr(A), Zirconium concentration in aqueous phase
Zr(O), Zirconium concentration in organic phase (Aliquat 336)
D(Zr), Distribution coefficient for zirconium (D(Zr)= Zr(A)/Zr(O))

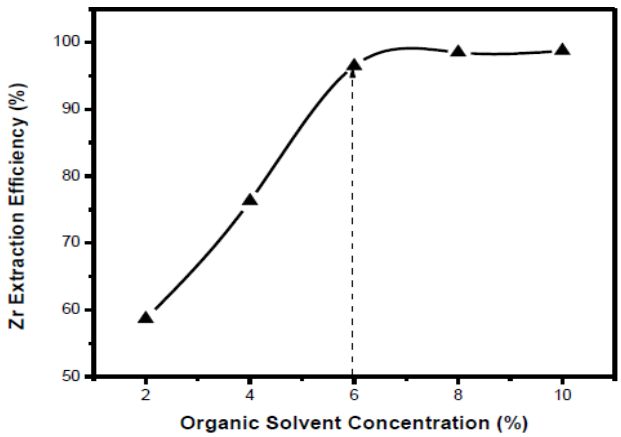

Figure (2): Effect of Aliquat 336 concentration on the extraction of zirconium from nitrate medium.

Effect of contact time

The contact time needed for the extraction of zirconium from nitric acid medium was determined by making experiments at contact time range from 1 to 30 minutes using 6% Aliquat 336 in kerosene and phase ration (O: A) 2:1. Table (2) and Figure (3) shows that the extraction efficiency of zirconium was increased with increasing contact time from 1 minute to 10 minutes (E% increased from 94.61 to 99.6%), but after 10 minutes the extraction efficiency became constant. Finally, the best contact time for the extraction of zirconium from nitrate medium is 10 minutes.

Table (2): Effect of contact time on the extraction of zirconium using Aliquat 336.

Contact Time, (min.)	Zr(A), g/l	Zr(O), g/l	D(Zr)	Extraction efficiency, %
1	0.22	1.94	8.8	94.61
5	0.14	1.98	14.14	96.46
10	0.015	2.05	136.6	99.6
15	0.020	2.04	102	99.5
20	0.048	2.03	42.29	98.81
25	0.22	1.94	8.81	98.82
30	0.14	1.98	14.14	98.84

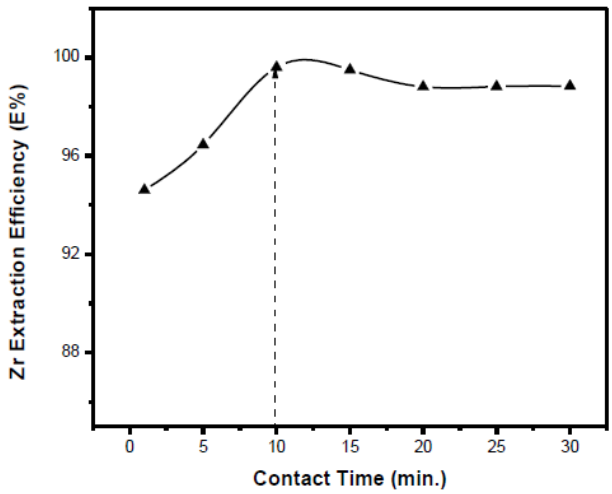

Figure (3): Effect of contact time on the extraction of zirconium using Aliquat 336 from nitrate medium.

Effect of diluent type

The effect of diluent type was studied by using four types of diluents (Benzene, Toluene, Carbon tetrachloride and kerosene) while the other variables were kept constant like 6% Aliquat 336, O:A ratio 2:1 and 10 minutes contact time. Figure (4) shows that zirconium extraction efficiency depends on the diluent type and the best diluent for Aliquat 336 and gave the highest extraction efficiency was kerosene (E% is 99.46%).

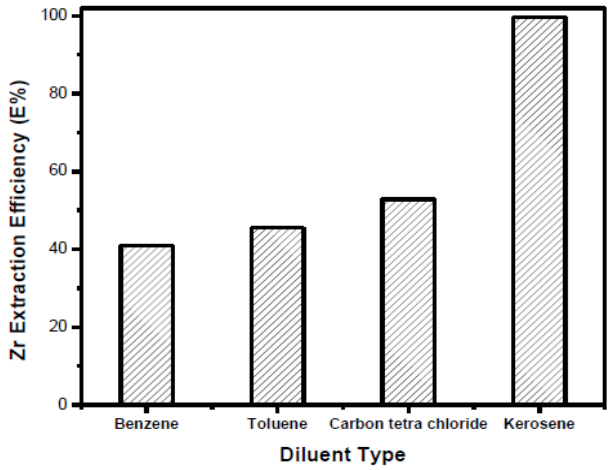

Figure (4): Effect of diluent type on the extraction of zirconium using Aliquat 336 from nitrate medium.

Effect of using different modifiers for dissolving the third layer

The third phase formation is troublesome to the both extraction and stripping processes because it changes the mass transfer and phase separation, and this decrease the extraction efficiency. Extensive studies have been conducted on the third phase formation in various extraction systems to understand the kinetics of third phase formation and they found that low temperature and high concentration of nitric acid greatly facilitate the formation of the third phase [(19)]. In addition, the metal concentration in the aqueous phase and the type of organic solvent also has great effect on the third phase formation [(20-23)]. Series of experiments were done using various types of alcohols like Ethanol, Butanol, Pentanol, Octanol and Decanol. The obtained results show that Ethanol, Butanol and Pentanol react with nitric acid in aqueous phase with the evolution of nitrogen oxides as reddish-brown color. On using Octanol and Decanol third phase dissolved and the ratio of Octanol to Aliquat 336 (v: v) was 2:10 and thus of Decanol to Aliquat 336 was 1:10, so that the best modifier used in this work was Decanol because less amount will be enough to dissolve third layer.

Distribution isotherm equilibrium curve

Distribution isotherm is a plot of the equilibrium concentration of the extracted species of zirconium in the extracted phase against its concentration in the raffinate layer. Distribution isotherm can be prepared for either the extraction process (and in this case it is called extraction isotherm) or the stripping process (which called stripping isotherm). Data for the extraction isotherm could be easily obtained from either ‘‘phase ratio variation’’ single contact of a fixed volume of aqueous feed (input leach liquor) with different volumes of organic solvent or else by ‘‘saturation process’’. Repeated contact of one and the same aliquot of the organic solvent with several aliquots of fresh input leach liquor. McCabe-Thiele diagram is a composite plot of the distribution isotherm and the operating line. The operating line could be established by only one point, which corresponds to the final raffinate composition and the ratio of the aqueous to organic phases that determines the slope of the line, as it is a straight line. The diagram can be used to evaluate the approximate number of theoretical stages required for the extraction process.

In this study, different volumes of the organic solvents (6 % Aliquat 336 in kerosene) were contacted with different volumes of the aqueous feed (nitrate solution) at the optimum conditions previously determined as, contact time 10 minutes, O: A ratio 2:1 and the diluent are kerosene, the aqueous and organic phases were separated and then analyzed for amount of zirconium extracted. The equilibrium line is then established by plotting the concentration of zirconium in the organic phase versus its concentration in the aqueous phase as in Table (3) and as represented in Figure (5) [(24)]. The next step is construction of McCabe-Thiele diagram. It is clear that two theoretical stages are quite suitable for zirconium extraction using Aliquat 336.

Table (3): The obtained data for construction of McCabe-Thiele diagram for Zirconium extraction using Aliquat 336.

No	Zr(A), g/l	Zr(O), g/l	Zr(O) total, g/l
1	0.042	4.09	4.09
2	2.67	2.78	6.88
3	6.83	0.70	7.58
4	7.34	0.44	8.03

5	7.85	0.19	8.22
6	8.04	0.09	8.32
7	8.24	-	8.32

Zr(O) total, Total zirconium loaded on Aliquat 336

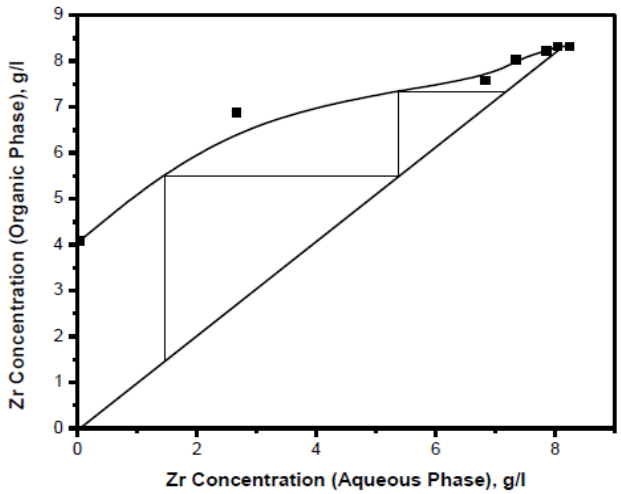

Figure (5): McCabe-Thiele diagram for the extraction of zirconium using Aliquat 336.

Stripping of the loaded zirconium Aliquat 336 in nitrate acid medium

Experiment of extraction using the optimum conditions was done for studying the factors affecting on the stripping efficiency of zirconium from loaded Aliquat 336. The amount of loaded zirconium on Aliquat 336 after the extraction process was found to be 3.35 g/l with extraction efficiency 97.56%.

Effect of stripping agent

The effect of stripping agent was studied by using different stripping agents (H_2O, NaCl 10%, HCl 1M, H_2SO_4 1M and HNO_3 1M) and the other factors kept constant thus A:O phase ratio 1:1 and contact time 5 minutes. Figure (6) shows that the stripping efficiency of zirconium from loaded Aliquat 336 greatly depends on the type of stripping agent thus the best stripping agent was found to be 1M H_2SO_4 (S% was 41.74%) and stripping factor (SZr) was 0.72 this mean great distribution of zirconium from loaded Aliquat 336 to 1M H_2SO_4.

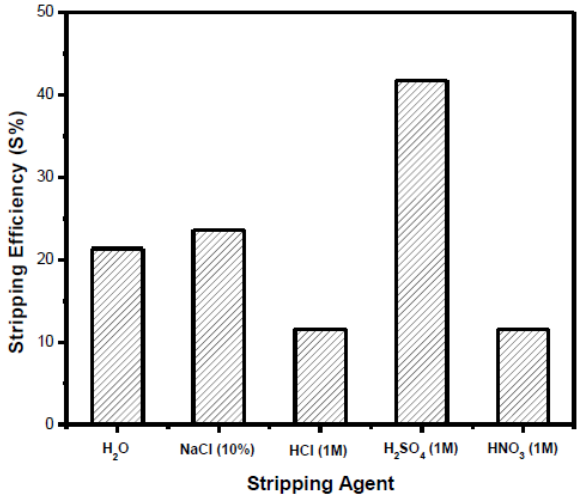

Figure (6): Effect of stripping agent on the stripping of zirconium from loaded Aliquat 336.

Effect of acid molarity

The effect acid molarity was studied using different molar concentration of H_2SO_4 at contact time 5 minutes and A:O phase ratio 1:1. Figure (7) shows that the stripping efficiency increases with increasing the molarity of acid

from 0.5 to 4M (S% increased from 20.54 to 72.1%) and then decreased, so that 4M H_2SO_4 is sufficient for stripping of zirconium from loaded Aliquat 336.

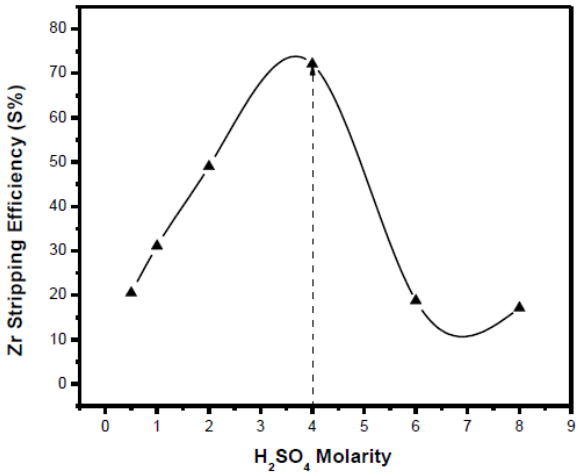

Figure (7): Effect of stripping agent concentration on the stripping of zirconium from loaded Aliquat 336.

Effect of A: O phase ratio

The effect of A: O (v: v) phase ratio was studied at range 5:1 to 1:3, the contact time was 5 minutes and 4M H_2SO_4. Figure (8) shows that the stripping efficiency of zirconium from loaded Aliquat 336 was slowly increased from phase ratio A:O 5:1 till reach 3:1 (S% increased from 23.46 to 31.13%), and after 3:1 the stripping efficiency highly increased till 1:1 (S% increased from 31.13 to 72.09%), but after 1:1 the stripping efficiency decreased, thus the best phase ratio is 1:1.

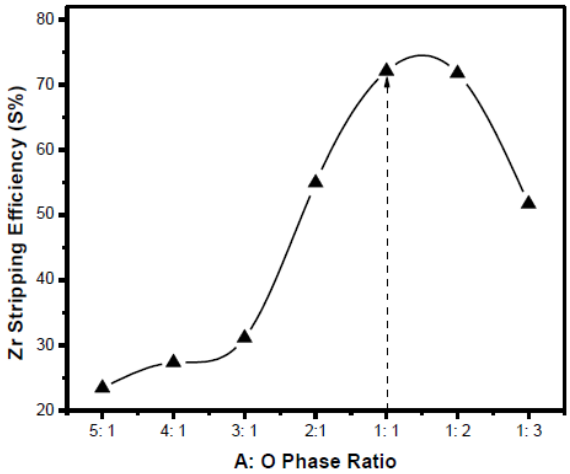

Figure (8): Effect of A:O phase ratio on the stripping of zirconium from loaded Aliquat 336.

Effect of contact time

The effect of contact time on the stripping of zirconium from loaded Aliquat 336 in nitrate medium was studied at different contact time from 5 to 30 minutes while other factors kept constant thus A:O phase ratio 1:1 and 4M H_2SO_4 stripping agent. The stripping efficiency was increased from contact time 5 minutes to 10 minutes (S% increased from 72.1 to 94.18%) then it decreased (Figure 9). It is clear that the best contact time for the stripping of zirconium from loaded Aliquat 336 is 10 minutes.

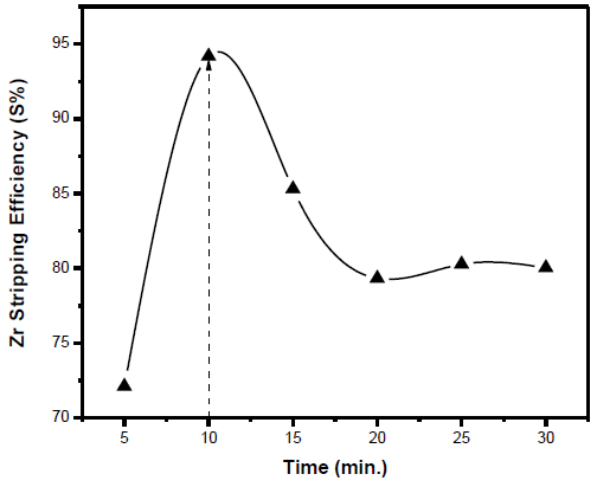

Figure (9): Effect of contact time on the stripping of zirconium from loaded Aliquat 336.

Distribution isotherm equilibrium curve

Fixed volume of the aqueous phase (4 M H_2SO_4) was contacted for 10 minutes with different volumes of the loaded Aliquat 336 until equilibrium is obtained keeping fixed phase ratio (A:O) 1:1. A measured portion of the aqueous phase was taken for Zirconium analysis. The results shown in Table (4) and represented in Figure (10). By construction of McCabe-Thiele diagram for stripping of zirconium from the loaded Aliquat 336, it is clear that three stages are sufficient for the stripping of zirconium from loaded Aliquat 336.

After stripping process, the stripping agent loaded with zirconium was adjusted by ammonia solution to pH 9 to precipitate zirconium as Zr $(OH)_4$ which filtrated and dried at 60 ℃ overnight, then burned at 550 °C for 3 hours to form ZrO_2 which used for complete characterization.

Table (4): The obtained data for construction of McCabe-Thiele diagram for stripping of zirconium from loaded Aliquat 336.

No	Zr(A) total, g/l	Zr(A), g/l	Zr(O), g/l
1	0.31	3.06	0.29
2	0.58	2.76	0.58
3	0.86	2.79	0.56
4	0.99	1.35	1.99
5	1.08	0.83	2.52
6	1.11	0.30	3.04
7	1.12	0.13	3.22
8	1.13	0.07	3.27
9	1.13	0.04	3.31
10	1.13	0	3.35

Zr(A) total, Total zirconium stripped in aqueous phase.

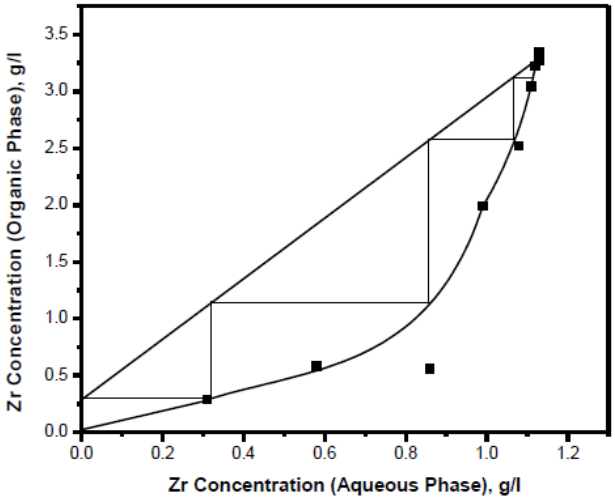

Figure (10): McCabe-Thiele diagram for the stripping of zirconium from loaded Aliquat 336.

Characterization of ZrO2 product

As shown in Figure (11-a) The ESEM-EDX for zirconia sample shows the zirconium oxide. In addition, Figure (11-b) shows that the particle size of the formed zirconia from solvent extraction using Aliquat 336.

Analysis of ZrO2 using ICP (Inductively Coupled Plasma)

Zirconia sample was analyzed for impurities using ICP and the results show the formation of pure zirconia using Aliquat 336, Thus the concentration of HfO_2 after solvent extraction became 105ppm, SiO_2 was found to be 99ppm, Al_2O_3 76ppm, TiO_2 48ppm, Fe_2O_3 880ppm, MgO 23ppm, CaO 23ppm and P_2O_5 87ppm. It is clear that all the procedures used in preparation of zirconium nitrate solution then solvent extraction Aliquat 336 gave zirconium oxide approximately free of all other metals like HfO_2, SiO_2, Al_2O_3, TiO_2, Fe_2O_3, MgO, CaO and P_2O_5.

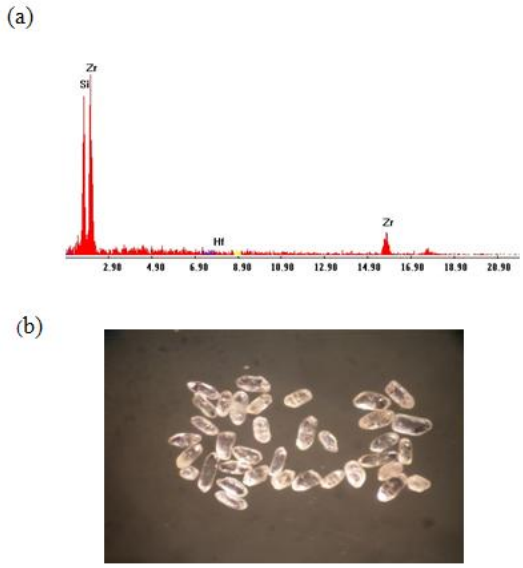

Figure (11): Characterization of the as-prepared ZrO_2 using (a) ESEM-EDX, (b) Microscopic.

IV. CONCLUSIONS

The extraction and separation of zirconium using Aliquat 336was studied with respect to various factors. These factors were the effect of Aliquat 336 concentration, contact time, diluents used, phase ratio (O: A) v: v and HNO_3 concentration. It was found that the optimum conditions for extraction of zirconium from the nitric medium were 6% Aliquat 336 in kerosene and 10 minutes contact time at phase ratio (2:1). The obtained

results also show that the extraction efficiency for Zr^{+4} at these optimum conditions was 99.6%. Additionally, Decanol was chosen to be the best modifier for solving third phase problem.

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