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TBP-Impregnated Siplite LX-16 Polymeric Resin for Thorium Extraction from Nitrate Medium, Part A: Preparation and Extraction Performance

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ABSTRACT

Impregnation of tri butyl phosphate (TBP) solvent into polymeric resin, commercially known as Siplite LX-16, using the two commonly used dry and wet methods were applied. The suitable conditions for wet impregnation were 70% solvent concentration in kerosene with solvent/resin (S/R) ratio of 6.0 and left 24 hours for impregnation at room temperature before separation and washing with hot water then drying overnight at a temperature of 50°C, where at these conditions the resin impregnated with 1.99 g TBP/g resin. In case of dry impregnation requires solvent concentration was 55% in acetone at S/R ratio of 5.0 and shaking for 4.0 h then left in the air till complete volatilization of acetone and finally drying overnight at a temperature of 50°C, where at these conditions one resin impregnated with 2.057 g TBP. Results of testing performance of the prepared TBP impregnated Siplite LX-16 resin against thorium extraction from nitrate medium reveal that extraction efficiency of 70.7% was achieved after 30 min. which can be enough to attain equilibrium. Thorium extraction from the nitrate medium requires high free acidity rather than nitrate from salts (salting out agent addition), however, for economical consideration and reagent saving, it can practically conduct the extraction at free normality of 3.0 N at which 62% extraction efficiency could be achieved at the condition of initial thorium concentration of 2.0 g/l, aqueous/resin "A/R" ratio of 25 and the phases contacted for 30 min. The calculated theoretical saturation capacity for the prepared TBP impregnated Siplite LX-16 resin was 292.7 mg Th / g impregnated resin which is considered high capacity, so it was recommended to use solutions containing high levels of thorium concentrations reached to several grams per liter to decrease the treated volumes at shorter time as well as verify almost complete saturation.

1- INTRODUCTION

Synthesis of selective ion-exchange resins is complex, time-consuming, and costly, so there are only a few notable large-scale industrial applications for the separation of metal ions from solutions. However, development of particulate adsorber's so-called solvent-impregnated resins (SIRs) renders them applicable to a wide range of potential liquid–solid separation and recovery of metal ions processes. These solvent impregnated resins possess a combination of the unique advantageous features of both liquid–liquid extraction and ion exchange resin. Accordingly, solvent impregnated resins (SIRs) can be considered as an

alternative adsorbent material since they are similarly capable of selective sorption. The attractiveness of SIR lies in the possibility to solve immediate pressing problems concerning the mostly commercial used solvents [1].

Solvent impregnated resins are relatively easy to prepare where is based on the incorporation of liquid organic solvent into a porous nonionic polymer matrix by a physical impregnation technique. Accordingly, synthesis of SIRs is relatively simple, versatile, and possible to obtain a whole range of materials tailored for particular ions in solution using various combinations of polymeric supports and ionic liquid extractants [1 – 3].

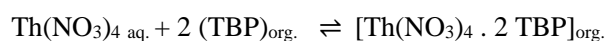
There are two main methods for impregnation namely wet and dry, where the wet method was essential for hydrophobic extractants, while the dry impregnation method was effective in the case of amphiphilic extractants. Dry impregnation may suffer from wetting problem of the polymeric support resin, while wet impregnation can overcome this problem. Also, wet method can be applied directly for the industrial scale through impregnation for the polymeric resin is a dynamic column that has the advantage of high impregnation efficiency in a short time. It is worthy to mention that, polymeric resin support requires pretreatment before the impregnation step to remove any inorganics and organic impurities from the resin beads to ensure an active surface during adsorption of the required impregnating solvent [4, 5].

Solvent stabilization onto a polymeric resin after impregnation aims to prevention or almost minimize the solvent leakage from the polymeric support, this leakage depends principally on the used support type and nature of the retained organic solvent as well as the extraction system itself. However, there are two main and simple methods proposed for the solvent stabilization into the freshly prepared impregnated resin; namely heat treatment and conditioning where they aid to orientation and re-ordering of the initially disordered solvent layer into a more compact and regular one which led to tightening of the layer to structure of the resin beads. Heat treatment (also known as wet drying) method involves keeping the freshly prepared impregnated resin in boiling water for a period of several hours. However, the conditioning method for the prepared solvent impregnated resin was carried out through several conventional ion exchange cycles [6, 7].

Solvent impregnated resins (SIRs) can be used technically in column systems like the conventional ion exchange resins, so any separation problem due to theoretical number of plates can be solved through elongation of the column length. However, the solvent impregnated resin is simply filled into columns of a suitable size and the aqueous phase to be handled is run through this resin bed. Because of the low specific gravity of the SIR resins, it will float and do not undergo appreciable volume changes during loading and elution, so the free space in the upper part of the column, which is a common practice with ion exchange resin, will not be required, also the typical advantages of fluidized bed technology could be utilized [8]. When the extraction system is working in a small column size, it would be advisable to use two or three columns in sequence and using up flow loading, but large extraction columns

systems can be used either up flow or down flow mode. However, the elution process can be achieved preferably by the down flow.

Thorium separation from rare earth elements and other metals was achieved selectively through a solvent extraction process using a neutral organophosphorus such as tributyl phosphate (TBP) [9]. This process has been practiced worldwide on commercial scale. Thorium extraction requires a higher TBP concentration and the extraction rates increase with increasing nitric acid molarity where concentration higher than about 2.0 M is practiced. In this regard, tetra valent thorium forming neutral species of thorium nitrate $[\text{Th}(\text{NO}_3)_4]$ in nitric acid medium and the extraction was occurred by solvation with two molecules of tributyl phosphate as $\text{Th}(\text{NO}_3)_4 \cdot 2\text{TBP}$ according to the following equation [10]:



The solvent Tri-n-butyl phosphate (TBP) impregnated polymeric resin shows a lower solubility value in water as well as in nitric acid than the traditional liquid solvent. In this regard, the solubility of free TBP in demineralized water is 0.38 g/l, while its solubility if impregnated into the resin varies from an undetectable amount (0.02 g/ l) to 0.2 g/ l of water ⁽¹¹⁾. Extraction - elution behavior of the solvent-impregnated resin with TBP is good where the decrease in capacity during extraction from nitric acid medium amounts to less than 5%. Also, the mean value of TBP losses due to both solubility and degradation to dibutyl phosphate was found to attain 0.163 g TBP/l solution. On the other hand, the only degradation product from tri-n-butyl phosphate during extraction from nitric acid medium is dibutyl phosphoric acid (HDBP) which is easily washed off by water. So, build- up of this degradation product is considered negligible due to the steady leaching during the elution operation [12].

2- EXPERIMENTAL

The chemicals and reagents used to conduct this work are analytical grade for resin and solvent pretreatments also for the analysis procedures while double distilled water is used. Drying oven model KOTIERMANN and weighing balance model Sartorius, TE2145 are used in drying and weighing the prepared impregnated resin.

UV/VIS spectrophotometer, multi positions, double beam model T 80 version 24-1884-010001, with glass cell of 10 mm was used for thorium determination. Thorium was determined using 0.1% Thoron (I) reagent according to the method described in Marczenko (1986) [13].

The two impregnation methods were initially tested to choose the most efficient, convenient, and easy in application as well as achieving maximum impregnation for the pretreated non-functional Siplite LX-16 polymeric resin with the pretreated tributyl phosphate (TBP) solvent. The diluent that used for solvent dilution were kerosene in case of wet impregnation and acetone in case of dry method. The main factors affecting the impregnation as initial solvent concentration, solvent-to-resin "S/R" ratio, drying temperature and impregnation contact time were studied.

Performance of the prepared tributyl phosphate (TBP) impregnated Siplite LX-16 resin under the suitable conditions was conducted against thorium extraction from nitrate medium. The main factors affecting the extraction step for thorium from nitric acid medium till attain equilibrium were studied involving contact time, free nitric acid normality, initial thorium concentration, salting out agent addition and solution to impregnated resin ratio.

3- RESULTS AND DISCUSSION

Results of studying the major factors affecting both wet and dry impregnation methods for TBP solvent into Siplite LX-16 polymeric resin are herein discussed.

3.1. Results of Studying Factors Affecting Wet and Dry Impregnation

3.1.1 Solvent Concentration Effect

Effect of initial Tributyl phosphate (TBP) concentration on impregnation of the polymeric resin Siplite LX-16 using the two methods was studied to identify the suitable solvent concentration that verify maximum impregnation for the resin under study. The study was carried out using different concentrations of TBP diluted in kerosene (in case of wet impregnation) ranging from 10 to 90% where solvent and resin "S/R" phases were contacted in ratio of 5.0, where 1.0 g of the pretreated dry resin is used in each test, and left 24 hours for impregnation at room temperature before separation and washing with hot water and finally drying overnight at 70°C.

In case of dry impregnation, the concentrations of TBP diluted in acetone ranging from 10 to 80% was used in ratio to the resin "S/R" of 5.0 where the phases were contacted with hand shaking each 30 min. for 4.0 h then left in air till complete volatilization of the surficial acetone that assured with free moving of the

resin. The impregnated resin samples were then dried overnight in an oven at 50°C to ensure complete volatilization of the acetone inside the resin grains.

The impregnated resin samples with the two impregnation methods were weighed to determine the total weight of the impregnated resin with the solvent, where results of this weight in grams were represented against the percent of initial tributyl phosphate (TBP) concentration in figures (1, 2) for the wet and dry impregnation respectively.

Results of wet impregnation (Fig. 1) indicated that the impregnated resin weight was increased gradually from 1.34 g at the initial solvent concentration of 10% to reach its maximum value of 2.76 g at the solvent concentration of 90%. It is worthy to mention that, due to insufficient dilution for the solvent of concentration 90%, the resultant impregnated resin aggregates in the form of clusters which reflect inefficient washing during separation of the excess solvent of relatively high viscosity. Accordingly, the solvent concentration of 70% was chosen for studying the subsequent factor of solvent/resin ratio which can be compensate the increase in solvent concentration.

The data from studying dry impregnation (Fig. 2) reveal also that the weight of the impregnated resin increased with increasing the initial solvent concentration where it was 1.47 g at the concentration of 10% and reached to 3.165 g at the concentration of 70%.

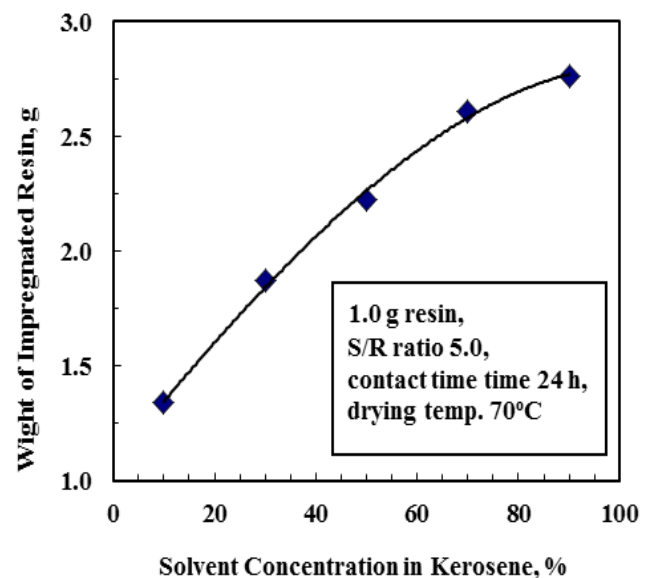


Fig. (1): Effect of Solvent Concentration on Wet Impregnation

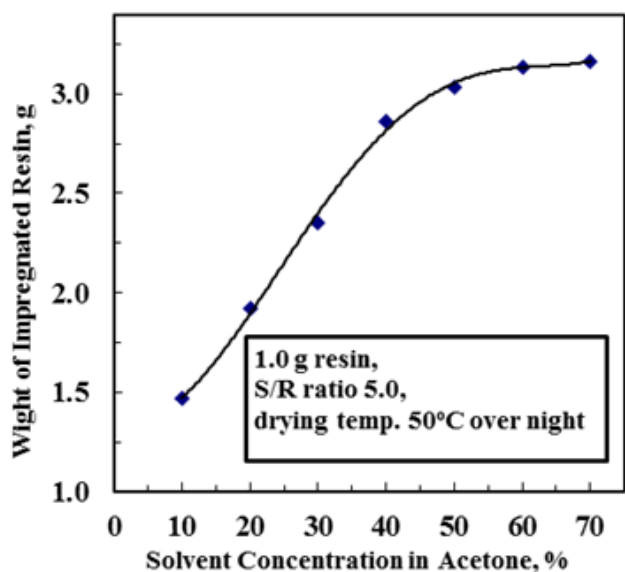


Fig. (2): Effect of Solvent Concentration on Dry Impregnation

Comparing the result with that of wet method we can indicate that impregnated resin with this dry method containing more solvent in spite of using lower concentration of the solvent. However, due to impregnation appreciable amount of kerosene diluent with the solvent tributyl phosphate (TBP) in case of the wet method ⁽¹⁴⁾, so the practical effective solvent in extraction process will be rather small. It is also obvious that the impregnated resin with solvent of concentration more than 50% seems to be have an excess of solvent that shown present free on the resin surface which led the resin grains to aggregate in the form of clusters. Accordingly, an initial solvent concentration in the range of 50% to 55% seems to be suitable to achieve the required maximum impregnation for the polymeric resin with the dry method where this close range was further studied. Results of studying this close range of solvent concentration, which are conducted at the same previous conditions, were tabulated in Table (1). These results indicate that the resin can be impregnated efficiently with the solvent till

55% TBP initial concentration and the impregnated resin was 3.05 gram at this solvent concentration without appearance excess of solvent.

3.1.2 Solvent to Resin (S/R) Ratio Effect

Effect of solvent to resin (S/R) ratio was studied also to verify maximum impregnation for Siplite LX-16 polymeric resin at lower concentration of solvent, 70% in kerosene and 55% in acetone for the wet and dry impregnation respectively, to avoid the previously observed problem at the higher solvent concentration. The studied ratios were in the range of 4.5 to 6.5 through increments of 0.5 between each ratio. The other conditions applied in the previous factor were concerned in the two methods wet and dry.

The total weight of the impregnated resin with the solvent were determined, where results of this weight in grams were related with the ratio of TBP to Siplite LX-16 polymeric resin (S/R) in Figures (3, 4) for the wet and dry impregnation respectively.

Results of studying wet impregnation (Fig. 3) show that the maximum weight of the impregnated resin of about 2.69 grams was achieved at the S/R ratio of 6.0 where there is slight increase in the weight over this ratio. However, increase in the impregnated resin weight from ratio 5.0 to 6.0 was about 3% without any problem, so increasing S/R ratio can be efficiently compensating the decrease in TBP concentration. With respect to the results of dry impregnation Fig. (4) it is clear that there is increase in the weight from the S/R ratio 4.5 to 6.5 where the weight was an increased from 3.0 g at the ratio 4.5 and reached to 3.168 g at the ratio 6.5. However, at ratio more than 5.0 the resultant impregnated resin was aggregated in the form of clusters which reflect presence of an excess solvent. Accordingly, the ratio of 5.0 can be chosen as a minimum ratio to ensure good wetting between resin and solvent as well as solvent distribution into the resin beads.

Table (1): Results of Studying Close Range of Solvent Concentration in Dry impregnation

TBP conc. in acetone, %	50	51	52	53	54	55
weight of impregnated resin, g	3.035	3.0354	3.0357	3.04	3.046	3.05

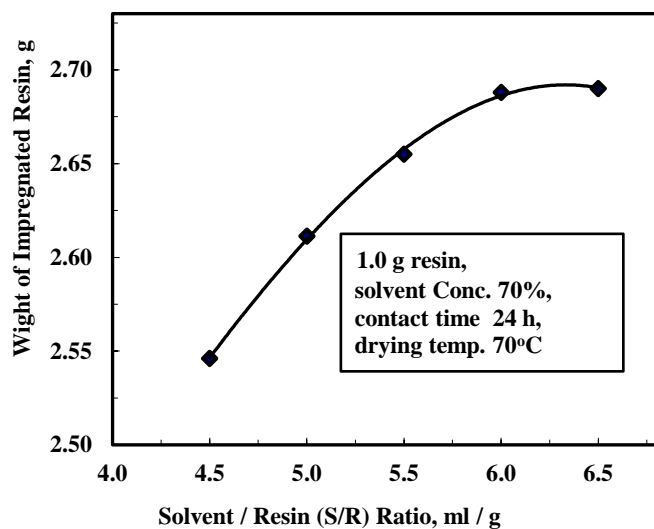


Fig. (3): Effect of Solvent / Resin Ratio on Wet Impregnation

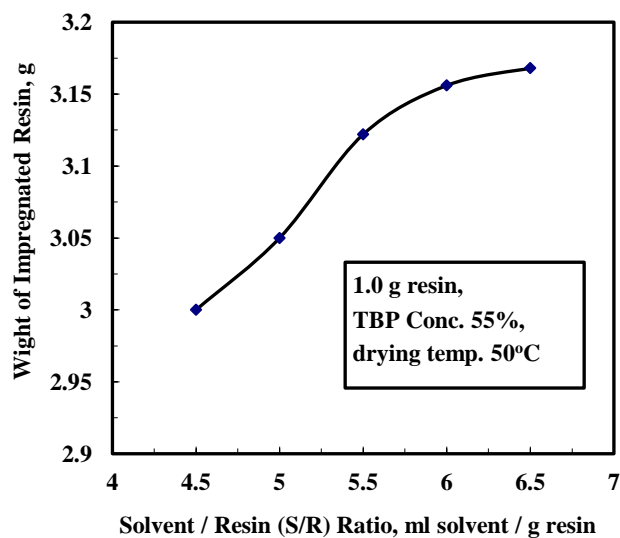


Fig. (4): Effect of Solvent / Resin Ratio on Dry Impregnation

3.1.3 Drying Temperature Effect

The applied temperature during the drying step was studied to know its effect upon the wet and dry impregnation where it was varied from 50 to 70°C through 5°C increase between each test. The conditions that prevailed during the wet impregnation were 70% TBP concentration in kerosene at a or the ratio to the polymeric resin of 6.0 and the S/R phases contacted for impregnation to 24 hours at room temperature before separation, washing with hot water and drying overnight at the selected tested temperature then weighing. The conditions of dry impregnation were 55% TBP

concentration in acetone at a ratio to the resin of 5.0 during the dry impregnation and the phases contacted for impregnation to 4.0 hours at room temperature before the drying overnight at the selected tested temperature then weighing.

The obtained results illustrated in Figures (5, 6) which reveal that dramatic decrease in the weight of the impregnated resin from 50°C to 70°C, where this decrease equivalent to 11.5% in the case of wet impregnation and about 8% in case of dry impregnation. So, it was recommended to accomplish the drying step at relatively low temperature of 50°C to prevent the loss of solvent through vaporization and to attain maximum impregnation for the resin.

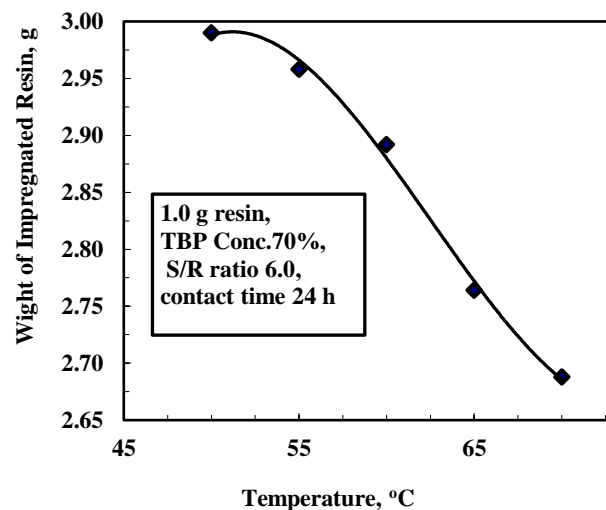


Fig. (5): Effect of Drying Temperature upon Wet Resin Impregnation

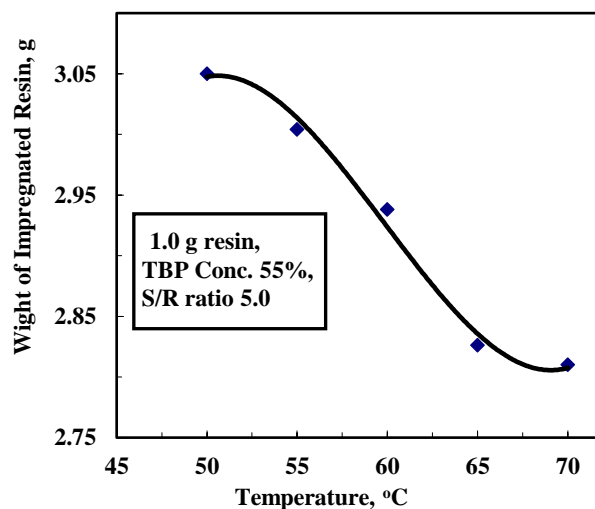


Fig. (6): Effect of Drying Temperature on Dry Impregnation

3.1.4 Contact Time Effect

The effect of impregnation contact time at ambient temperature, before the drying step, was studied upon 1.0 g of the polymeric resin to identify the minimum time that the resin left to impregnate and verify maximum level with TBP solvent. The experiments were conducted under constant conditions of 70% TBP concentration at S/R ratio 6.0 in case of wet impregnation and 55% TBP concentration at S/R ratio of 5.0 in case of dry impregnation and the drying temperature of 50°C was applied. The studied impregnation contact time in the range of 4.0 to 24 h through increments of 4.0 h between each test and in the range of 2.0 to 10 h through increments of 2 h between each test were carried out in case of wet and dry impregnation respectively.

Results of studying this factor were shown in Figures (7, 8) from which it is obvious that in case of wet impregnation Fig. (7) appreciable impregnation was occurred after 4.0 h where the impregnated resin weigh more than 2.65 g. However, the impregnation increased slowly until 16 h where the increase is just 2.75% while this increase from 16 to 24 h was 9.6% that verify maximum level near 3.0 g for the impregnated resin. Results of studying dry impregnation Fig. (8) reveal that there is no appreciable impregnation occurred all over the tested contact times. In this regard, the increase in resin weight from contact time 2.0 to 10 h was only 0.008 g which equivalent to 0.26%. Accordingly, lifting the sample to impregnate till almost volatilization of the diluent before drying at any time may be enough for impregnation. However, we can avoid forced volatilization of the diluent through direct drying where slow volatilization of the diluent aid in orientation of the solvent into a more compact and regular layers which led to good tightening on the resin bead's structure.

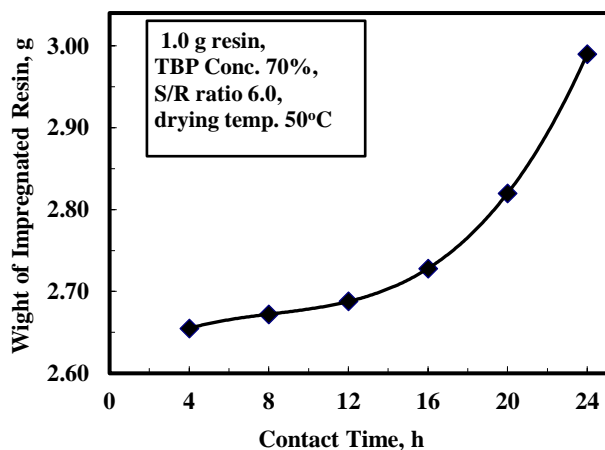


Fig. (7): Effect of Contact Time on Wet Impregnation (before Drying)

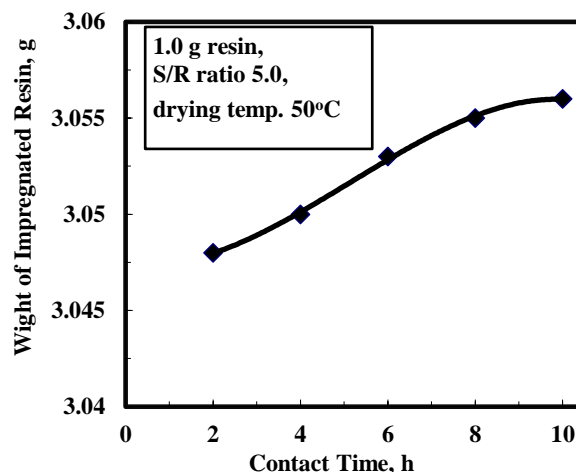


Fig. (8): Effect of Contact Time on Dry Impregnation (before Drying)

3.2 Preparation of Stock TBP-Siplite LX-16 Resin for Thorium Extraction Studies

In spite of wet impregnated method proves some advantages over that of dry, the dry method was chosen for preparation of the stock TBP Impregnated Resin sample for conducting thorium extraction experiments in lab scale for its simplicity, but if column system is used for extraction in pilot scale wet impregnated method is the preferred as previously mentioned [4, 5]. The suitable conditions of preparation sample by the dry method of impregnation were applied upon 25 g of the pretreated resin where solvent concentration of 55% in acetone was used in ratio to the resin of 5.0. The phases were contacted with hand shaking each 30 min. for 4.0 h then left in the air till complete volatilization of the surficial acetone that assured with free moving of the resin then the impregnated resin samples were then dried at 50°C overnight in an oven. The resultant TBP impregnated resin sample weighing 73.225 g which reveal that the impregnation was 1.929 g TBP/g resin, so the calculated theoretical capacity for thorium extraction was 292.7 milligram thorium per gram impregnated resin.

3.3 Results of Equilibrium Batch Studies for Thorium Extraction from Nitrate Medium using Tributyl Phosphate Impregnated Siplite LX-16 Resin

Results of experimental work that conducted to test performance of the prepared tributyl phosphate (TBP) impregnated Siplite LX-16 resin against thorium extraction from nitrate medium will be herein discussed. The main studied factors affecting the extraction step for thorium from nitric acid medium till attain equilibrium involve contact time, free nitric acid normality, salting

out agent addition, initial thorium concentration, and solution to impregnated resin ratio, however, 1.0 gram impregnated resin was used in each set of experiments.

3.3.1. Extraction Contact Time

Contact time determines the rate of extraction and its kinetics, so it was firstly studied in wide range from 1.0 to 60 min. under constant of the other factor as initial thorium concentration of 1.0 g/l dissolved in 3.0 N nitric acid and added in ratio of 25 to the tested TBP impregnated Siplite LX-16 resin and this ratio will be represented as Aqueous/Resin "A/R" ratio.

Results of studying this factor are illustrated in Fig. (9) which related each contact time with the corresponding thorium extraction efficiency. The results reveal that the extraction is proceeding rapidly where more than 18% extraction efficiency was achieved at the first minute and this efficiency was steadily increased to reach equilibrium after 60 min where the efficiency was 73.15%. However, an extraction efficiency of 70.7% was achieved after 30 min. which can be enough to attain equilibrium.

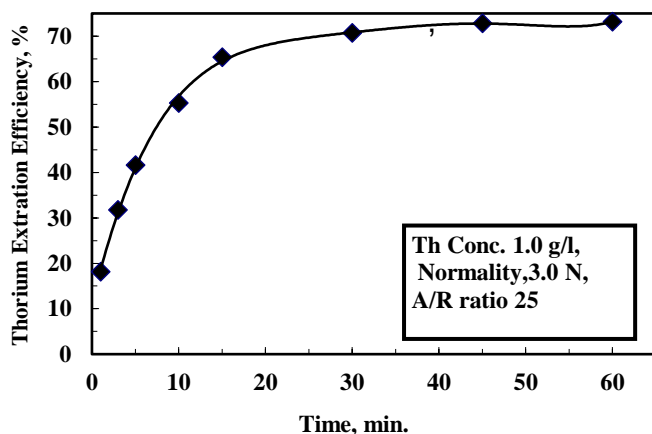


Fig. (9): Effect of Contact Time on Thorium Extraction Efficiency

3.3.2. Effect of Free Nitric Acid Normality

The medium-free normality of nitric acid has a greater effect on the extraction of thorium where their effect includes the overall efficiency as well as the co-extracted impurities especially cerium, so it was studied. This important factor was studied using TBP-impregnated resin where free nitric acid normality in the range from 1.0 to 9.0 N, through 2.0 N increment between each normality, was tested under the conditions of an initial thorium concentration of 2.0 g/l, Aqueous/Resin "A/R" ratio 25 and the phases contacted for 30 min.

Results of study this factor are indicated in Fig. (10) which revealed that the extraction efficiency of thorium increased steadily with increasing the medium nitric acid normality from 1.0 to 9.0 N. In this regard, thorium extraction efficiency increased from 48% at the normality of 1.0 and reached its maximum value of 92% at the normality of 9.0. However, for economical consideration and reagent saving, it can practically conduct thorium extraction at normality in the range of 1.0 to 5.0 N, however, normality of 3.0 N seems to be an intermediate value to conduct the extraction efficiently and economically where 62% extraction efficiency could be achieved.

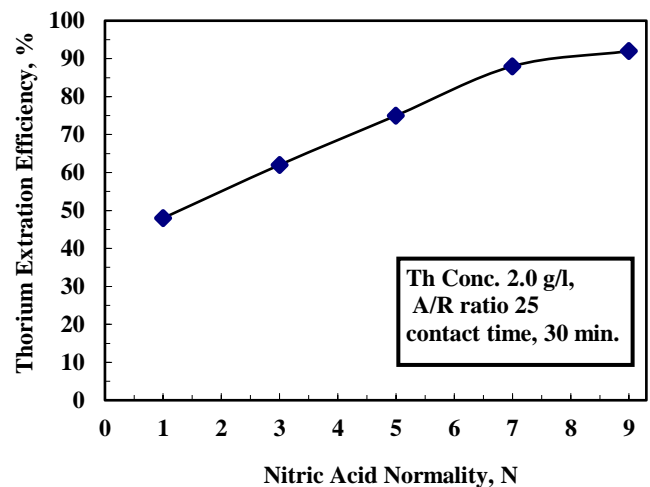


Fig. (10): Effect of Nitric Acid Normality on Thorium Extraction Efficiency

3.3.3. Effect of Salting out Agent Addition

Salting out agent may have an effect upon the improvement of thorium extraction efficiency, so it was investigated for studying its addition under the conditions of total free nitrate of 4.0 molar, where ammonium nitrate was used in the test as salting out agent. The other constant prevailed conditions include thorium concentration of 1.0 g/l, in the required salting out agent molarity and acid normality, added in a ratio 25 to TBP impregnated resin then the phases contacted for 30 min.

The results of studying this factor were illustrated in Fig. (11) from which it is clear that the salting out agent has a reverse effect where thorium extraction efficiency decreased with increasing the salting out-agent concentration. In this regard, thorium extraction efficiency decreased from about 85% at the concentration 0.5 M NH_4NO_3 to reach as low as about 74% at the concentration 3.5 M NH_4NO_3 which means 11% decrease in thorium extraction efficiency. This is assured that the extraction requires high molarity of nitric acid rather than

nitrate salt. Accordingly, it was recommended to conduct the extraction without salting out agent addition

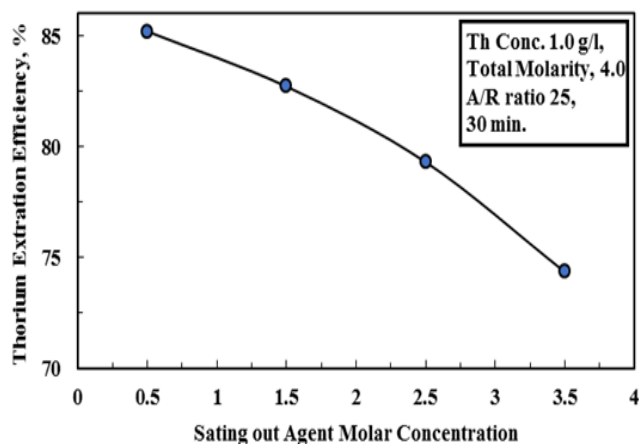


Fig. (11): Effect of Addition Salting out Agent on Thorium Extraction Efficiency

3.3.4. Effect of Initial Thorium Concentration on Impregnated Resin Uptake

Initial thorium concentration effect on impregnated resin uptake was also studied using the prepared TBP impregnated Siplite LX-16 resin under the conditions of contacting the solution of 3.0 N nitric acid with the resin at an Aqueous/Resin "A/R" ratio of 25 for 30 min., where thorium concentrations in the range of 0.5 - 2.0 g/l were studied.

The results represented as thorium uptake mg Th/g of TBP impregnated resin are shown in Fig. (12) which indicate that the uptake was sharply increased from 11.25 to 31 mg Th/g impregnated resin when the initial feed concentration of thorium increased from 0.5 to 2.0 g/l.

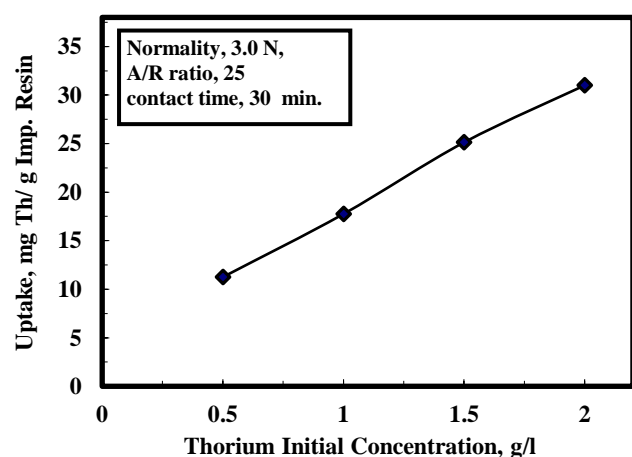
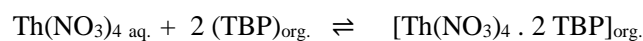


Fig. (12): Effect of Thorium Initial Concentration on Uptake the Impregnated Resin

So, it was recommended to conduct the extraction with high levels of thorium concentrations. It is worthy to mention that the calculated theoretical saturation

capacity for the prepared TBP-impregnated Siplite LX-16 resin was high as 292.7 milligram thorium per gram of the impregnated resin according to the following equation



3.3.5. Effect of Aqueous/Impregnated Resin "A/R" ratio on Thorium Uptake

The aqueous to impregnated resin "A/R" ratio was studied under the conditions of thorium concentration of 1.0 g/l with nitric acid free normality of 3.0 N and the phases were contacted for 30 min. The results of studying this effect were indicated in Fig. (13) from which it is clear that the uptake of TBP impregnated resin was increased with increasing the pregnant solution ratio from 13.6 mg Th/g impregnated resin at the ratio 15 to reach 20.8 at the ratio 45, however, this increase was relatively low because Th concentration is relatively low. Taking into consideration the high capacity of the TBP-impregnated Siplite LX-16 resin as previously mentioned, so if this resin is used for thorium extraction, it was recommended to use solutions containing high levels of thorium concentrations reached to several grams per liter to decrease the treated volumes at shorter time as well as verify almost complete saturation for the TBP impregnated Siplite LX-16 resin.

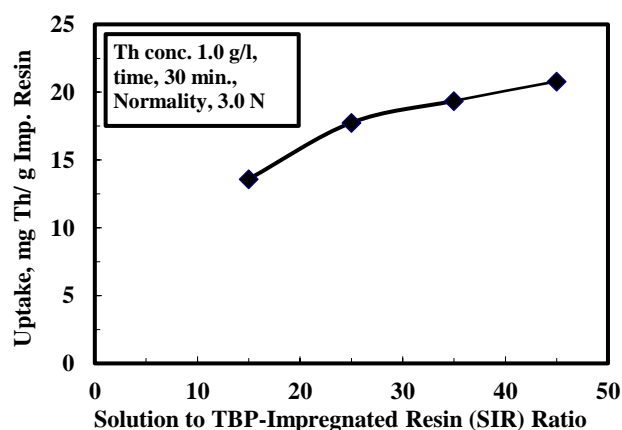


Fig. (13): Effect of Solution to TBP-Impregnated Resin (SIR) Ratio on Thorium Extraction Efficiency

4- CONCLUSION

Impregnation of tri butyl phosphate (TBP) solvent into Siplite LX-16 polymeric resin, using the two commonly used dry and wet methods were applied. Dry impregnation requires less solvent (55% in acetone) and less (S/R) ratio than that of wet impregnation that requires 70% solvent concentration in kerosene with (S/R) of 6.0 while the drying temp. was 50°C overnight for both methods, where at these conditions the resin was

impregnated with 2.057 g and 1.99 g TBP/g resin for the two methods respectively. This means that dry impregnation verifies more impregnation with the solvent in addition to the simplicity in preparation. However, wet impregnation method can be applied in the case of applications in industrial scale through impregnation for the polymeric resin is a dynamic column that has the advantages of high impregnation efficiency in a short time.

Results of testing performance of the prepared TBP-impregnated Siplite LX-16 resin against thorium extraction from nitrate medium reveal that extraction thorium extraction from the nitrate medium requires high free acidity rather than nitrate from salts (salting out agent addition). However, for economical consideration and reagent saving, it can practically conduct the extraction at free normality of 3.0 N at which 62% extraction efficiency could be achieved at the condition of initial thorium concentration of 2.0 g/l, aqueous/resin "A/R" ratio of 25 and the phases contacted for 30 min. The calculated theoretical saturation capacity for the prepared TBP-impregnated Siplite LX-16 resin was 292.7 mg Th/g impregnated resin which is considered high capacity, so it was recommended to use solutions containing high levels of thorium concentrations reached to several grams per liter to decrease the treated volumes at shorter time as well as verify almost complete saturation.

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