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Removal of dissolved tri-*n*-butyl phosphate from aqueous nitric acid solutions: kinetic studies

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ABSTRACT

The present work deals with the kinetic study for the removal of dissolved TBP from aqueous nitric acid solutions using Normal Paraffin Hydrocarbon (NPH) as the solvent. Effect of different parameters like type of solvent, concentration of organic phase, concentration of aqueous phase, organic to aqueous phase (O/A) ratio and temperature on the extraction of TBP, has been successfully optimized. Around 62% of dissolved TBP was extracted from aqueous feed solution containing 0.3 M HNO_3 with 3% TBP in NPH as the solvent and O/A ratio of 1:1 in only 5 min at 35°C. The graphical representation of the kinetic expressions for different parameters predicted that the extraction of TBP follows the first-order behaviour. The rate constants values obtained from the first-order equation have also verified the optimized parameters. This study will provide an aid in development of a robust and effective process for removal of dissolved TBP with minimum waste generation.

Keywords: TBP; Nitric acid; NPH; Kinetic study; Rate constants

1. Introduction

PUREX process deals with the extraction of Uranium and Plutonium from feed solution obtained from dissolution of spent nuclear fuel in nitric acid. 30% tri-*n*-butyl phosphate (TBP) diluted with dodecane is widely used solvent in PUREX process. During the extraction process, a small amount of TBP also gets transferred into the aqueous solution due to limited solubility of TBP in nitric acid. The concentration of TBP in this aqueous waste is approximately 200 ppm depending upon the concentration of nitric acid. This dissolved TBP in aqueous nitric acid solutions even at such lower concentrations can lead to many exothermic reactions causing explosive violence at elevated temperature. Many such type of red oil explosions have been reported in the past [1]. Thus, removal of this dissolved TBP from aqueous solution is necessary before it is discharged. Extraction of TBP by suitable solvent can be the best technique due to well documented limitations of other separation techniques such as steam stripping and adsorption. High boiling point of TBP makes steam stripping economically unfeasible, while adsorption process leads to generation of secondary waste for disposal [2]. However, very few papers are available in literature reporting kinetics and extraction of TBP and related systems. Kinetic study helps in understanding various factors which affect the extraction rate. The extraction is triggered if carried out at optimized conditions

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stated by kinetic study. It makes the process economical by saving time, money and energy. The batch kinetic extraction studies for TBP-diluent-nitric acid system will not only help in selection of an appropriate solvent but also facilitates the extraction process by reducing the solvent volume and time. Hence, proper removal of dissolved TBP from aqueous feed will be possible before its disposal for environmental safety.

Naito and Suzuki have explained mechanism for the extraction of several uranyl salts (nitrate, chloride, perchlorate, sulphate, acetate and phosphate) by TBP. The formation of the complexes $UO_2(NO_3)_2(TBP)_2$, $UO_2Cl_2(TBP)_2$, $UO_2(OAc)_2(TBP)_2$ and $UO_2(ClO_4)_2(TBP)_4$ was confirmed by partition and infrared study [3]. Baumgartner and Finsterwalder have determined the transfer rates of U(IV) and Pu(IV) between aqueous nitric acid and TBP dissolved in dodecane using single drop method as the experimental technique. The results of kinetic measurements have been interpreted in terms of chemical interface reactions [4]. Hoh and Wang have performed rate studies for optimizing conditions for extraction and stripping of nitric acid for nitric acid–TBP system [5]. Sovilj et al. have carried out studies to extract nitric acid using TBP in kerosene as solvent in the concentration regions 2–6 kmol m^{-3} of nitric acid and 0.5–2 kmol m^{-3} of TBP [6]. Lawson and Hughes have developed a kinetic model for the extraction of nitric acid by TBP in kerosene using rotating disc cell technique [7]. Wang et al. have made investigation on the extraction kinetics of palladium with bis (2-ethylhexyl) sulphoxide (DEHSO) from hydrochloric acid solution by the use of a constant interfacial area cell, which permits continuous and automatic measurements [8]. Nabheshima has studied interphase transfer kinetics of neodymium and nitric acid using a single drop column with recycling organic phase via an external mixing vessel in H_2O-HNO_3/$NaNO_3$-$Nd(NO_3)_3$-TBP system [9]. Kopecni and Petkovic have developed chemical model on the equilibrium constant expression for a solvent-extraction reaction for the extraction of uranyl nitrate with TBP [10]. Tochiyama et al. have studied kinetics for the redox reaction of Np (IV) catalyzed by nitrous acid using TBP in dodecane as an extractant. The rate of the reaction was investigated by measuring the rate of the extraction of initially present Np(V) [11]. Xianghong and Zhou have investigated the extraction kinetics of nitric acid by TBP in heptane in an improved Lewis cell. The activity of component was calculated by Pitzeris equation for aqueous phase and UNIFAC model for organic phase [12]. Huang and Huang have utilized a transfer cell of constant interfacial area to measure initial rates of the extraction and the stripping with aqueous phases containing 0.1114 mol/dm^3 of nitric acid and up to 0.0267 mol/dm^3 of uranium and kerosene diluted organic phases containing 0.01265–0.0894 mol/dm^3 of dimeric HDEHP and up to 0.00963 mol/dm^3 of uranium [13]. Marchenko et al. have published data on the kinetics of oxidation and reduction of U, Pu and Np ions with various reagents (HNO_3, HNO_2, U(IV), etc.) in organic solutions of TBP. The rate equations, rate constants and thermodynamic functions of activation of the reactions were presented [14]. Yadav et al. have studied kinetics of the mass transfer processes involved in uranium recovery from wet process phosphoric acid by solvent extraction employing synergistic extractants. The kinetics of separation of entrained solvent by diluent washing and uranium mass transfer behaviour were discussed [15]. Smitha et al. have carried out accelerating rate calorimetry studies on the TBP and nitric acid reactions at various acid concentrations which revealed that the conditions for red oil formation may vary with acid strength [16].

Most of the investigators have mainly focussed on extraction of U, Pu and Np using TBP and similar solvents as extractant and have developed kinetic model for the process. Reports on kinetics for removal of dissolved TBP from nitric acid solution are not yet available. Thus, it is necessary to study kinetics of TBP–nitric acid system for efficient TBP removal. This study will provide an aid in deciding the appropriate solvent and time required for maximum removal of dissolved TBP from aqueous nitric acid solutions.

The main aim of this work is to perform kinetic study for the extraction of TBP for nitric acid solution. The kinetic study has been carried out using liquid–liquid extraction process which involves mass transfer of TBP from aqueous nitric acid phase to organic solvent phase. The effect of different parameters like type of solvent, concentration of organic phase, concentration of aqueous phase, organic to aqueous phase (O/A) ratio and temperature on the extraction of TBP has been studied. The kinetic data have also been expressed graphically to find the order of extraction and to calculate the rate constants.

2. Experimental

2.1. Reagents

TBP, nitric acid, Normal Paraffin Hydrocarbon (NPH) and dodecane were supplied by Prabhat chemicals, Mumbai, India. Their purity was around 97, 70 and 95%, respectively. Acetonitrile, odourless kerosene and dodecane were purchased from SD fine chemicals, Mumbai, India. All the reagents used were of analytical grade.

2.2. Kinetic study

The kinetics for removal of dissolved TBP from nitric acid solutions was carried out in a 500 mL baffled batch reactor made up of glass equipped with a six bladed (pitched blade) glass turbine for agitation. The entire assembly was kept in water bath for temperature control during extraction. Feed solution for extraction was TBP dissolved in nitric acid which was taken in a glass reactor as the aqueous phase. Solvent used for extracting TBP from the feed constituted the organic phase of the extraction process. The mixture was then agitated at 1,000 rpm for around 8 min for each study. Samples were withdrawn regularly at different intervals and then analysed for TBP using High-Performance Liquid Chromatography (HPLC). Different parameters affecting the extraction such as type of solvent, concentration of organic phase, concentration of aqueous phase, organic to aqueous phase (O/A) ratio and temperature have been optimized for maximum extraction of TBP by organic solvent.

2.3. Analysis of TBP

The quantification of the dissolved TBP in nitric acid was done on HPLC as described earlier [17]. All the samples were analysed three times to check the reproducibility of the results. The uncertainty in solubility values obtained was precise within ±2%.

2.4. Analysis of kinetics data

The different parameters affecting the kinetics of TBP removal were analysed graphically to obtain the rate constant of extraction. The kinetic expression for all parameters was found to be fitted well as per first-order reaction model which is given as follows:

$$\ln\left(\frac{C_o}{C_{At}}\right) = k_{obs}t \tag{1}$$

where C_o is the initial concentration of TBP in the feed, C_{At} is TBP concentration in the raffinate at time t, k_{obs} is an observed first-order rate constant and t is the extraction time.

The values for rate constants for each parameter were obtained from the slope of the graph of ln (C_o/C_{At}) against t which was a straight line.

3. Results and discussion

The effect of different parameters such type of solvent, concentration of organic phase, concentration of aqueous phase, organic to aqueous phase (O/A) ratio and temperature has been optimized for efficient TBP removal from the aqueous nitric acid solutions and rate constants have been calculated for each studied parameter.

3.1. Effect of type of solvent

Solvent plays a very important role in the extraction and the rate of extraction depends upon the nature of the solvent used. The solvent properties like polarity, solubility, dielectric constant, etc. play a deciding role in extraction. The extraction of TBP was carried out using different aliphatic hydrocarbons like dodecane, NPH and kerosene, aromatic hydrocarbon like benzene and polar solvent like chloroform. All these solvents were loaded by 3% TBP for their economic consumption during the extraction process. The O/A ratio was kept constant as 1:1. The dissolution of TBP in non-polar solvents takes place due to dimerization of TBP while in polar solvents takes place because of association with the solvent. TBP has high solubility in non-polar solvents than polar solvents as dimerization constant value is higher than the association constant value [1]. Thus, high-extraction rate of TBP was observed in dodecane, kerosene and NPH as shown in Fig. 1. The amount of TBP in raffinate was 65, 70, 75, 85 and 95 ppm after extraction with NPH, dodecane, kerosene, benzene and chloroform, respectively. The rate of extraction of TBP is in the following order: NPH > dodecane > kerosene > benzene > chloroform. The rate constants values obtained from the first-order equations for solvents NPH, dodecane, kerosene, benzene and chloroform are 3×10^{-3},

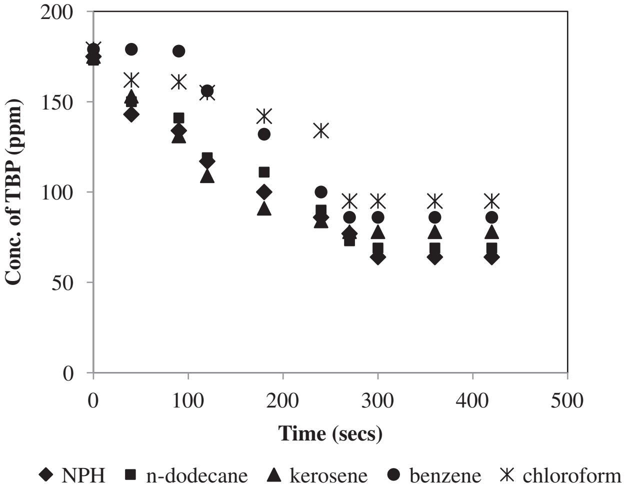

Fig. 1. Effect of different solvents on extraction of TBP.

2×10^{-3}, 3×10^{-3}, 2×10^{-3} and $1 \times 10^{-3}\,s^{-1}$, respectively. R^2 values obtained from the graphical representation of kinetic expressions for benzene and chloroform are less than that of NPH, kerosene and dodecane. This clearly elucidates deviation in the linearity of first-order kinetics for extraction of TBP using polar and aromatic solvents. High value for rate constants indicates high rate of extraction. There is marginal change in rate constant values because amount of TBP present in the aqueous phase was very less. Saleh has studied the effect of different diluents on the extraction of thorium nitrate with TBP. TBP was diluted with commercial kerosene than with toluene, rising sun kerosene, benzene, *n*-hexane, xylene, carbon tetrachloride, diethyl ether and di-isopropyl ether for extraction purpose. It was observed that the distribution co-efficient of thorium nitrate increases with decrease in dielectric constant of diluent. The extraction of thorium nitrate was found to be maximum in commercial kerosene and minimum in di-isopropyl ether [18]. Aghaie et al. have also observed that the solubility product of lithium carbonate increases with decreasing dielectric constant of solvent [19]. Similar results are obtained from the present kinetic study. As NPH has lowest dielectric constant while chloroform has highest dielectric constant, the extraction of TBP was found to be maximum in NPH and minimum in chloroform. Hence, NPH was chosen as the solvent to achieve a good overall performance for removal of dissolved TBP in the further studies.

3.2. Effect of solvent concentration

The effect of TBP amount in NPH on extraction has been studied by varying concentrations of TBP in NPH in range of 0–4%. All these different concentrations of solvents were contacted with 0.3 M HNO_3 in 1:1 ratio. The purpose of this study was to know the extent of TBP concentration in NPH beyond which the organic would have to be rejected, so that limited amount of organic is employed for the extraction process. It was found that as the concentration of TBP increased in NPH i.e. organic phase, the extraction of TBP from nitric acid decreases as shown in Fig. 2. The amount of TBP in the raffinate phase increases from 35 to 80 ppm with an increase in TBP loading in solvent from 1 to 4%. The rate constants values obtained from the first-order equations for 1, 2, 3 and 4% TBP in NPH are 2.8×10^{-2}, 1.0×10^{-2}, 3×10^{-3} and $1 \times 10^{-3}\,s^{-1}$, respectively. The decrease in R^2 values obtained from the kinetics graph replicates deviation in the linearity of first-order kinetics with increase in solvent concentration. It has been observed that the rate constant value decreases with the concentration of TBP in NPH i.e. the rate of extraction of TBP by NPH slows down with the loading of NPH by TBP. The rate of the extraction is highest in 1% TBP and slowest in 4% TBP. This is because when TBP is already present in the organic phase, it resists further removal of dissolved TBP from aqueous phase as the organic starts getting saturated with the TBP. Manohar et al. have also carried out batch studies to ascertain the distribution values of TBP in different concentrations of diluent and reported that when the diluent starts getting loaded with TBP, the percent extraction of TBP from aqueous phase decreases [20]. Around 65% extraction of TBP from aqueous feed was observed with 3% TBP in NPH as solvent. Thus, NPH loaded up to 3%TBP can be further reused for extraction in the continuous process like PUREX. Hence, by taking the economical and waste generation view into consideration, the kinetic study was carried using 3%TBP in NPH as the solvent.

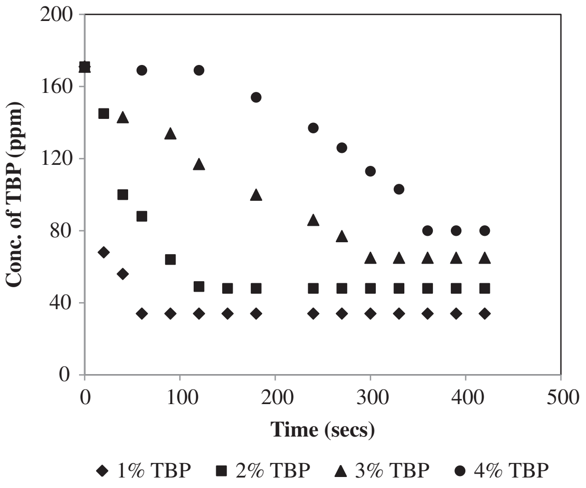

Fig. 2. Effect of solvent concentration on extraction of TBP.

3.3. Effect of O/A ratio

The ratio of feed to solvent also plays an important role in the extraction studies. It is necessary to have a proper amount of solvent for better contact between TBP and solvent for its efficient extraction from the aqueous feed. The ratio of aqueous to organic phase was varied to study its effect on mass transfer of TBP. The study was performed using 3% TBP in NPH as the organic phase and 0.3 M HNO_3 with dissolved TBP as the aqueous phase. The different O/A ratios used were 1:1, 1:2, 1:4 and 1:6 as shown in Fig. 3. The rate constants values obtained from the first-order

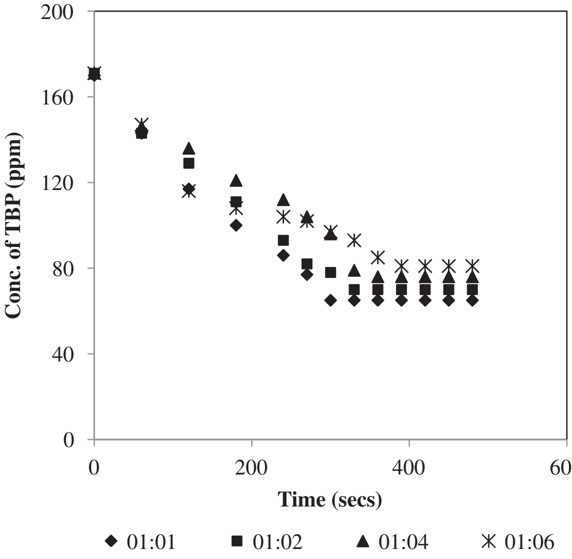

Fig. 3. Effect of organic to aqueous ratio on extraction of TBP.

equations for O/A ratios of 1:1, 1:2, 1:4 and 1:6 are 3×10^{-3}, 2×10^{-3}, 2×10^{-3} and $2 \times 10^{-3}\,s^{-1}$ respectively. High R^2 values obtained from graphical representation of kinetics data for different ratio proves that even at different solvent to organic ratio, linearity in first-order extraction kinetics is maintained. There is marginal change in rate constant values because amount of TBP present in the aqueous phase was very less. The amount of TBP left in the raffinate increases from 65 to 85 ppm with decrease in O/A ratio from 1:1 to 1:6. When the O/A ratio was 1:1 the amount of solvent available for extraction of dissolved TBP from nitric acid was two, four and six times greater than that of O/A ratio of 1:2, 1:4 and 1:6, respectively, and thus, the extraction was fast. Manohar et al. have also observed greater extraction of TBP at O/A ratio of 1:1 as compared with 1:6 because of accessibility of adequate amount of solvent for extraction [20]. As the rate constant value was highest with O/A ratio of 1:1, the kinetic study was done by keeping the O/A ratio of 1:1.

3.4. Effect of nitric acid concentrations

TBP dissolves in nitric acid because of hydrogen bonding and forms 1:1 complex with nitric acid. Investigators have reported that the solubility of TBP decreases with the concentration of nitric acid [21]. The concentration of nitric acid in the aqueous phase was varied from 0.3 to 3 M to check the effect of feed concentration on the extraction of TBP as shown in Fig. 4. During the entire study the organic phase was 3% TBP in NPH which was contacted with feed in 1:1 ratio. The rate constants values obtained from the first-order equations for 0.3, 1, 2 and 3 M HNO_3 in feed are 3×10^{-3}, 3×10^{-3}, 2×10^{-3} and $2 \times 10^{-3}\,s^{-1}$, respectively. High R^2 values in kinetics graph for different feed concentration proves linearity in first-order extraction kinetics. As the nitric acid concentration in the feed increases from 0.3 to 3 M, the amount of TBP in the feed decreases from 170 to 100 ppm. It is observed that as the extraction proceeds, the amount of TBP in the raffinate phase decreases from 65 to 45 ppm with an increase in the concentration of nitric acid from 0.3 to 3 M. The rate of extraction decreases with the concentration of nitric acid in feed because of back extraction of nitric acid by TBP due to complex formation in the organic phase. Thus, the concentration of TBP in the raffinate phase is more in 0.3 M HNO_3 than 3 M HNO_3 because of higher concentration of TBP in feed.

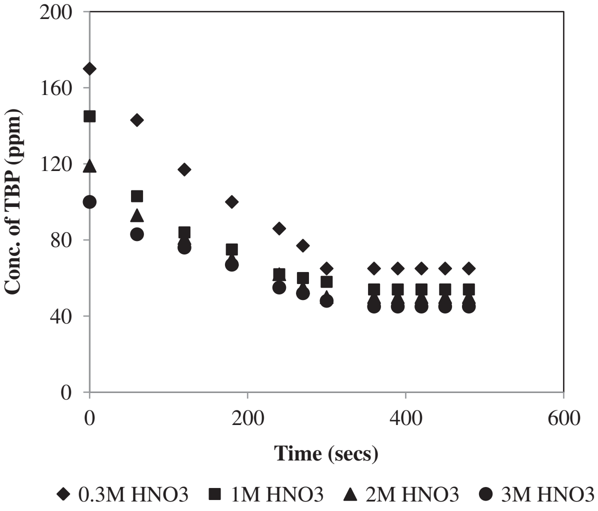

Fig. 4. Effect of feed concentration on extraction of TBP.

3.5. Effect of temperature

The temperature in the study has been varied from 35 to 100˚C as solubility of TBP in nitric acid is dependent on temperature [20]. The temperature effect was studied using 3% TBP in NPH as the solvent and 0.3 M HNO_3 as the aqueous feed in 1:1 ratio. Fig. 5 shows that the amount of TBP in raffinate phase decreases from 65 to 40 ppm, as temperature of the water bath increases from 35 to 95˚C. The rate constants values obtained from the first-order equations at 35, 45, 65, 85 and 95˚C are 3×10^{-3}, 2×10^{-3}, 3×10^{-3}, 3×10^{-3} and $4 \times 10^{-3}\,s^{-1}$, respectively. R^2 values obtained from

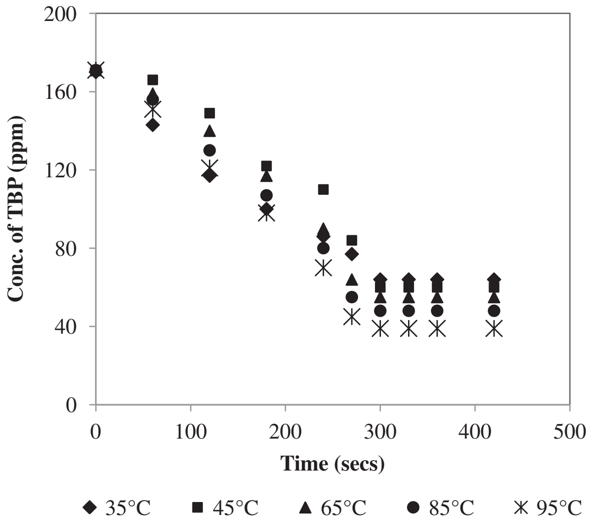

Fig. 5. Effect of temperature on extraction of TBP.

kinetics graph were also high which indicates first-order kinetics even at high temperature. It has been found that as the temperature increases, the extraction time also increases. There is a marginal change in rate constant values because amount of TBP present in the aqueous phase was very less. The extraction of TBP takes place slowly but at greater extent with an increase in temperature from 35 to 100˚C. The high amount of extraction is due to decrease in the solubility of TBP in water at higher temperature. Higgins et al. have also found decrease in the solubility of TBP in water with increasing temperature [22]. Thus, it can be concluded from this study that temperature triggers the extent of TBP extraction. As the change in the rate constants values with temperature is marginal due to limited amount of TBP presence in the aqueous phase, the selection of 35˚C as the extraction temperature for kinetic study was justified.

4. Conclusions

Different parameters affecting the extraction such as type of solvent, concentration of organic phase, concentration of aqueous phase, organic to aqueous phase (O/A) ratio and temperature have been successfully optimized for better overall performance in removing dissolved TBP from aqueous acidic feed solutions. The graphical representation of the kinetic expressions for different parameters has predicted that the extraction of TBP follows the first-order behaviour and the rate constants for each parameter have been calculated using this first-order equation. All the experimental graphs are representing exponential decay curve which is also an indicative of the first-order system. Approximately 62% of dissolved TBP was extracted from 0.3 M HNO_3 as the feed using 3% TBP in NPH as the solvent with O/A ratio of 1:1 in only 5 min at 35˚C. The amount of TBP left in the raffinate after extraction is within permissible limit for nuclear waste disposal to prevent red oil explosion. This batch kinetic study can be used in development of a robust and effective process for removal of dissolved TBP with minimum waste generation.

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