

Evaluation of Uranium Recovery Potential from Solvent Waste through Liquid-Liquid Extraction

Attempted recovery of uranium at the University of Leeds from stored solvent wastes containing uranium

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SUMMARY

- Uranium in the form of uranyl nitrate in tributyl phosphate arising from the UK Advanced Fuel Cycle Programme has been stored since 2019 due to its potential fiscal value in recovery and circular use
- Conducted back-extraction of solvent waste containing uranium with an unknown composition that has been stored in the Nuclear Engineering Laboratory at the University of Leeds

1. INTRODUCTION

1.1 Background

Despite the substantial benefits nuclear energy offers, radioactive waste management remains a primary challenge for nuclear industries globally. The Nuclear Decommissioning Authority reported that the total volume of radioactive waste in stock in the UK reached 137,000 m³ in 2022 [1]. To address the increasing volume of radioactive waste, some countries, including the UK until recently, operate under a closed nuclear fuel cycle, which employs reprocessing of irradiated nuclear fuel. The Plutonium Reduction Extraction (PUREX) process is widely adapted to recover uranium and plutonium from the spent nuclear fuel. This process aims to maximise resource utilisation by recycling uranium and plutonium, reducing the demand for fresh uranium mining and milling, and ensuring a more sustainable and long-term use of nuclear energy [2].

Liquid-Liquid Extraction (LLE) technique is crucial in achieving a high extraction efficiency by partitioning uranium and plutonium, facilitating the recovery of uranium from complex waste systems. A significance of uranium recovery through LLE is its ability to mitigate environmental risks associated with long-term waste storage by reducing the volume of radioactive waste. LLE not only offers environmental and technological advantages but also provides economic benefits by reducing waste disposal and processing expenses. Without reprocessing, the toxicity of the stored spent nuclear fuel is considered the primary contribution to toxicity associated with the fuel cycle for millions of years [3].

It has been reported that the nuclear energy research and development sector contributes 4 % of the total nuclear waste generated in the UK [4]. This indicates that not only large industries but also research facilities play a significant role in waste generation. The nuclear laboratory facility at the University of Leeds also owns nuclear waste from research activities, which was part of the Advanced Fuel Cycle Programme. For instance, recent uranium extraction using annular centrifugal contactors [5] generated the solvent containing uranium, which is stored in a liquid storage container in the facility without undergoing any processing. Therefore, the work in this manuscript assesses the viability of uranium recovery from tank waste through liquid-liquid extraction, promoting circular use of uranium within the facility and optimising resource utilisation.

Furthermore, with the increasing attention to sustainable utilisation of resources, this project holds novelty in presenting its environmental and economic benefits. The cost of the solid uranyl nitrate hexahydrate, $\text{UO}_2(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$, utilised at the University of Leeds was £6398 per kg at the time of purchase in 2021. It was originally planned to dispose of the solvent directly, but the waste hierarchy and best available technique assessment identified that the uranium could be recovered for reuse, and that this material is not waste but a residue, which is an important distinction.

Moreover, storing uranium solvent waste in containers at the facility without appropriate treatment poses risks of contamination. It also leads to the production of long-term waste, thereby increasing the costs associated with monitoring and maintaining regulatory compliance. Furthermore, the solvent system TBP/OK can degrade High Density Poly Ethylene (HDPE) bottles and can be leached plasticisers from the bottles. Therefore, this project aims to enhance resource utility and provide suggestions for effective uranium waste management practices by attempting uranium recovery.

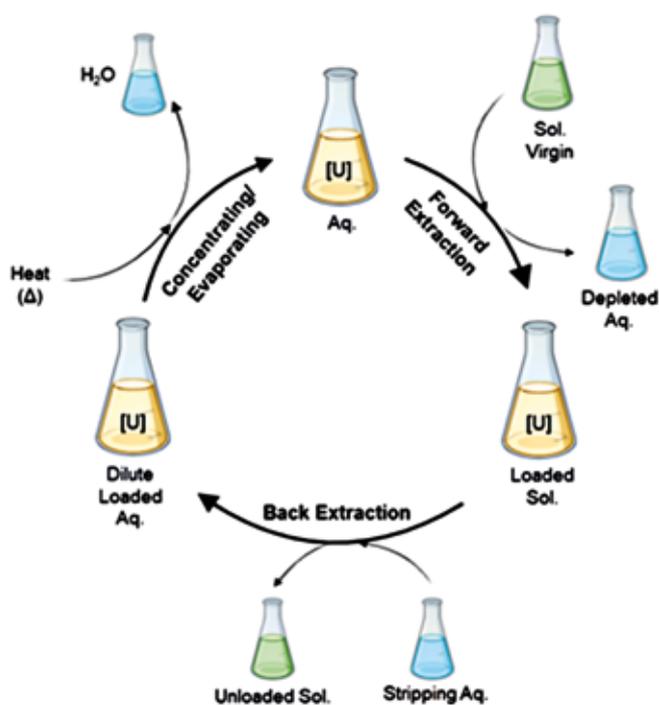


FIGURE 1: Schematic of envisaged circular use of uranium in UoLeeds, first the liquid-liquid forward extraction and followed by the back-extraction, then ultimately the concentration via evaporation to close the cycle.

1.2. Liquid-liquid extraction (LLE)

Liquid-liquid extraction (LLE) is a separation method in which the target solute is separated by being transferred from one solvent to another. The liquid solvents involved are immiscible or partially miscible with each other. The liquid phases are typically comprised of an aqueous mixture, and a nonpolar organic liquid is used as the second liquid phase [6]. The separation of two phases can arise from purely physical differences such as density and polarity, where separations are rarely specific. On the other hand, LLE can be obtained by the differential solubility arising from the chemical interaction of one solute with the solvent to form a complex, which is often utilised in metallurgical separations [7]. The liquid phases are contacted via mixing, followed by a phase separation. Interfacial turbulence yields an increase in the mass transfer with concentration driving force, where the complexation of uranyl nitrate and TBP occurs at the interface of aqueous and organic phases in the plutonium uranium reduction extraction [8] also known as PUREX [9]. Hence, vigorous agitation promotes an initial rapid rise in the interfacial area by reducing average drop size, which increases the mass transfer between the phases. Nevertheless, further increases in mixing beyond the optimal mixing point can hinder the separation as the rate of increase in the interfacial area begins to decrease and form an emulsion [6]. The rate at which the system in the separatory funnel reaches equilibrium after mixing is dependent on the surface area of the interface between the two phases. Therefore, the surface area between the two phases is minimised at rest.

Considerable research has been dedicated to the extraction of metal complexes using dialkyl esters of phosphoric acid. The solubility of the metal-dialkyl ester salt in aqueous solutions is studied to be approximately 10^{-4} M, with solubility decreasing as the alkyl radical chain length increases [7]. The salts encompass a low solubility in non-polar solvents; however, the solubility increases significantly in the increasing presence of dialkyl phosphoric acid.

Uranium nitrate is involved in most uranium reprocessing processes due to its high solubility. It is highly soluble in aqueous and key organic substances, including ethers, alcohols, and ketones [10]. Its high solubility, specifically in organic solvents, has a practical importance that allows the solvent extraction of uranium for the selective extraction processes.

Tributyl Phosphate (TBP), $C_{12}H_{27}O_4P$, is an odourless liquid that is used in the extraction of lanthanide and actinide elements [11]. The phosphate anion PO_4^{3-} and organic phosphates are strong complexing agents for actinide ions. It is due to the behaviour of phosphate anion as a bridge between metal ions, leading to the formation of insoluble aggregates in water. Likewise, TBP is one of the primary chemicals utilised for the PUREX process. TBP has a high viscosity of 3.39 cP, equivalent to 0.0039 Pa·s, with a density similar to that of water, which limits the use of pure TBP as an extractant. For this reason, desirable physical properties of organic solvents for reprocessing are attained by diluting the solvent with an inert hydrocarbon, kerosene. It is selected due to the low specific gravity and viscosity, allowing enhanced dispersion and phase disengagement [12]. 30 vol% TBP diluted with kerosene is used as the standard extractant for uranium as well as plutonium, both on a research and industrial scale.

2. METHODS

2.1. Back extraction of loaded solvent

The loaded solvent tank waste (10 mL) from the LLE was measured using a pipette (10 mL) after calibrating the pipette and added to a beaker (50 mL). Aqueous nitric acid (0.01 M, 10 mL) was measured and added to the beaker. A magnetic stirrer was placed within the beaker and placed on a magnetic stirrer. It was then stirred at 700 RPM for 15 min at room temperature. Once the contact was completed, the mixture was poured into a separatory funnel (50 mL). The sample was left for 20 minutes to allow phase separation. After 20 minutes, the stopcock was opened to disengage the reloaded aqueous into a labelled beaker. The unloaded solvent was disengaged to a sample tube. This procedure was repeated at different temperatures: 25, 40, 60 °C.

2.2. Ultraviolet-visible (UV-VIS) spectrophotometry

Cary WinUV software was opened to start the data collection. A prepared sample from the sample tube was transferred into a 4.5 mL quartz cuvette using a plastic pipette, filling approximately three-quarters of the cuvette's volume. The cuvette was positioned inside the cuvette holder of the UV-Vis instrument, ensuring the transparent side facing the beam. The measurement parameters were adjusted using the Setup function, wherein the minimum and maximum wavelengths were defined as 200 and 800 nm, respectively. The scan speed was set to "Survey" and the "Baseline" function was selected to collect a baseline spectrum. The sample was scanned by clicking the "Start" button. The data were collected at an interval corresponding to a uranium concentration of 1.5 g/L U, with the lowest concentration of 0.1 g/L U.

3. RESULTS AND DISCUSSION

3.1. Calibration curve and Liquid-Liquid Extraction

The solution containing uranyl nitrate at different dilutions was measured to obtain calibration spectra. The UV-Vis spectra of the stock solution, uranyl nitrate dissolved in nitric acid (4 M) at eight different dilutions are shown in Figure 2. Prior to measuring the stock samples at different dilutions, a blank HNO₃ solution was measured as a baseline. From the obtained spectra of the baseline and solutions of different uranium concentrations, the detection limit for uranium based on the sensitivity of equipment was determined as 0.1 g/L U. The uranyl spectra are not fully resolved due to unoptimized data intervals.

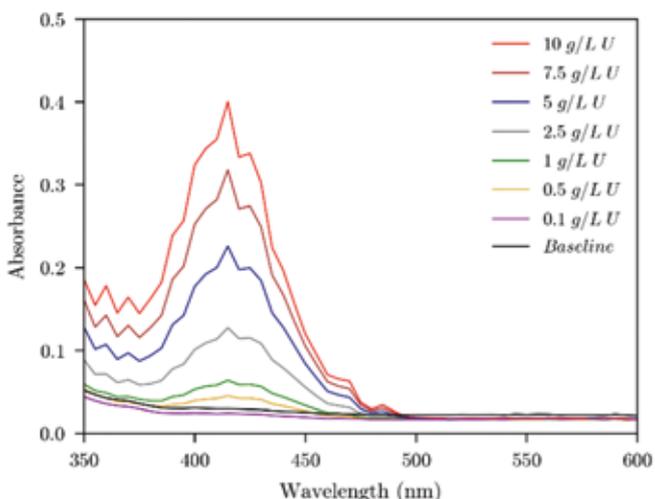


Figure 2 UV-Vis spectrum of uranyl nitrate dissolved in 4 M nitric acid

Uranyl nitrate solid does not dissolve directly in organic solvents. Therefore, the calibration curve for the solvents has to be obtained using a loaded solvent achieved through LLE. Solvents at different concentrations were obtained by conducting LLE with the 10 g/L U stock solution, followed by dilution. As a result, the concentration of uranium in the solvent phase exhibits a slightly lower absorbance compared to that of the aqueous phase, falling below the exact target concentration of 10, 7.5, 5, 2.5, 1, and 0.5 g/L U.

The absorbance of the Final Aqueous phase after LLE at 414 nm was measured to be 0.09046, corresponding to a concentration of 1.67 g/L using the aqueous calibration curve. Therefore, subtracting the concentration of the unextracted amount present in the Final Aqueous (1.67 g/L) from the concentration in the Initial Aqueous (9.82 g/L), yields the concentration of the Final Solvent to be 8.15 g/L. Using the solvent concentration of 8.15 g/L U, the concentrations of solution at different dilution factors are tabulated in Table 1. The calibration spectra for the solvent phase are shown in Figure 3. The initial target concentrations (10, 7.5, 5, 2.5, 1, and 0.5 g/L U) are used as labels in the legend, for the convenience of understanding. Whilst this work reports percentage extraction, other prefer distribution ratios, often denoted as (D). The extinction coefficient from the literature is 2.2×10^4 L/mol/cm [13].

Target Concentration (g/L)	[U] _{Initial Aqueous} (g/L)	[U] _{Final Aqueous} (g/L)	[U] _{Final Solvent} (g/L)	Extraction Efficiency (%)
10	9.815	1.670	8.145	83.0
7.5	7.650		6.109	79.9
5.0	5.233		4.073	77.8
2.5	2.640		2.036	77.1
1.0	0.965		0.815	84.5
0.5	0.473		0.407	86.0

Table 1 Table showing U concentration of Final Solvent

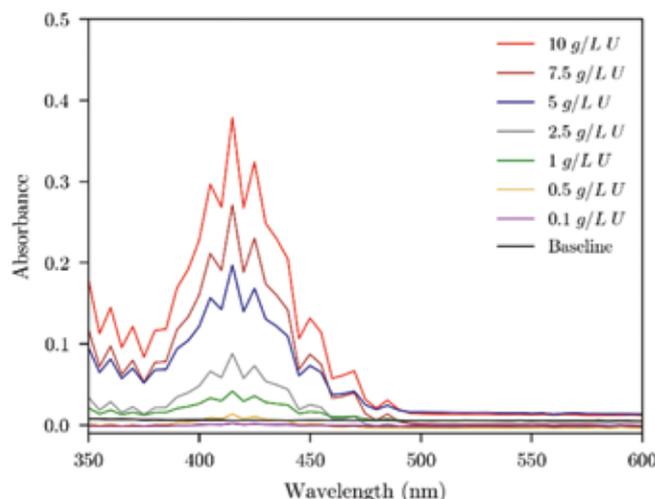


Figure 3 UV-Vis spectra of uranyl nitrate dissolved in 30% TBP in kerosene

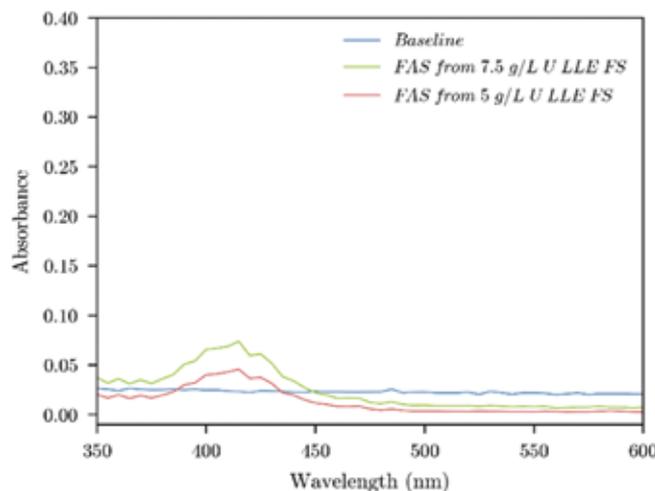


Figure 4 UV-Vis spectra of Final Aqueous from stripping using fresh stock

3.2. Stripping of Uranium from Solvent Waste

The study investigated the extraction of uranium from a stock liquid of uranium solvent waste, which was stored in the

nuclear laboratory at the University of Leeds. The waste has been generated from experiments for academic and research purposes. It is contained in designated plastic waste disposal containers. Production records indicate that waste production from 2019 to 2022, with storage maintained at room temperature without further management. Additional waste generated from subsequent experiments has been continually added to the tank waste. Consequently, the exact composition of the solvent waste, along with its constituent chemicals remains unknown. However, it is predominately composed of 30 vol% TBP in kerosene, containing uranium and nitric acid concentration.

To explore the feasibility of recovering uranium from the solvent tank waste, the stripping was performed using various concentrations of aqueous nitric acid: 0.1, 0.01, 0.001, and 0 M. It was observed that the stripping efficiency of the LLE was calculated to be <1%.

In addition to the concentration of the stripping agent, factors such as temperature, contact time, and the solvent-to-aqueous ratio significantly influence the stripping equilibrium of U (VI). Due to the unsuccessful stripping observed with various concentrations of dilute nitric acid, these variables were investigated to assess the impact on extraction efficiency and, thereby the feasibility of achieving successful stripping.

Despite the changes in the experimental matrix influencing the extraction process and efficiency, no absorbance peak at 414 nm was observed. According to the literature in the study on U(IV) stripping by Rahman et al., the stripping was conducted using S/A ratios of 1:3 and 1:5. Additionally, the temperature was increased to 60 °C, and the contact time was extended to 60 minutes. However, the collected spectra consistently showed a similar trend.

3.3. Stripping of Uranium from Loaded Solvent Stock Solution

To validate the accuracy of the methodology and to compare it with a standard solution, stripping was performed employing the loaded solvent acquired from the liquid-liquid extraction of a freshly prepared uranyl nitrate stock solution (10 g/L U) for the calibration. Due to the limited volume of the stock solution at 10 g/L U, concentrations of 7.5 g/L and 5 g/L U were utilised for the experiment. The resulting UV-Vis spectra are presented in Figure 4.

The stripping efficiency of 79.9% and 88.1%, indicating that effective stripping has taken place. The data is summarised in Table 2:

Theoretical [U] in FS (g/L)	Initial [U] in FS (g/L)	Final [U] in FAS (g/L)	Stripping Efficiency (%)
7.5	6.11	4.88	79.9
5.0	4.07	3.59	88.1

Table 2 Extraction efficiency

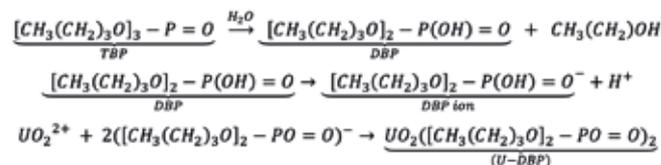
The spectrum of the stock solvent waste was measured to assess the uranium content in the original waste. Interestingly, instead of exhibiting a uranium peak at 414 nm, it showed a curve and noise pattern highly similar to that of aqueous nitric acid. In contrast, the spectrum of the tank waste differed significantly from the solvent baseline.

3.4. Acid Degradation of Tri-Butyl-Phosphate

The investigation demonstrated that the extraction of uranium from solvent waste stored for an extended period is impractical. The UV-Vis spectrum of the stock solvent waste, primarily comprising TBP and kerosene, exhibited a highly similar peak and noise pattern to that of aqueous nitric acid. Consequently, the underlying chemistry was further researched. The result may be attributed to the behaviour of TBP which undergoes hydrolytic reactions when exposed to acidic conditions and gamma radiation.

It was determined from the experiment that the extraction of uranium is not practical for the solvent waste that has been stored for several years. The long-term storage of PUREX solvent waste containing metal ions and TBP undergo hydrolysis, resulting in the formation of dibutyl phosphate (DBP) and monobutyl phosphate (MBP) [14]. The hydrolysed products react with uranium to form stable U-DBP and U-MBP complexes. These complexes present vital difficulties for uranium extraction, resulting in irreversible extraction. U MBP has relatively higher solubility, and thus remains in solution. In contrast, U-DBP complex encompasses limited solubility, hence precipitates and forms a coating on the surfaces of tanks or containers [14].

The degradation of TBP into DBP results in undesired components, mainly comprising DBP as shown in the equations below showing the reactions during the formation of the U-DBP complex:



The primary difference between TBP and DBP is the structure of the atoms and bonding. TBP consists of a phosphorus atom surrounded by hydrocarbon chains, and three butyl groups. Whereas the phosphorus atom in DBP is bonded to two butyl hydrocarbon chains, and one bonded to a hydroxyl group, as shown in Figure 5. The strength of complexation is influenced by the charge density of anion or ligand as it approaches the metal [3]. The bond dissociation energy of OH is 460 kJ/mol, whereas the value for the CH bond is 389 kJ/mol [15]. As ligands bind to the metal ion through coordinate covalent bonds, the additional hydroxyl group present in DBP may enhance binding, allowing for stronger coordination, hence leading to a more stable complex. The actinide-ligand bonds in complexes are ionic, with the order of complexing power of typical anion of $\text{F}^- > \text{NO}_3^- > \text{Cl}^- > \text{ClO}_4^-$, indicating a relatively high complexing power associated with the investigated system [3]. Therefore, if the formation of DBP has occurred in the solvent waste, it would result in a challenge to break the complex.

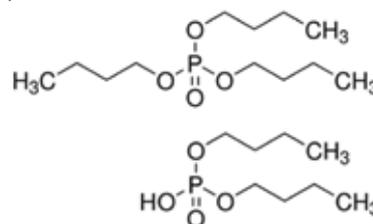
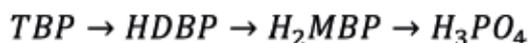


Figure 5 Structure of (top).TBP and (bottom).DBP

To facilitate an effective extraction process, chelating agents can be utilised, such as NTA and EDTA which promote fractional precipitation. The metal ion is fractionated as it gets dissolved in a weak alkaline solution of NTA and acidified to cause precipitation of oxalates [16]. EDTA acts as a chelating ligand forming a strong complex with uranium ions.

TBP decomposition is shown below, leading to an increased rate of acidic byproduct formation [17]:



The described reaction shows the accumulation of degradation products in solvents that cause adverse effects. It was determined from research that there is a significant contribution of TBP decomposition on the extraction performance which is caused by the alpha radiolysis and metal-ion hydrolysis [18]. Furthermore, from another literature reference, it was also evident that the rate of acid hydrolysis increases with the concentration of nitric acid and metal-ion, radiation dose, and temperature [17]. The degradation product results in an unfavourable impact on extraction, such as emulsification and crud formation [19].

The presence of HDBP is studied to have a significant effect on the extraction of actinides, which influences the distribution of tetravalent and hexavalent actinides [19]. Strong and organic-soluble complexes are formed between the phosphate and actinide ions, for instance, a U(VI) polymer, $UO_2(DBP)_2$. Furthermore, it was observed that the presence of HDBP inhibits the stripping of uranium, thereby lowering the stripping efficiency [19]. HDBP is a moderately strong acid, which partially deprotonates and exists as an anion. In the presence of a low concentration of aqueous nitric acid (0.1 to 0.5 M), the DBP anion acts as a chelating agent which displaces nitrites to promote the formation of strong complexes. In contrast, the high concentration of nitric acid inhibits the deprotonation of HDBP and remains as an HDBP molecule. It acts as a monodentate ligand, leading to reduced binding capacity [19].

Conventional solvent management in reprocessing plants utilises a bleed/replace and solvent wash process to maintain solvent quality. The solvent wash uses a sequential dilute nitric acid, sodium carbonate, sodium hydroxide and dilute nitric acid. Alkaline washed are used to reduce acidic degradation products, including DBP. Carbonate is used as the first alkaline wash to keep metals soluble and prevent accumulation due to precipitation [20].

Solvent cleanup practice in reprocessing plants utilises sodium carbonate solution for scrubbing the contaminated TBP solvent before commencing solvent extraction. The use of sodium carbonate removes the HDBP and H_2MBP and forms metal-carbonate complexes [21]. However, unsatisfactory results are obtained as it causes severe emulsions between the organic and Na_2CO_3 in the presence of degradation products. For this reason, PUREX solvent at the Oak Ridge Y-12 plant is discarded at an interval of one month, due to the buildup of DBP causing operational difficulties. It was reported that the recycle solvent from 30 days of operation contains around 20 g/L of uranium, with an approximate DBP concentration of 0.01 M [21]. It provides the DBP production rate of 0.0015 mol/h with an approximated inventory of 100 L of solvent waste [21]. The rate of TBP concentration can be expressed as:

$$\frac{dC}{dt} = \frac{n}{V}$$

where C is the DBP concentration (mol/L), t is time (h), n is the rate of DBP production (mol/h), and V is the total volume of solvent (L). It is assumed that the initial concentration of DBP present is negligible.

The equation integrates to:

$$C - C_0 = \frac{nt}{V}$$

Rearranging for time, t gives:

$$t = \frac{(C - C_0)V}{n}$$

$$t = \frac{(0.01 \text{ mol L}^{-1} - 0 \text{ mol L}^{-1}) \times 15 \text{ L}}{0.0015 \text{ mol h}^{-1} \times 24 \text{ h}} = 4.2 \text{ days}$$

Assuming that each waste container at the University of Leeds has 15 L of solvent waste, after 4.2 days or 100 hours, the tank waste would have reached the DBP concentration of 0.01 M which is the level for disposal used at Oak-Ridge Y 12.

4. CONCLUSIONS

This project aimed to investigate the potential for recycling uranium from solvent waste to support sustainable uranium management. The stripping of solvent waste containing uranium was conducted to assess the feasibility of uranium recovery.

Metal contents in samples were analysed using UV Vis spectroscopy. Despite varying experimental parameters that affect stripping efficiency such as temperature, solvent-to-aqueous ratio, and contact time, uranium stripping from the solvent waste was unsuccessful.

In contrast, the experiment demonstrated that successful LLE and stripping were achieved with a freshly prepared solution of uranyl nitrate, resulting in a stripping efficiency exceeding 77 %. This outcome suggests potential degradation of TBP in the solvent waste due to the extended storage period. A by product DBP results in the formation of a U-DBP complex, exhibiting high solubility, thereby leading to ineffective stripping using nitric acid.

Assuming each container contains 15 L of solvent waste with intense alpha and gamma radiation dose rates [21] as in reprocessing facilities, it is estimated that the DBP level will reach the recommended disposal threshold within 4 days. While the DBP production rate for solvent waste with high radiation exposure is available, data on the DBP production rate for residue with low uranium content and minimal radiolytic degradation is limited. This highlights the necessity of performing uranium recovery soon after the extraction to prevent DBP accumulation and the formation of unextractable compounds. Furthermore, this suggests identifying a factor for the rate of DBP buildup in uranium solvent residues is expected to be valuable for further investigation, to establish a correlation between DBP buildup under intense radiolytic degradation and that resulting only from hydrolysis.

The waste could be utilised for further studies to determine the

exact amount of DBP and MBP present in the waste for stripping using suitable extractants. Additionally, the use of alkaline reagents is necessary for uranium recovery as dilute nitric acid is ineffective. However, the use of alkaline reagents complicates the recovery process, increasing both complexity and costs, thereby making uranium recovery economically unfavourable.

Therefore, the investigation has successfully achieved the aim of assessing the potential for uranium recovery, contributing to the practice of the most sustainable and economical utilisation of resources. If untreated, the TBP/OK could be exported for off-site disposal using thermal treatment. As a result of conducting this project 'Best Available Techniques' (BAT) for the Management of the Generation and Disposal of Radioactive Wastes [22] resource has surfaced and will be consulted as part to future waste management.

5. FUTURE WORK

Quantification of the presence of TBP or DBP in the solvent phase could be performed with the utilisation of Fourier Transform Infrared Spectroscopy, Gas Chromatography, Electrospray Ionization Mass Spectrometry. Laser Induced Breakdown Spectroscopy (LIBS) could be used for uranium concentration, is anticipated to be beneficial as LIBS allows precise metal content analysis in solutions with minimal sample preparation, making it highly suitable for applications in nuclear research and industries.

Moreover, alternative extractants with appropriate chelating properties that facilitate the effective stripping of solvents that

contain acid degradation products can be investigated. Literature suggested that 2 ethyl hexanol aids the extraction of HDBP, promoting further investigation into its effect in facilitating the circular use of uranium from waste solvents. Additionally, the rate of DBP formation and its effect on the formation of complexes of DBP and MBP can be further investigated, given its significant operational implication in nuclear facilities.

Furthermore, diverse experimental matrices, such as broader variation in the S/A ratio and ranges of metal concentrations, may be employed to attempt to achieve successful stripping from solvent wastes. Based on the result and findings of the present work, further research on DBP formation and its effect on stripping is valuable and is anticipated to contribute to methodology development for identifying the extractability of solvent solutions with unspecified compositions of chemical constituents.

Ultimately it is known that an alkaline wash of sodium carbonate or sodium hydroxide or ammonium carbonate solution to remove acidic organic degradation products [20] and will be investigated in future work. Uranium has been recovered via alkalification from contaminated liquid solvent wastes, better known as organic residues, at the National Nuclear Laboratory (NNL) Preston Laboratory Residue Processing Plant using several suites of treatments [23]. Building on the conducted research, established industrial practices and identified challenges, future efforts should aim to optimise solvent extraction techniques to manage waste containing acid degradation products, thereby enhancing resource recovery.

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