



Mineral Acids in Chloroform Solution of 1-Phenyl-3-methyl-4-Trichloroacetyl 5-pyrazolone (HTCP) Schiff Base: Abstraction Effect on Uranium (VI)

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Article Information

Article # 10032

Received: 4th April, 2024

1st Revision: 20th April, 2024

2nd Revision: 3rd May 2024

Acceptance: 8th May 2024

Available online:

15th May 2024.

Key Words

Chloroform, chloride,

Abstraction, Acetate

Sulphate ions

Mineral acids

Abstraction

Abstract

A Chloroform solution of 1-Phenyl-3-methyl-4-Trichloroacetyl 5-pyrazolone (HTCP) as a medium for the abstraction of UO_2^{2+} was performed to examine the synergetic effect of mineral acids on the mode of distribution. The study was performed using solvent-solvent extraction method with 1×10^{-3} , 5×10^{-3} , 1×10^{-2} , 5×10^{-2} , 1×10^{-1} , and 5×10^{-1} M liquid phases of CH_3COOH , HCl , and H_2SO_4 respectively. The solutions were manually agitated for 30 mins, and the extraction raffinate collected, and analyzed using colorimetry. Ratios of distribution (D) and percentages extraction (% E) were analysed statistically. Results obtained showed that the interference of chloride, acetate and sulphate ions was more conspicuous as the mineral acid concentrations got greater than 1×10^{-2} M. There was an optimum % abstraction of 94.71, 91.63 and 46.26 %, at 1×10^{-3} , 1×10^{-2} and 1 M CH_3COOH . Further, there was an optimum % abstraction of 80.62, 77.97, and 38.77 % at 1×10^{-3} , 1×10^{-2} , and 1 M HCl . A gradient of 1 was obtained with CH_3COOH and HCl , indicating a displacement of 1 proton during the reaction. An optimum % abstraction of 52.86 and 41.85 % at 1×10^{-3} and 1×10^{-2} M H_2SO_4 were achieved. A gradient of 2 was obtained statistically, indicating a displacement of 2H^+ . 1M SO_4^{2-} concentration showed no abstraction at the concentrations studied. Overall, the results indicate that CH_3COOH , HCl and H_2SO_4 are only efficient abstraction media of Uranium metal ion at low concentrations.

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Introduction

Increasing pursuance for unadulterated products and eco-friendlier processing pathways for abstracting heavy metals from the environment has prompted various abstraction methods (Nugraha *et al.*, 2017; Silva *et al.*, 2005). Liquid-liquid extraction is one method that has recorded tremendous success in the abstraction of heavy metals (Murdoch, 2010). In liquid-liquid extraction, an analyte is distributed between two immiscible, organic and liquid phases (Jrose, 2001; Singh, 2002; Ashall, 2007). Liquid-liquid extraction is influenced by factors such as equilibration time, pH of liquid phase, concentration of ligand, presence of masking agents, presence of salting out agents, oxidation state of metal and type of solvent used (Rashid and Ejar, 1985; Okafor and Uzuokwu, 1990; Mhaske and Dhadke, 2001; Godwin and Young, 2020).

Schiff bases and their derivatives have been synthesized and applied successfully in solvent-solvent extraction of metals (Zainab *et al.*, 2014; Ramhari *et al.*, 2023; Zyadanogullaris *et al.*, 2008). Sulfamethoxazole-based Schiff base was successfully synthesized and characterized by Hussain *et al.*

(2014). *Kołodziejńska* (2013) and Azra *et al.* (2022) affirmed the effectiveness of different ligands and chelates for the abstraction of heavy metal ions at different pH, equilibration time and concentrations. Studies further affirmed that reagents used for the abstraction can have either salting out or masking effect at various concentrations (Watanabe *et al.*, 2001).

Cd (II) chloride and Fe (III) were successfully separated by a Schiff base (Liu *et al.*, 2007). The synergetic effect of HBuP in the distribution of Cadmium between buffered aqueous solution and chloroform solution of H_2BuEtP was studied by Godwin and Tella (2017). Quantitative extraction of Thorium and other metal ions from Schiff bases was reported by Zoubi (2013). Zoubi *et al.* (2016) studied the abstraction of chromium and copper with a Schiff base and reported that Cu^{2+} showed highest abstraction and selectivity at pH 6.27. Biologically and analytically the usefulness of Schiff bases and derivatives had been reported (Ebosie *et al.*, 2021). Bennett and Ayawei (2022) studied the effect of nitric acid on the distribution of UO_2^{2+} with HTCP. They

reported an efficient % extraction (%E) at the various acid concentrations. Khoutoul *et al.* (2016) investigated the abstraction of metal ions with 1,2,4-triazole Schiff base derivatives. They reported a high extraction selectivity of Fe^{2+} (90.1 %) and Pb^{2+} (94.3 %). Godwin and Bennett (2022) studied the role of acids in multi-metal distributions using H_2BuEtP and reported that only 0.01– 0.05 M H_3PO_4 showed efficient % E at pH 7.5

The current study examines the abstraction of UO_2^{2+} with three mineral acids in a chloroform solution of HTCP Schiff base.

Materials and Methods

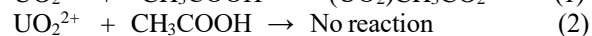
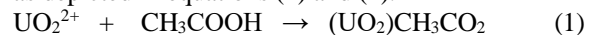
Analytical-grade reagents were purchased and used without further purification. HTCP was synthesized and characterized using the method reported in the literature (Bennett *et al.*, 2020). Roughly 108 abstraction bottles (10 mL) were used for the study. These were arranged in nine sets for the three acids (CH_3COOH , HCl and H_2SO_4). Molar concentrations of the acids were in the range 1×10^{-3} – 1 M.

5×10^{-2} M HTCP stock solution was prepared. 2000 mg/L metal stock solution was prepared as described in the literature (Bennett and Ayawei, 2022). The working concentration of the aqueous phase was 50 mg/L. The liquid phase was prepared by transferring 0.2 mL of 2000 mg/L UO_2^{2+} stock into fifty-four of the 10 mL abstraction bottles and the volumes made up to 2 mL from stock solutions of the acids of different molar concentrations (1×10^{-2} –1 M).

2 mL of the organic phase, HTCP in chloroform was transferred into each of the abstraction bottles and agitated for 30 mins. 1 mL of the raffinate was withdrawn from each of the abstraction bottles and transferred to fifty-four (54) clean abstraction bottles and analysed using a UV Spectrophotometer at 520 nm. The potentiometric titration method was used in determining the pKa of the base (Godwin and Uzoukwu, 2012; Godwin and Bennett, 2022). UV Spectrophotometer was used to determine the amount of UO_2^{2+} in the liquid phase.

Results and Discussions

CH_3COOH : About 10^{-2} M solutions of CH_3COOH showed no abstraction of the metal ion, UO_2^{2+} . Non-extraction was ascribed to non-extractable acetate-uranyl complex formation at this molar concentration as depicted in equations (1) and (2).



Nonetheless, UO_2^{2+} was extracted at concentrations 1×10^{-3} M – 1×10^{-1} M. The percentage abstraction (% A) chart of 200 ppm UO_2^{2+} from 1×10^{-2} M SO_4^{2-} solutions of CH_3COOH into 5×10^{-2} M HTCP in chloroform is presented in Figure 1. The gradient was statistically obtained as 1, indicating a displacement of 1 proton during the reaction. The percentage abstractions were 94.71, 93.83, 92.07 and 40.97 % at concentrations 1×10^{-3} , 5×10^{-3} , 1×10^{-2} , 5×10^{-2} and 1×10^{-1} M respectively.

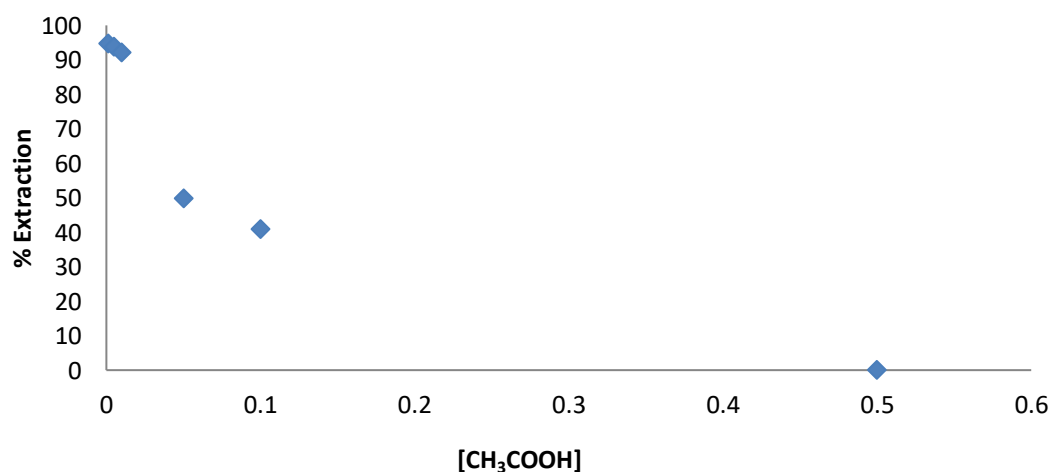


Figure 1: Percentage abstraction chart of UO_2^{2+} from 5×10^{-2} M HTCP solutions of CH_3COOH containing 1×10^{-2} M SO_4^{2-}

Equation (3) represents the abstraction process of UO_2^{2+} from liquid media containing 5×10^{-2} M HTCP from various molar concentrations of CH_3COOH .



The mole ratio interaction of the metal, and acid was 1:1. Equations (4) and (5) gave the distribution ratio (D) and the percentage abstractions (% A).

$$D = \frac{\text{Standard Absorbance} - \text{Raffinate Absorbance}}{\text{Raffinate Absorbance}} \quad (4)$$

$$\% A = \frac{\text{Standard Absorbance} - \text{Raffinate Absorbance} \times 100}{\text{Standard Absorbance}} \quad (5)$$

The % A chart of 200 ppm UO_2^{2+} from 1×10^{-1} M SO_4^{2-} solutions of CH_3COOH into 5×10^{-2} M HTCP in chloroform is presented in Figure 2. There was abstraction at molar concentrations 1×10^{-3} , 5×10^{-3} and 1×10^{-2} M, but very minimal abstraction at

concentration 5×10^{-2} M and no extraction at concentrations 1×10^{-1} and 5×10^{-1} M. This result suggests that obstruction of acetate CH_3COO^- became pronounced as the molar concentration of CH_3COOH was greater than 1×10^{-2} M.

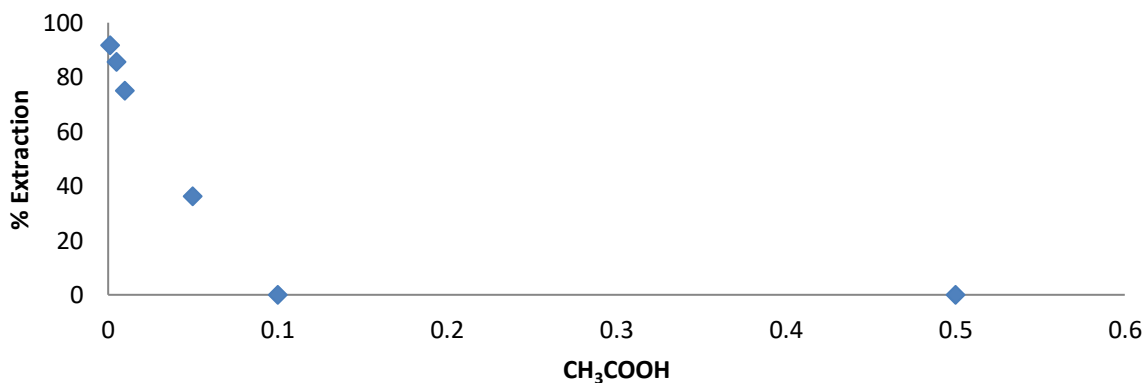


Figure 2: Percentage extraction chart of UO_2^{2+} from 5×10^{-2} M HTCP solutions of CH_3COOH containing 10^{-1} M SO_4^{2-}

A gradient of 1 was also achieved suggesting the displacement of 1 proton in the course of the reaction. Percentage abstractions were 91.63, 85.46, and 74.89 % at 1×10^{-3} , 5×10^{-3} , and 1×10^{-2} M respectively. The % A chart of 200 ppm UO_2^{2+} from 1 M SO_4^{2-} solutions

of CH_3COOH into 5×10^{-2} M HTCP in chloroform is presented in Figure 3.

Results displayed in Figures 1, 2 and 3 suggest that the optimal % A of UO_2^{2+} decreased as the concentration of CH_3COO^- increased in the buffered phase

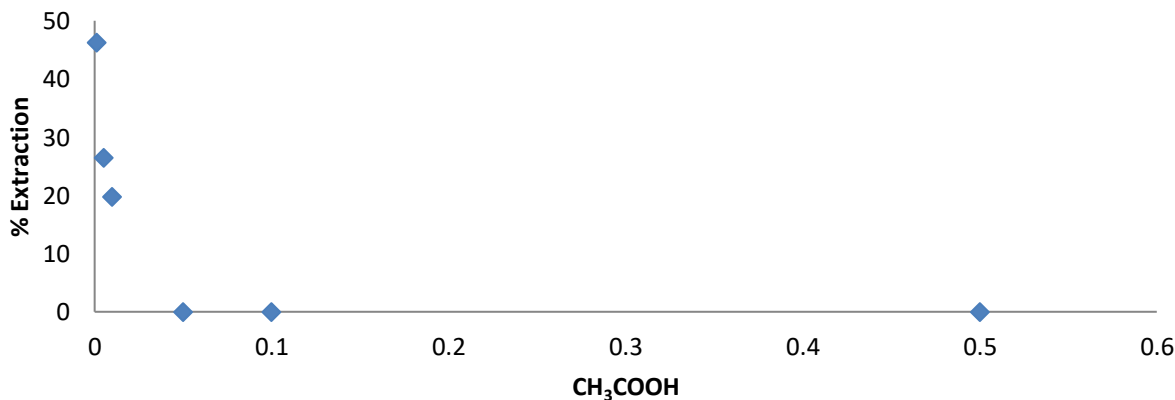


Figure 3: Percentage extraction chart of UO_2^{2+} from 5×10^{-2} M HTCP solutions of CH_3COOH in 1 M SO_4^{2-}

Interestingly, the results showed that abstraction of the UO_2^{2+} was better between CH_3COOH concentration 1×10^{-3} and 1×10^{-2} M, though the maximum % A of UO_2^{2+} reduced with an increase in the molar concentration of CH_3COO^- in the buffered phase. Furthermore, the CH_3COO^- ion concentrations (10^{-2} – 1 M) showed a gradient of 1, suggesting that 1 proton took part in the reaction. The result also suggests that distribution of the metal ion depends on the molar concentration of the acetate ions in the liquid phase. Method of gradient analysis of the abstracted metal ion was reported by Yang *et al.* (2013). Equations (6), (7) and (8) represent the interaction of the molecules.

$$n = \frac{d[\log D]}{d[\log HA]} \quad (6)$$

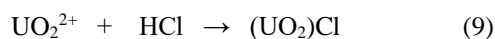
$$n = \frac{d[\log D]}{d[\log CH_3COOH]} \quad (7)$$

$$n = \frac{d[\log D]}{d[\log M^{2+}]} \quad (8)$$

Applying equation 7, the number of molecules, n, is 1; meaning 1 proton was released from 1×10^{-3} – 1 M

CH_3COOH concentrations. Statistically, a mole of UO_2^{2+} reacted during the process. The result was comparable with what was reported by Rajan and Martell (1965). Thus, a complex, $(UO_2)CH_3CO_2$, was formed with a 1:1 ligand metal ratio.

HCl: Abstraction of UO_2^{2+} from 1×10^{-2} M SO_4^{2-} solutions of HCl showed UO_2^{2+} abstraction at concentrations 5×10^{-3} – 5×10^{-1} M. Non-abstraction was ascribed to the formation of chloride-uranyl complex at these concentrations as shown in equation (9).



The mole ratio interaction of the metal and acid was 1:1. Nonetheless, there was an optimal abstraction, 80.62%, of UO_2^{2+} at molar concentration 1×10^{-3} M as shown in Figure 4. A gradient of 1 was attributed to a displacement of 1 proton during the interaction.

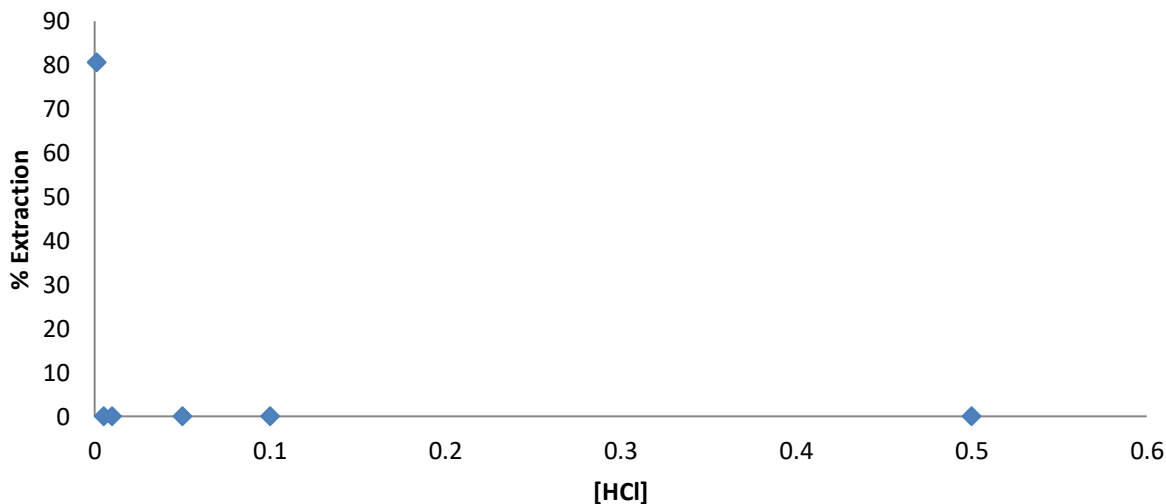


Figure 4: Percentage extraction chart of UO_2^{2+} from 5×10^{-2} M HTCP solutions of HCl containing 10^{-2} M SO_4^{2-}

Equation (11) represents the abstraction process of UO_2^{2+} from liquid media containing 5×10^{-2} M HTCP from concentrations of HCl.

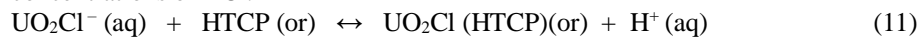


Figure 5 presents the %A plot from molar concentrations of HCl containing 1×10^{-1} M SO_4^{2-} . There was optimal abstraction of 77.97 % at molar concentration 1×10^{-3} M but no abstraction at

concentrations 5×10^{-3} – 5×10^{-1} M. This result indicates that abstraction of UO_2^{2+} was masked as Cl^- concentration in the liquid phase got greater than 1×10^{-2} M.

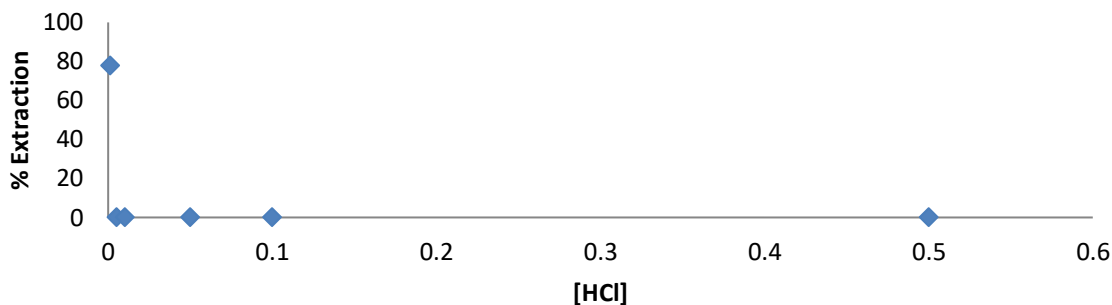


Figure 5: Percentage extraction chart of UO_2^{2+} from $5 \times 10^{-2} M$ HTCP solutions of HCl containing $10^{-1} M SO_4^{2-}$

The % A chart of 200 ppm UO_2^{2+} from $1 M SO_4^{2-}$ solutions of HCl into $5 \times 10^{-2} M$ HTCP in chloroform is presented in Figure 6. The % A of 38.77 % was recorded at molar concentration $1 \times 10^{-3} M$. A gradient of 1 was calculated. Figures 4, 5 and 6 indicate that the maximum % A of UO_2^{2+} decreased with increasing Cl^-

in the buffered phase. The % A of UO_2^{2+} was optimal at $1 \times 10^{-3} M$ hydrochloric acid. Bennett and Ayawei (2022) and Sangoremi *et al.* (2013) reported comparable results in their studies of the abstraction of UO_2^{2+} with the HTCP Schiff base.

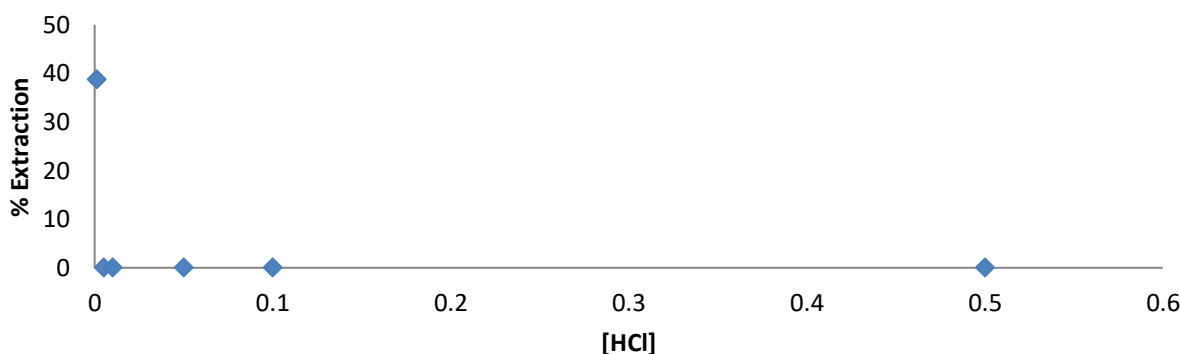


Figure 6: Percentage extraction chart of UO_2^{2+} from $5 \times 10^{-2} M$ HTCP solutions of HCl containing $1 M SO_4^{2-}$

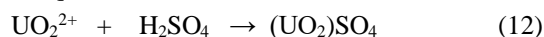
Applying equation 7, the three Cl^- ion concentrations, $1 \times 10^{-2} - 1 M$, all showed a gradient of 1, suggesting that 1 proton was released as UO_2Cl^- interacted with the ligand (HTCP). The results further suggest that the distribution of UO_2^{2+} is dependent on the amount of Cl^- in the liquid phase. Statistically, one mole of UO_2^{2+} reacted with one mole of the ligand, HTCP. A complex similar to $(UO_2)Cl$ was formed during the interaction. An abstraction of 38.77 % was recorded at molar concentration $1 \times 10^{-3} M$ as presented in Figure 6.

H₂SO₄:

The UO_2^{2+} abstraction from $1 \times 10^{-2} M SO_4^{2-}$ solutions of H_2SO_4 showed no abstraction of UO_2^{2+} at concentrations $5 \times 10^{-3} M - 5 \times 10^{-1} M$. This attributed to the formation of non-extractible uranyl- sulphate $((UO_2)SO_4)$ complex at these concentrations as shown in equations (12) and (13).



Figure 7 presents the % A chart of 200 ppm UO_2^{2+} at $1 \times 10^{-2} M SO_4^{2-}$ solutions of H_2SO_4 into $5 \times 10^{-2} M$ HTCP in chloroform. The % A was 52.86 % at concentration $1 \times 10^{-3} M$ and a gradient of 2 was calculated, insinuating that 2 protons were displaced during the reaction process.



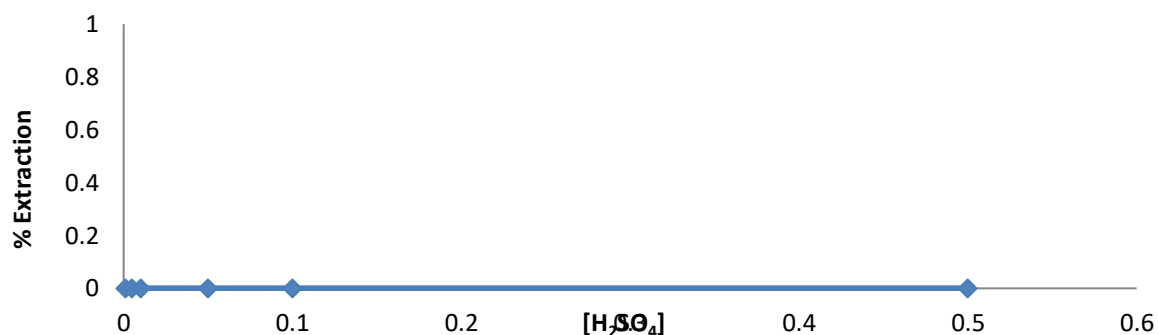
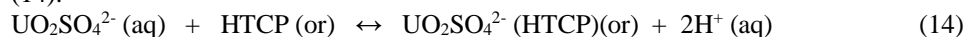


Figure 7: Percentage extraction chart of UO_2^{2+} from 5×10^{-2} M HTCP solutions of H_2SO_4 containing 10^{-2} M SO_4^{2-}

The reaction for the abstraction of UO_2^{2+} from 5×10^{-2} M HTCP in chloroform from H_2SO_4 is represented in equation (14).



Acid: Uranium mole ratio interaction was 1:2. Figure 8 presents the % A chart of 200 ppm UO_2^{2+} from 1×10^{-1} M SO_4^{2-} solutions of H_2SO_4 . There was abstraction at $1 \times 10^{-3} - 1 \times 10^{-2}$ M, but no abstraction

from $5 \times 10^{-3} - 5 \times 10^{-1}$ M, suggesting that interference of SO_4^{2-} in the abstraction of UO_2^{2+} became very pronounced as H_2SO_4 concentration got greater than 1×10^{-2} M.

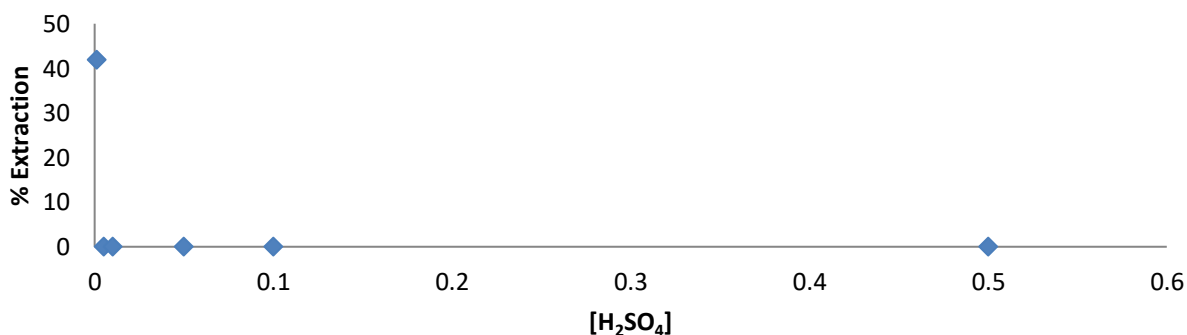


Figure 8: Percentage extraction chart of UO_2^{2+} from 5×10^{-2} M HTCP solutions of H_2SO_4 containing 10^{-1} M SO_4^{2-}

A gradient of 2 was achieved suggesting the displacement of 2 protons in the course of the reaction. The % A was 41.87 % at concentration 1×10^{-3} M. Figure 9 presents the % A chart of 200 ppm UO_2^{2+}

from 1 M SO_4^{2-} solutions of H_2SO_4 . No abstraction was obtained at any molar concentration as shown in Figures 7, 8 and 9.

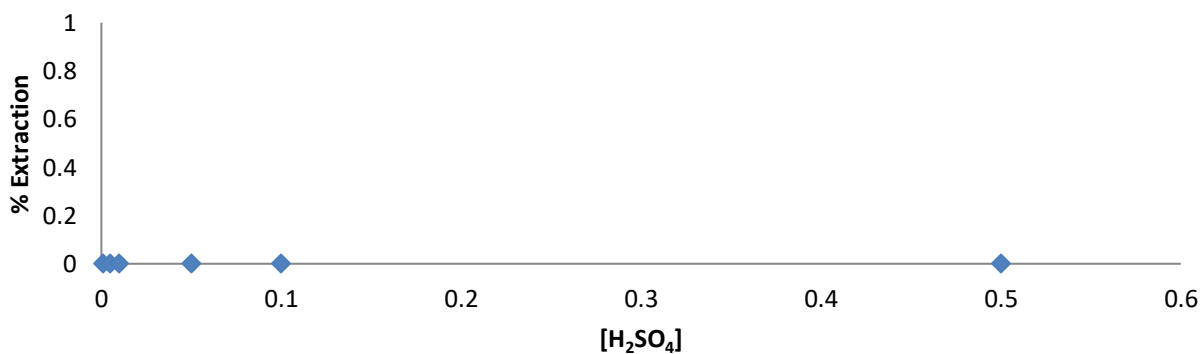


Figure 9: Percentage extraction chart of UO_2^{2+} from 5×10^{-2} M HTCP solutions of H_2SO_4 containing 1 M SO_4^{2-}

Interestingly, the results show that abstraction was optimal at sulphuric acid concentrations 1×10^{-3} , 1×10^{-2} and 1×10^{-1} M SO_4^{2-} ion concentrations, although the maximum % A of UO_2^{2+} decreased with increasing H_2SO_4 concentration in the buffered phase. 1×10^{-2} and 1×10^{-1} M SO_4^{2-} concentrations showed a gradient of 2, indicating that 2 protons were released when UO_2SO_4 and the ligand (HTCP) interacted. 1 M SO_4^{2-} ion concentration showed no abstraction at the concentrations studied as shown in Figure 9. This suggests that the abstraction of UO_2^{2+} from H_2SO_4 is more efficient at low molar concentrations (Sangoremi *et al.*, 2013).

Conclusion

In the abstraction study of UO_2^{2+} , the synergetic effect of three mineral acids, CH_3COOH , HCl and H_2SO_4 in HTCP in chloroform was examined. The study revealed that UO_2^{2+} was better abstracted at slight acid concentrations. Results obtained showed that the interference of chloride, acetate and sulphate ions was more obvious as the mineral acid concentrations got greater than 1×10^{-2} M. There was an optimum % abstraction of 94.71, 91.63 and 46.26 % from 1×10^{-3} M - 1 M CH_3COOH , and an optimum % abstraction of 80.62, 77.97, and 38.77 % at molar concentrations 1×10^{-3} , 1×10^{-2} and 1 M HCl .

A gradient of 1 was obtained with CH_3COOH and HCl , indicating a displacement of 1 proton during the reaction. An optimum % abstraction of 52.86 and 41.85 % at 1×10^{-3} M and 1×10^{-2} M H_2SO_4 was obtained. The calculated gradient was 2 at 1×10^{-3} and 10^{-2} M H_2SO_4 , indicating that 2 protons were released during the reaction. 1 M SO_4^{2-} ion concentration showed no abstraction at the molar concentrations studied. Thus, results indicate that CH_3COOH , HCl and H_2SO_4 are the only efficient abstraction media for UO_2^{2+} at slight concentrations. The observed increase in % abstraction of UO_2^{2+} from mineral acids follows the sequence: $\text{CH}_3\text{COOH} > \text{HCl} > \text{H}_2\text{SO}_4$, indicating that removal of UO_2^{2+} decreased as the acid strength increased.

Acknowledgement: The authors wish to immensely acknowledge Mr. Bennett Purae Inengite for his unalloyed moral and emotional support during the cause of this research and late Prof. B.A. Uzoukwu, our MSc supervisor of the university of Port Harcourt for his unprecedented mentorship.

Funding: This research work received no financial aid of any form from any funding body or institution.
Competing interests

Competing interests: None

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