

Kinetic study of tantalum extraction using beta-diketones

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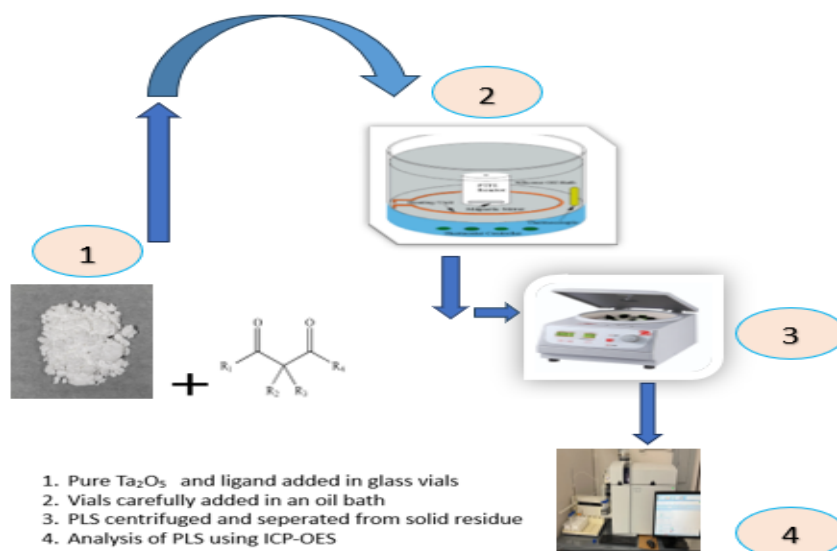
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Tantalum has attracted considerable attention as it is a high-tech metal promoting artificial intelligence through the production of various electronic components, hence extraction from its ores is necessary to fulfill the soaring demand for this precious metal. This work focuses on the kinetic study of a tantalum leaching process using beta-diketones. In the kinetic investigation, four organic ligands (beta-diketones) namely, acetylacetonate (ACAC), dipivaloyl methane (DPVM), hexafluoro acetylacetonate (HFAA), and trifluoro acetylacetonate (TFAA) were used to leach tantalum from pure tantalum(V)oxide. Experiments were carried out in a temperature range between 30 °C and the boiling point of the respective ligand. In this study, operating parameters including temperature, agitation speed, time, and solid-liquid ratio were used to assess their effect on tantalum recovery. In each trial, one parameter was varied whilst keeping others constant. The pregnant leach solution (PLS) was separated from the residue by centrifugation and then analyzed using ICP-OES and AAS techniques. Rate constants were obtained using the shrinking core model and the activation energy was derived based on the Arrhenius equation. According to the shrinking core model, linear relationships indicated that the rate of tantalum dissolution in HFAA was diffusion controlled. The rate of tantalum dissolution in TFAA was mixed controlled, whilst ACAC, and DPVM rate of dissolutions were confirmed to be chemical reaction controlled. Activation energies when using HFAA, TFAA, ACAC, and DPVM were found to be 16.8 kJ/mol, 27.9 kJ/mol, 51.6 kJ/mol, and 64.5 kJ/mol, respectively. The ligand with the highest yield was HFAA with 33 %, while the lowest yield of 10% was DPVM. These organic ligands may offer an alternative extraction route to tantalum recovery compared to the traditional one which uses conventional acids if the tantalum can be recovered from it in such a way that the ligands can be recycled.

Keywords: Organic ligand, Beta-diketones, Tantalum, Leaching, Kinetic study, Activation energy.

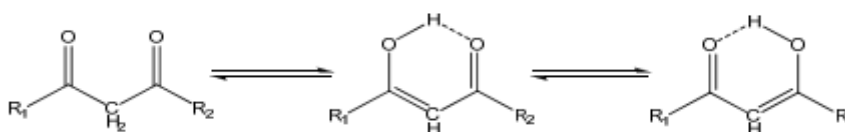
Graphical abstract



Graphical abstract to illustrate the extraction of tantalum using beta-diketones.

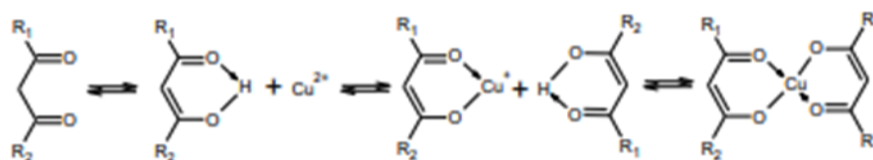
INTRODUCTION

Currently, tantalum has proved to be one of the most valuable metals (V.K.Balla et al., 2010). Forecasts have determined its demand through 2030 and beyond (Watari et al., 2020). Its value worldwide can be easily illustrated by conflicts and war among some African countries that are leading in the production of the precious metal, for example in regions such as Rwanda and the Democratic Republic of Congo (Montague, 2002). Processing of tantalum from its high-grade and low-grade tantalum ores using traditional ways has posed an environmental threat as more toxic substances are produced during the environmentally unfriendly processes (Drahansky et al., 2016). Several processes and techniques have been developed and used to extract tantalum from its ores such as hydrometallurgical extractions using hydrofluoric acid (HF) or a combination of H₂SO₄ and HF mineral acid dissolutions (Nzeh et al., 2022). Beta-diketones are potential alternatives that can be used to extract metals in aqueous solutions, (Sepúlveda et al., 2014). Researchers have proved that organic ligands such as beta-diketones can be used to leach different metals like copper (Cu), Zinc (Zn) and Iron (Fe) from their ores (Przeszlakowski and Wydra, 1982). Organic ligands such as beta diketones can be a substitute for acids used during tantalum extraction as these beta diketones possess preferred properties such as environmentally friendly, low melting, low boiling point, and high thermal stability. Different leaching models are used to describe the leaching kinetics when extracting metals depending on their suitability (Sun et al., 2018). Kinetic models such as the shrinking core model were used in gold extraction (Teimouri et al., 2020) and copper extraction (Quezada et al., 2020). In solutions, β-diketones in keto-enol tautomeric equilibrium give a resonance structure which is a six-membered ring depending on temperature, solvent polarity, temperature concentration, and structure of the compound (Urbaniak et al., 2011). This is illustrated by reaction 1 below.



reaction 1 (Urbaniak et al., 2011).

Decades ago, researchers and scientists successfully extracted metals using alkyl-substituted beta-diketones (Netherlcuidr et al.,1969). In the case of copper extraction, it was found that beta-diketones form chelate complexes with Cu (II) ions (Witt and Radzaminska-Lenarcik, 2017) as shown in reaction 2 below. Witt and Radzaminska-Lenarcik (2017) also concluded that beta-diketones containing an aromatic ring in their molecule e.g. benzoyl acetone and dibenzoyl methane, are more effective extractants than acetylacetone, with dibenzoyl methane being the most effective. Beta-diketones with halogen atoms attached to them can make them react effectively compared to those with beta-diketones without (Gutiérrez-Quintanilla et al., 2023).



reaction 2 (Gutiérrez-Quintanilla et al., 2023)

Reaction 2 above illustrates how beta-diketones react with most metal ions, for example, copper ions. It can be noticed that the reaction is reversible such that conditions that favour forward reactions should adhere to achieve the desired metal complex. Similar work on establishing how tantalum can be recovered using beta-diketones will be carried out in this study.

EXPERIMENTAL PROCEDURE

Tantalum oxide sample

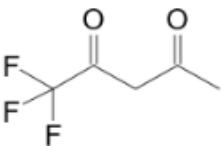
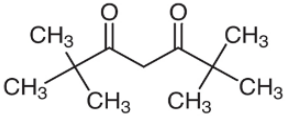
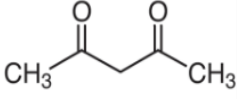
The pure tantalum (V) oxide sample purchased from Merk South Africa was used in this experiment. The tantalum oxide was analyzed using the XRD technique to confirm purity. Bruker D2 Phaser equipped with EVA software was used to characterize and ensure the sample crystallinity. The tantalum oxide sample lattice was found to be orthorhombic. Pure tantalum oxide with purity of 99.6% was used in all experiments. Furthermore, pure tantalum oxide was mixed with silica sand to vary tantalum oxide composition in solid samples within a range of 2 - 100% maintaining the solid- liquid ratio of 1:10 when running experiments.

Reagents

Organic ligands that were used in this experiment were acetylacetone (ACAC), hexafluoro acetylacetone (HFAA), dipivaloyl methane (DPVM) and trifluoro acetylacetone (TFAA). All these reagents were purchased from Sigma and Aldrich South Africa. When purchased purity of acetylacetone, hexafluoro acetylacetone, dipivaloyl methane, and trifluoro acetylacetone was 99.9%, 99%,99.9%, and 99% respectively.

Table 1: Material used in this study with their purities and suppliers.

Name	Structure	Boiling Point	Supplier and Purity
Hexafluoro acetylacetone (HFAA)		70°C	Sigma and Aldrich-99%
Trifluoro acetylacetone (TFAA)		106 °C	Sigma and Aldrich-99%

		
Dipivaloyl methane (DPVM)		72 °C
Acetylacetone (ACAC) c		140 °C
		Sigma and Aldrich-99.9%
		Sigma and Aldrich-99.9%

Leaching experimental method.

Ligands were measured and pure tantalum oxide was weighed before being carefully added to 30 ml glass vials depending on the solid-liquid ratio required for experiments ranging between 1:1 and 1:10. Each vial contained a magnetic stirring rod. Vials were tightly closed to avoid escape of any reactant or product and each was carefully immersed into the pre-heated silicone oil bath placed on a magnetic stirrer using a vial holder. Experiments were carried out at temperatures relative to the boiling points of each beta-diketone since the beta-diketones used in this study have different boiling points. For all trials, the operating temperatures ranged between 30 °C and the boiling point of each respective ligand. For every trial, each operating parameter was kept constant whilst varying others. The parameters considered in this experiment were time, temperature, agitation speed, and solid-liquid ratio. Pregnant leach solution (PLS) was separated from the residue by centrifugation, collected using a syringe fitted with a 45µm filter, and then analyzed using ICP-OES and AAS techniques. Furthermore, the same procedure mentioned above was done for pure tantalum oxide mixed with silica sand at various proportions to vary tantalum oxide composition within a range of 2 - 100%.

KINETIC MODELING

Kinetic study

Reaction rates can be affected by concentration, temperature, and pressure among other factors (Karsli and Ayas, 2014). In this study, the following assumptions will be adhered to; all solid particles were the same, the concentration of reagents will not affect dissolution kinetics, first order on the surface, constant concentration of the reagents and lixiviant is covering entire particles. There are different types of models such as the shrinking core model (SCM), shrinking particle model (SPM), Avram model, and empirical model amongst others. Most suitable models can be selected based on assumptions and the nature of reactants. In this study shrinking core model was selected. Previous researchers used the SCM rate equation for a surface reaction-controlled process in the conversion of tantalum pentoxide with carbon tetrachloride (Kim and Choi, 2005). The shrinking core model is used to describe situations in which solid particles are being consumed either by dissolution or reaction resulting in the amount of the material being consumed 'shrinking' for example dissolution of pills in the stomach (Limitations and Systems, 1972). A solid-liquid interface formed allows reactant A to diffuse through reacting with reactant B. Using the SCM Equation 2 for a surface reaction-controlled process, the conversion of pure Ta₂O₅ can be related to reaction time. Equations 1 and 3 can also be used for dissolution kinetics of tantalum using beta-diketones.

Shrinking core model (SCM).

Various fields of science and engineering have used process kinetics to come up with fundamental knowledge enabling a deep understanding of biochemical, chemical metallurgical, processing, and petrochemical engineering. Generally, time is a very important factor in many processes as it is aligned with economic leverage and profitability amongst many other factors. Kinetic studies also help us in investigating the rate of chemical reactions and they are a critical tool in the design of processes. Heterogeneous reactions including the leaching of metals can be very complex such that investigating kinetics can be a tremendous step to understand the mechanisms involved in processes. The shrinking core model was employed and used by applying the equations below:

$$1 - 3(1 - x)^{\frac{2}{3}} + 2(1 - x) = K_d t \quad \text{Eq. 1}$$

$$1 - (1 - x)^{\frac{1}{3}} = K_r t \quad \text{Eq. 2}$$

$$1 - \left(\frac{2}{3}\right)x - (1 - x)^{\frac{2}{3}} + \frac{K_r}{K_d} (1 - (1 - x)^{\frac{1}{3}}) = K_m t \quad \text{Eq. 3}$$

Where k_d , k_r , and k_m are rate constants for diffusion, chemical reaction, and mixed control respectively. Equations 1, 2 and 3 represent diffusion-controlled, chemical reaction controlled, and mixed-controlled respectively. The process can either be diffusion-controlled or chemical-controlled and, in some instances, mixed-controlled. Changes in concentration with time establish rate of reactions and activation energies can be calculated using the Arrhenius equation shown in equation 4 below using graphs:

$$\ln k = \ln A - \frac{E_A}{RT} \quad \text{Eq. 4}$$

Where E_A is the activation energy.

k is the pre-exponential factor

R is the universal gas constant

T is the absolute temperature in K

Generally, activation energies more than 40 kJ/mol are chemical reaction controlled, and less than 40 kJ/mol are diffusion controlled (Teimouri et al., 2020). The shrinking core model assists in generating data that can be used to calculate activation energy.

RESULTS AND DISCUSSION

The effect of temperature, agitation speed, solid-liquid ratio, and time on tantalum extraction using beta-diketones was assessed during all experimental runs. In the case of tantalum recovery using beta diketones, optimizing the experimental conditions practically adds value to the extraction process and plays a pivotal role in all economic aspects of tantalum production. Figure 1 shows that HFAA performed better than TFSA, ACAC, and DPVM in their optimized conditions. Temperature had the greatest effect on ta extraction compared to other conditions including agitation speed and solid-liquid ratio. Percentage tantalum extraction using beta-diketones at time intervals ranging between 10 minutes to 18 hours is shown in Figure 1 below.

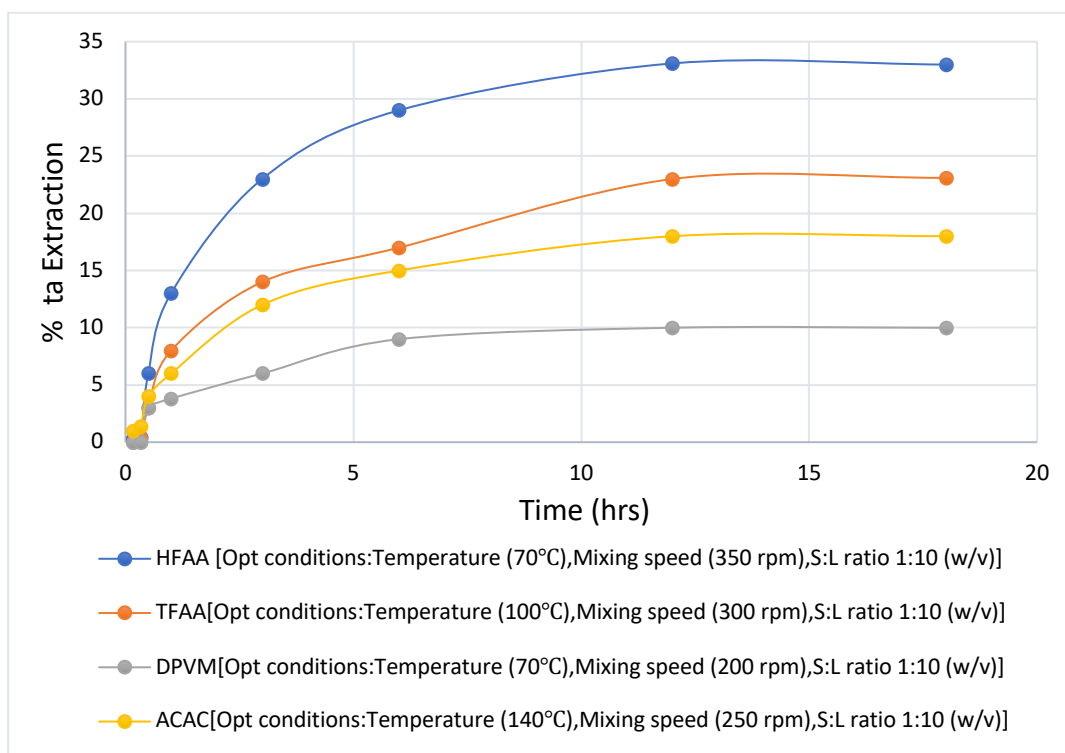


Figure 1. Percentage of tantalum extraction when using beta-diketones at different optimum conditions.

Effect of leaching agent

The type and concentration of leaching agents have proven to be very important parameters influencing the rate of leaching (Sayan and Bayramoglu, 2004). The effect of leaching also varies with leaching mechanisms such as acidolysis, complexolysis and redoxolysis (Wu & Ting, 2006). The type of leaching agent can also change the kinetics and mechanisms of a process. Mostly when leaching with organic liquids complexolysis is experienced (Dusengemungu et al., 2021). In this study, organic ligands including HFAA, TFAA, DPVM, and ACAC were compared to find out which organic ligand extracts the most tantalum. The order of extraction was as follows HFAA > TFAA > ACAC > DPVM. The highest extraction was from HFAA extracting 33 % tantalum followed by TFAA at 23%, ACAC was 18% and DPVM was 10%. HFAA and TFAA are halogenated analogues of acetylacetone. Regarding acetylacetone and its halogen-derivatives, intramolecular hydrogen bond strength changes as follows F6-ACAC < F3-ACAC < ACAC. The O-O bond length and O-H-O bond angle are longer and smaller respectively in HFAA than in ACAC explaining how HFAA has more affinity for metal ions compared to ACAC (Buemi, 2000). Additionally, strong repulsive effects between two CF₃ causes a non-planar in HFAA. Some work is still in progress as it was established by researchers that the addition of methanol to beta diketones in certain ratios increases metal extraction (McGeachin, 1968). Also, Beta-diketones with a benzene ring showed better extractions in the case of copper.

Effect of temperature

Temperature is a very critical factor in leaching kinetics. It can accelerate diffusion rate, reduce viscosity, and affect solubility to the extent of changing the direction of reversible reactions. In most cases, chemical reactions are more sensitive to temperature than diffusion. In this study temperature showed a significant role in tantalum extraction experiments using beta-diketones. Experiments were carried out at temperatures relative to the boiling points of each beta-diketone since the beta-diketones used in this study have different boiling points. Each chosen operating temperature was relative to the boiling point of the respective ligand. After using HFAA, TFAA, DPVM and ACAC to leach tantalum, it was found that extraction using all the ligands increased with an increase in temperature. HFAA boiling at 70°C showed better tantalum extraction of 33%. TFAA which boils at 106 °C followed with a tantalum extraction of 23%. Economically, HFAA proved to be a better leaching extractant than the other beta-diketones since less energy is required to heat the ligand whilst obtaining higher extraction levels.

Effect of agitation

The agitation effect is more significant when the leaching rate is under diffusion control and less significant when the chemical reaction controls the leaching kinetics (Ramadan et al., 2016). In this study, agitation was varied between the range of 100 - 700 rpm. Focusing on HFAA as the best ligand, at a lower agitation speed of 100 rpm, extraction of tantalum was very low, but extraction increased to 33% at agitation speeds of 350 rpm. Optimum agitation speed for TFAA, ACAC and DPVM was 300 rpm, 250 rpm and 200 rpm respectively. Particle size is also an important parameter that inversely affects the leaching rate (Zhou et al., 2005). In this study pure Ta₂O₅ powder with uniform particle size was used.

Solid ratio

The solid-liquid ratio of pure tantalum oxide to each beta-diketone was varied within the range of 1:1 and 1:10. The solid-liquid ratio of 1:10 performed very well for HFAA as a maximum extraction of 33% was obtained at this ratio. It was also established that minimum extraction was noticed at a 1:1 solid-liquid ratio for all ligands as the liquid would not be enough to extract much tantalum. At solid-liquid ratios below 1:5, ligand saturation would quickly be reached hindering extraction. Regarding the four ligands used in this study, the highest extraction of 33% was obtained by HFAA at a solid ratio of 1:10.

Kinetic Study Results

Interpretation of the rate data

According to this study, it was determined that rates of leaching experienced both diffusion and chemical reaction controlled when all beta-diketones were used. Equations 1,2 and 3 above representing diffusion, chemical reaction and mixed control were used to determine the rate constant K_d , K_r and K_m . The rate constant for each respective ligand was then used to determine the activation energy in conjunction with the Arrhenius equation.

Table 2: K_d , K_r , and K_m values for HFAA at different temperatures ranging from 30 to 70 °C

Ligand	Temperature °C	Diffusion Control	Chemical Reaction Control	Mixed Control
		$1 - 3(1-x)^{\frac{2}{3}} + 2(1-x) = K_d t$	$1 - (1-x)^{\frac{1}{3}} = K_r t$	$1 - \left(\frac{2}{3}\right)x - (1-x)^{\frac{2}{3}} + \frac{K_r}{K_d}(1 - (1-x)^{\frac{1}{3}}) = K_m t$
		K_d value	K_r value	K_m value
HFAA	30	0.0002	0.0036	0.016
	40	0.0060	0.0010	0.0194
	50	0.0015	0.0061	0.0204
	60	0.0022	0.0034	0.0260
	70	0.0068	0.0144	0.0370

Table 3: K_d , K_r and K_m values for TFAA at different temperatures from 30 to 100 °C

Ligand	Temperature °C	Diffusion Control	Chemical Reaction Control	Mixed Control
		$1 - 3(1-x)^{\frac{2}{3}} + 2(1-x) = K_d t$	$1 - (1-x)^{\frac{1}{3}} = K_r t$	$1 - \left(\frac{2}{3}\right)x - (1-x)^{\frac{2}{3}} + \frac{K_r}{K_d}(1 - (1-x)^{\frac{1}{3}}) = K_m t$
		K_d value	K_r value	K_m value
TFAA	30	0.0001	0.0015	0.0202
	40	0.0003	0.0026	0.0322
	50	0.0015	0.0062	0.0391
	60	0.0017	0.0064	0.0491
	70	0.0042	0.0103	0.0818
	100	0.0071	0.0194	0.1653

Table 4: K_d , K_r , and K_m values for ACAC at different temperatures from 30 to 140 °C

Ligand	Temperature °C	Diffusion Control	Chemical Reaction Control	Mixed Control
ACAC		$1 - 3(1-x)^{\frac{2}{3}} + 2(1-x) = K_d t$	$1 - (1-x)^{\frac{1}{3}} = K_r t$	$1 - \left(\frac{2}{3}\right)x - (1-x)^{\frac{2}{3}} + \frac{K_r}{K_d}(1 - (1-x)^{\frac{1}{3}}) = K_m t$
		K_d value	K_r value	K_m value
	30	0.0001	0.1881	0.0063
	40	0.0002	0.1356	0.0160
	50	0.0008	0.1483	0.0263
	60	0.0013	0.1318	0.0448
	70	0.0034	0.1306	0.0730
	140	0.0069	0.1302	0.2967

Table 5: K_d , K_r , and K_m values for DPVM at different temperatures from 30 to 70 °C

Ligand	Temperature °C	Diffusion Control	Chemical Reaction Control	Mixed Control
DPVM		$1 - 3(1-x)^{\frac{2}{3}} + 2(1-x) = K_d t$	$1 - (1-x)^{\frac{1}{3}} = K_r t$	$1 - \left(\frac{2}{3}\right)x - (1-x)^{\frac{2}{3}} + \frac{K_r}{K_d}(1 - (1-x)^{\frac{1}{3}}) = K_m t$
		K_d value	K_r value	K_m value
	30	0.0005	0.0010	0.0020
	40	0.0001	0.0018	0.0041
	50	0.0003	0.0017	0.0091
	70	0.0020	0.0073	0.0489

Activation energy

All beta-diketones used in this study showed that temperature played a critical role in tantalum recovery using beta-diketones. Table 6 below shows that HFAA requires the least activation of 16.8 KJ/mol, TFAA requires 27,9 kJ/mol, ACAC 51,6 kJ/mol, and DPVM 64.6 kJ/mol. In most industrial setups, energy efficiency is a critical factor in terms of reducing the cost of running processes. HFAA requires less activation energy compared to ACAC, DPVM, and TFAA. The general equation $y = mx + c$ was used to match the Arrhenius equation shown in equation 4 followed by the calculation of activation energy.

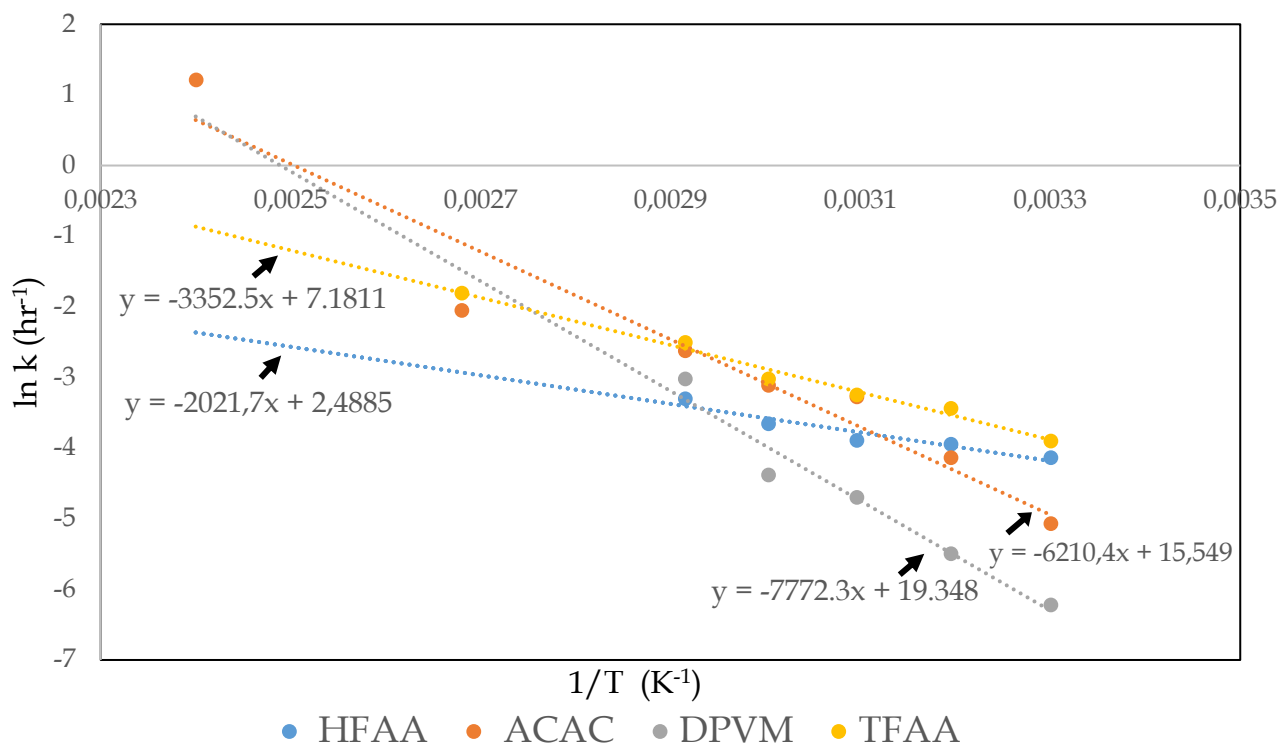


Figure 2: Arrhenius plot for tantalum extraction using beta-diketones (HFAA, ACAC, DPVM, and TFAA)

Table 6: Beta-diketones HFAA, TFAA, DPVM, and ACAC showing respective activation energies required for tantalum extraction.

Ligand name	Activation energy (Ea)
HFAA	16.8 KJ/mol
TFAA	27.9 KJ/mol
ACAC	51.6 KJ/mol
DPVM	64.6 KJ/mol

CONCLUSION

Beta-diketones have proven that they are useful in tantalum recovery processes. In this study, temperature played a pivotal role when extracting tantalum using all four beta diketones. Agitation speed, solid-liquid ratio, and time also affected tantalum extraction. The effect of particle size is a very important factor to consider in such studies, however, it was not investigated as pure oxide powder from the supplier was used, which had a single uniform particle size. The order of extraction was as follows HFAA > TFAA > ACAC > DPVM. The highest tantalum extraction was 33 % from HFAA followed by TFAA (23%), ACAC (18%), and DPVM (10%). It was also observed that HFAA reacted better with the beta-diketones due to longer O-O bond length and smaller O-H-O bond angle. The highest extraction was obtained by HFAA at the following conditions: temperature (70°C), mixing speed (350 rpm), and solid-liquid ratio of (1:10) after 12 hours. HFAA required 16.8 KJ/mol of activation which is less than the activation energy required by TFAA, ACAC, and DPVM supporting the economic aspect of using HFAA, as less heat energy is required by the extraction process compared when using the other

three ligands. Further work will be undertaken using beta-diketones consisting of benzene rings. Also adding a form of alcohol to beta-diketones will be assessed to increase chances of better tantalum extraction.

ACKNOWLEDGEMENTS

The authors acknowledge Afritin Mining Company for their support in funding the project. The University of Witwatersrand Chemical and Metallurgical Engineering Department is thanked for its involvement in making this study a success.

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