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Extraction of Tantalum from locally sourced Tantalite using Polyethylene glycol solution

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ABSTRACT: The ability of polyethylene glycol solution to extract tantalum from locally sourced tantalite ore was studied. The amount of tantalum pentoxide extracted increased with increase in concentration of Polyethylene glycol (PEG) solution from 28.08% to 60.23%. However, the amount of niobium pentoxide extracted increased from 0.1194% to a maximum of 40.52% at increasing concentration of PEG and dropped to 26.1477% at PEG solution concentration of 60%. The separation factor ß of tantalum pentoxide and niobium pentoxide at 30, 40, 50, and 60% PEG concentrations were 0.0031, 0.3110, 0.7874 and 0.2338 respectively. The distribution coefficient (D1) of tantalum pentoxide at 30, 40, 50 and 60% PEG concentrations were 0.3904, 0.6477, 0.8651 and 1.5145 respectively; while at 30, 20, 30, 40, 50 and 60% PEG concentration the distribution coefficients (D2) of niobium pentoxide were 0.0012, 0.2014, 0.6812 and 0.3541 respectively.

KEYWORDS: Tantalum, tantalite, extraction, niobium, polyethylene glycol (PEG).

INTRODUCTION

Regulatory pressure is increasingly focusing on the use, manufacture and disposal of organic solvents, and thus the development of non-hazardous alternatives for the continued and sustainable development of the chemical enterprise. There are many potential advantages of replacing volatile organic compounds (VOC) with water or various types of aqueous solutions. The most obvious are low cost, reduced flammability, reduced toxicity and reduced environmental risk as a result of discharges of the supporting phase (Chen *et al.,* 2005). Water and aqueous-based solvent systems may represent increasingly significant choice for the replacement of traditional solvents. It is in view of these, that this work was conducted on polyethylene glycol (PEG) solution-molecular weight 3,350 (PEG3,350) in aqueous biphasic (a system formed when a water soluble polymer e.g. PEG is mixed with certain water-structuring salts e.g., $(NH_4)_2SO_4$, K_2CO_3, etc. (Rogers *et al*, 1998) reactive extraction of tantalum from locally sourced tantalite as an alternative path to the traditional volatile organic compounds used e.g. hexone, Tributyl phosphate (Habashi, 1968; Hedrick *et al.*,, 1997). The interest on PEG is because of its environmental friendliness and the availability of complete toxicity profiles for a range of PEG molecular weights and chemical reaction (Chen *et al.*, 2005). The research was aimed at using polyethylene glycol solution to extract tantalum from locally sourced tantalite. PEG is a condensation polymer of ethylene oxide and water with the general formula $H(OCH_2CH_2)_nOH$, where n is the average number of repeating oxyethylene group typically from 4 – 180 (wikipedia, 1998), and with molecular weight ranging from 200 to tens of thousands. At room temperature, the water soluble and hygroscopic polymer is a colourless viscous liquid at molecular weight < 600 and a waxy, white solid at molecular weight > 800. Liquid PEG is highly soluble in water, for example, PEG-2000 has a solubility of about 60% in water at 20^oC (Chen *et al.*, 2005), and can be used as solvent in their own rights with or without addition of water. Unlike VOCs, low molecular weight liquid PEGs are non-volatile. PEG also has low flammability and it is biodegradable (Chen *et al.*, 2005), it also undergoes various reactions including salt formation and can be used for solvent extraction (Parker, 1982). It may be recovered from aqueous solution by extraction with a suitable solvent or by direct distillation of water or solvent. Aqueous PEG solution may display phase separation under controlled conditions. Such phase separated solutions are known as Aqueous Biphasic systems (ABS) or alternatively Aqueous Two-phase systems

(ATPS). Many factors affect phase catalytic activity, such as PEG molecular weight chain end effect, and the nature of associated cations and anions. PEG and its aqueous solutions represent interesting solvent system for solvent replacement and may stand in comparison to other currently favoured systems such as ionic liquids, supercritical carbondioxide and micellar systems. PEG is hydrophilic polymer, easily soluble in water and many organic solvents including; toluene, dichloromethane, alcohols and acetones but it is not soluble in aliphatic hydrocarbons such as hexane, cyclohexene etc (Chen *et al.,* 2005).

EXPERIMENTAL

The tantalite ore was procured from the market in Jos, Plateau state. It was crushed to particle size of 300 microns using Retsch crushing machine, type BB1 Masch No 70992. This was ground in a grinding machine. The ground ore was then sieved using ASTM testing sieve No 120 to an oversize of 125 microns. The undersize particles of the ground ore was ball milled at an hourly interval of 2, 4, 6 using medium ball mills (Suesterberg machine No 6083). This sample was then sieved to particle size of 75µm (200mesh) using sieve (Test sieve B.S 410/43, endecotts filters Mesh No 72 PAT) and then taken to Center for Energy Research and Training, Ahmadu Bello University Zaria for elemental/compositional analysis using Energy dispersive X-ray Florescence.

Fifty grammes (50g) of the six hours ball milled sample was treated with 81.6 ml of 98% tetraoxosulphate six acid at an acid/ore ratio of 3:1 and then with 25M solution of HF to improve the dissolution of the ore at an acid/acid ratio of 1:1 (El Hussain *et al.,* 2004). The solution was then heated for three hours at 80°C to improve the leaching process in a conical flask. The leached liquor was then decanted and the sample taken for elemental/compositional analysis at the Center for Energy Research and Training Ahmadu Bello University, Zaria using X-ray fluorescence method.

The PEG solution was prepared by dissolving polyethylene glycol (MW of 3350) in water to make the required concentrations (20, 30, 40, 50 and 60%). To each of these 3.5M Ammonium sulphate solution was added to make a PEG/salt ratio of 1:1 (Chen *et al.*, 2005) and the samples were shaken for 30 seconds to facilitate mixing. The decanted feed solution was then divided into 5 parts and each of the respective parts was treated with 10, 30, 40, 50 and 60% concentrated PEG/Salt solutions at leached liquor/PEG solution ratio of 1:1 and shaken for 30 seconds. The PEG-rich phase has more of the PEG and is expected to extract more of the tantalum in the phase. The PEG-rich phase was then separated from the salt- rich phase in a separating funnel after attaining equilibrium. The loaded PEG-rich phase was then taken to Center for Energy Research and Training Ahmadu Bello University, Zaria for elemental/compositional analysis using XRF. The results are shown in Figures 1 and 2.

RESULTS AND DISCUSSION

Table 1 shows the composition of the ball milled ore. Tantalum pentoxide (29.06 - 30.75%) is the major constituent of the ore and hence it can be called tantalite (Habashi, 1968); even though it contains other compounds like niobium pentoxide (15%), iron oxide (12%) etc. Particle size is significant since it is a direct function of the total surface area that will be available for reaction (Othman, 1980; Parker, 1982).

Table 1: Compositional analysis of the ball milled ore

Compounds	2hr	4 hr	6 hr
TiO_2 (%)	11.9 ± 1.770	9.16 ± 1.619	11.88 ± 1.662
Mn_2O_3 (%)	4.57 ± 0.435	3.69 ± 0.389	2.59 ± 0.956
Fe_2O_3 (%)	11.59 ± 0.487	11.32 ± 0.458	11.86 ± 0.476
Ta_2O_5 (%)	29.55 ± 0.952	30.04 ± 0.806	29.06 ± 0.830
Nb_2O_5 (%)	15.45 ± 0.180	15.39 ± 0.158	15.31 ± 0.175
Y_2O_3 (ppm)	340 ± 0.013	740 ± 0.013	640 ± 0.013
U_3O_8 (ppm)	890 ± 0.021	1120 ± 0.021	980 ± 0.020
ZrO_2 (ppm)	2690 ±0.018	330 ± 0.017	2910 ± 0.019

Table 4 show the leaching of tantalum pentoxide (30.75%) and niobium pentoxide (14.33%) which was achieved as predicted by El Hussain and Rice (2004) using Acid/Ore ratio of 3:1.

Table 4: Compositional analysis of Leached liquor.

Compounds	Composition
Fe_2O_3 (%)	1.119 ± 0.0381
Ta_2O_5 (%)	30.746 ± 1.2650
Nb_2O_5 (%)	14.33 ±1.6851
Y_2O_3 (ppm)	40 ±1.0765
ZrO_2 (ppm)	126 ± 0.00
TiO_2 (ppm)	4056 ± 0.00
Mn_2O_3 (ppm)	757 ± 0.00

Table 5 gives the distribution coefficient of tantalum pentoxide (D1) and niobium pentoxide (D2). D1 increased with increase in concentration of PEG from 0.3904 at 30% PEG, 0.6477 at 40% PEG, 0.8651 at 50% PEG and finally reaching its maximum of 1.5145 at 60% PEG concentration. However, the distribution coefficient D2 increased from 0.00195 at 30% PEG to 0.20144 at 40% PEG concentration reaching its maximum of 0.68120 at 50% PEG and decreased to 0.35405 at 60% PEG concentration.

The separation factor is a measure of the ability of the system to separate two solutes. Table 5 gives the Separation factor ß as 0.0031, 0.3110, 0.7874 and 0.2338 for 30, 40, 50, and 60% PEG solutions respectively.

Table 5: Distribution coefficient of Ta_2O_5 (D1) and Nb_2O_5 (D2) at different PEG concentration

Concentration of PEG (%)	Concentration in salt rich phase (fraction)		Concentration in peg rich phase (fraction)		D1	D2	ß
	Ta_2O_5	Nb_2O_5	Ta_2O_5	Nb_2O_5			
30	0.0935	0.05004	0.0365	0.0000598	0.3904	0.0012	0.0031
40	0.0789	0.0417	0.0511	0.0084	0.6477	0.2014	0.3110
50	0.0697	0.0298	0.0603	0.0203	0.8651	0.6812	0.7874
60	0.0517	0.037	0.0783	0.0131	1.5145	0.3541	0.2338

$D=\frac{\text{Concentration in PEG rich phase}}{\text{Concentration in salt rich phase}}$; Where D is the distribution coefficient (Fontana et al., 2000)

ß = D2/D1, where ß is the separation factor (www.wikipedia.org/wiki/..., 2007)

From Fig. 1 it can be seen that the percentage of tantalum pentoxide extracted increased with increase in PEG concentration (Habashi, 1968). At 30, 40, 50, and 60% PEG concentration 28.08%, 39.31%, 46.38 and 60.23% of tantalum pentoxide were extracted respectively. The percentage of niobium pentoxide extracted which varied from 0.12% at 30% PEG concentration to 16.77% at 40% PEG concentration reaching its maximum of 40.52% at 50% PEG concentration and then at 60% PEG concentration it decreased to 26.15% (Fig. 2). The sudden drop from 40.52% to 26.15% of the niobium pentoxide, for now could not be ascertained. Figure 3 gives the Extraction Isotherm, which is a straight line graph with slope of -1 and an intercept of 0.13 as also given by Habashi, (1968) and Othman, (1980). Such a plot is very useful, since at each point it directly displays the concentration in both phase and consequently the distribution coefficients can be evaluated. A plot of log of PEG concentration (LogC) against Log D1 gave a straight line graph with a slope of 1.8688 and 0.5545 intercept (Habashi, 1968). The distribution coefficient shows the effect of extractant concentration on the process. The higher the value of D at equilibrium, the higher is the extractability of the metal ion in question by the particular extractant. The time required to reach equilibrium increased with increase in concentration of PEG (Figure 5).

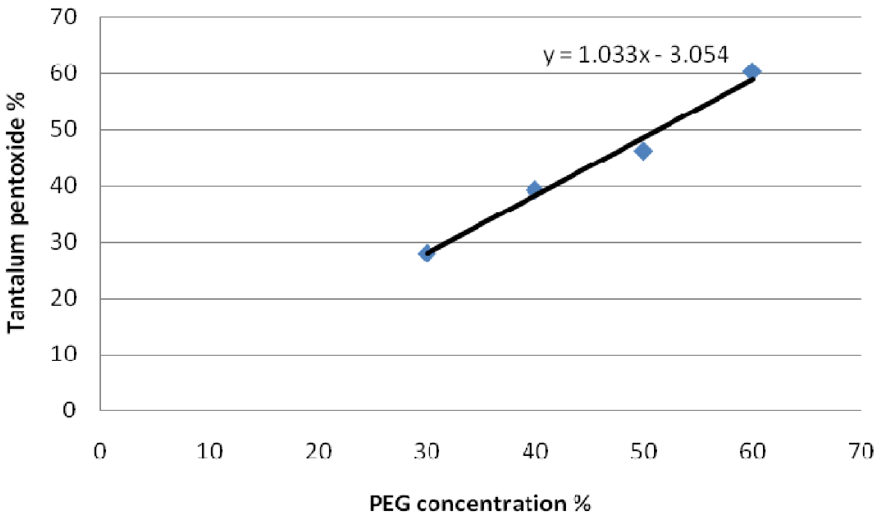

Figure 1: Tantalum pentoxide extracted against PEG concentration

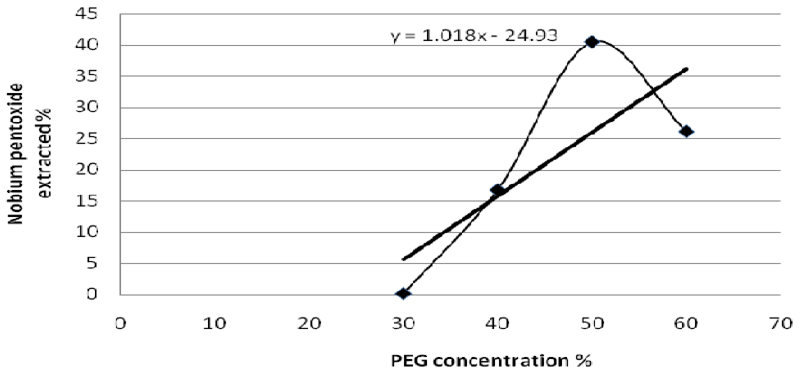

Figure 2: Niobium pentoxide extracted against PEG concentration.

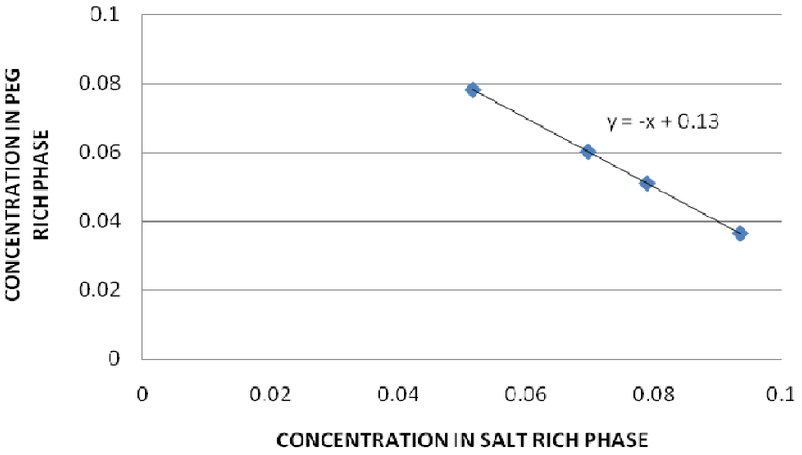

Figure 3: Extraction Isotherm for Tantalum pentoxide.

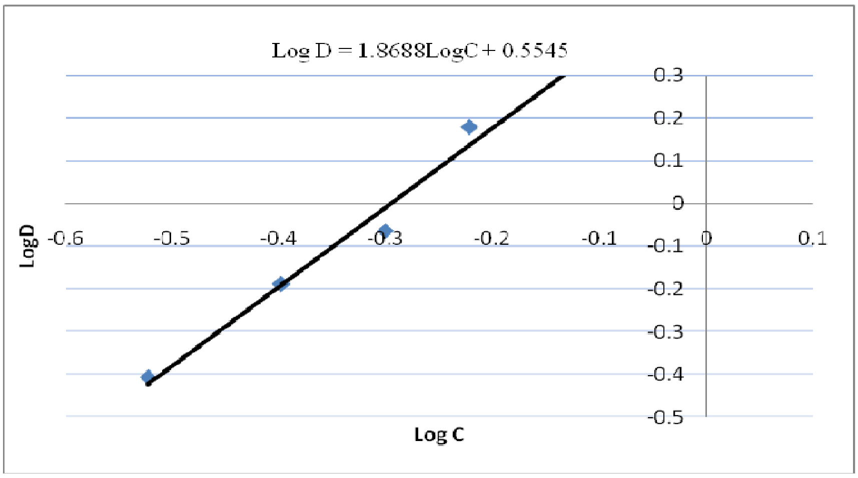

Figure 4: Log of PEG concentrations (LogC) against Distribution coefficient (LogD1)

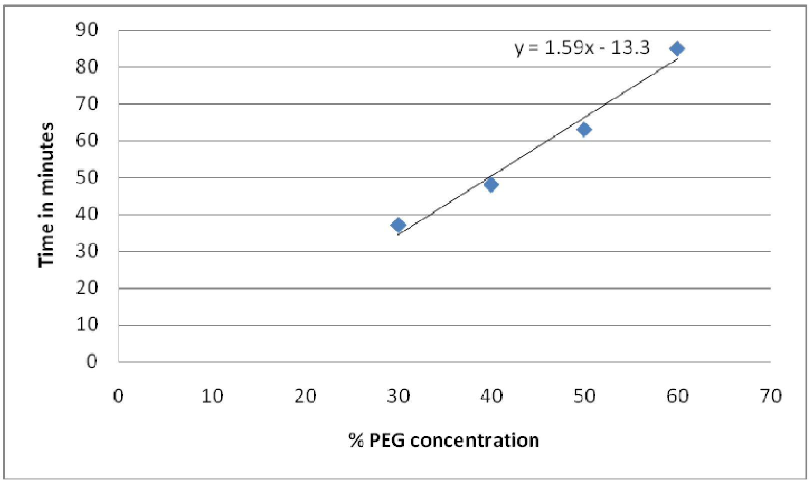

Figure 5: Equilibrium time graph

CONCLUSION

The following conclusion can be drawn from the work: PEG solution can be used to extract tantalum from tantalite through the formation of an aqueous biphase with ammonium sulphate.

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