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The effects of bi-metal extractions on separation factors using 4, 4'-(1*e*, 1*e*')-1, 1'-(ethane-1, 2-diybis (azan-1-yl-1ylidene)) bis (5-methyl-2-phenyl-2, 3-dihydro-1*h*-pyrazol-3-ol)

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Abstract

The effect of the presence of a second metal on the theoretically calculated Separation Factors β_{TXY} from distribution ratios obtained in the metal alone extractions from aqueous solutions buffered to either pH 4.75 or 7.5 containing 0.001 M and 0.005 M of either H_3PO_4 or H_2SO_4 using the Schiff base ligand 4,4'-(1*E*,1*E*')-1,1'-(ethane-1,2-diybis(azan-1-yl-1ylidene))bis(5-methyl-2-phenyl-2,3-dihydro-1*H*-pyrazol-3-ol) (H_2BuEtP) alone and 0.1 M Oxalate ion using a mixed ligands H_2BuEtP - $HBuP$ in 9:1 volume ratio was studied by comparing with Separation Factors β_{EXY} obtained from distribution ratios from bimetal extractions of a pair of metals from Cadmium, Iron, Nickel and Lead under same aqueous media condition using same organic phases. 60 minutes equilibration time was used and 2 batches of extractions were done based on calculated n batches of extractions needed to obtain 99.9% extraction of Cadmium which had the highest distribution ratios in the metals alone extractions. The aqueous raffinate were analysed using colorimetric method for iron with 1, 10-phenanthroline and the other metals with atomic absorbance spectrophotometer and distribution ratios calculated by difference. All the metals that were masked in their metal alone extraction were all extracted above 82% with all distribution ratios in the bimetal extractions statistically significantly different from those obtained in the metal alone extractions resulting in experimental Separation Factors β_{EXY} all < 5 . The results indicated that the ligand H_2BuEtP has more potentials for multimetal extraction than in the separation of Cadmium from Iron, Nickel and Lead. pH 7.5 bimetal extraction results were far better than those for pH 4.75 and it was recommended that multimetal extractions with other metals be studied with the ligand H_2BuEtP .

Keywords: Extraction; aqueous phase; organic phase; distribution ratio; separation factor; ligand

Introduction

Heavy metals have a wide range of applications in the industries, medicine and agriculture and thus have generated lots of environmental concerns resulting from their reported high toxicity (Luevano and Damodaran, 2014; Kamran *et al.*, 2016) ^[14, 11] and most researches have been geared towards efficient methods for removing these metals from the environment (Vodyanitskii, 2006; Gunatilake, 2015) ^[21, 10].

However, with the continuous discovery of new high-tech applications of these heavy metals and the high cost of exploration and extraction of these metals, there is an urgent need for developing efficient methods for recycling these metals. Studies have also shown that separation of metals with a parent/daughter relationship can also be utilized for dating (Godwin and Uzoukwu, 2013) ^[8] and thus, there is a need to look for efficient methods for their separations.

Schiff bases, which are a class of ligands with carbon double bonded to nitrogen (C=N), have been extensively used in metals extraction studies (Al Zoubi, 2013; Nworie and Nwabue, 2014) ^[1, 15] and many have been reported to be excellent heavy metal extractants due to the high hydrophobicity and stability of the metal complexes formed by these class of ligands with the studied heavy metals (Wong *et al.*, 1999; Kostova and Saso, 2013) ^[22, 13]. The extraction of a particular metal by a Schiff base is dependent on the following factors; Equilibration time, oxidation state of the metal, pH of aqueous solution containing the metal ion, concentration of ligand, presence of salting out, and masking agents (Uzoukwu, 2009) ^[20].

The most important parameters in these extraction studies are the distribution ratio, D , and percentage extraction (% E). The distribution ratios of two metals (X and Y) being studied under the same conditions with same ligand organic phase can be utilized in determining the separation factor β_{XY} for the two metals where $\beta_{XY} = D_X/D_Y$. A separation factor, $\beta_{XY} \geq 1 \times 10^4$, indicates that the ligand can be utilized in the separation of the two metals under the specified conditions (Uzoukwu and Gloe, 1998) [19].

Potential for quantitative separation can be determined by calculating n the number of batches needed to achieve 99.9% extraction of the metals with the higher distribution ratios X by applying reagents that completely masks the metal (Y) with the lower distribution ratios. The quantitative equation is $C = C_{aq} \left[\frac{V_{aq}}{DV_{or} + V_{aq}} \right]^n$; where C is concentration of metal in aqueous phase after extraction and C_{aq} is initial concentration of metal in aqueous phase before extraction. Since equal volume of aqueous phase and organic phase are usually employed during these extractions' studies, the equation reduces to $C = C_{aq} \left[\frac{1}{D+1} \right]^n$. (Uzoukwu, 2009) [20].

Studies have shown that the Schiff base, 4, 4'-(1*E*, 1*E'*)-1, 1'-(ethane-1, 2-diylbis (azan-1-yl-1ylidene)) bis (5-methyl-2-phenyl-2, 3-dihydro-1*H*-pyrazol-3-ol) (H₂BuEtP), is excellent extractant for Lead (Godwin and Uzoukwu, 2012a) [5], Uranium (Godwin and Uzoukwu, 2012b) [6], Nickel (Godwin *et al.*, 2012) [9], Iron (Godwin *et al.*, 2013; Godwin *et al.*, 2014) [8, 2] and Cadmium (Godwin and Tella, 2017; Godwin

and Tella, 2019) [4, 3] from buffered aqueous media; the synthetic route is shown in Figure 1 (Uzoukwu *et al.*, 1998) [19]. The various pH ranges at which optimal extraction of the metals were achieved and the effect of some acids, anions and auxiliary complexing agents in these extractions were also investigated and reported. Apart from H₂SO₄, all other acids used for the studies at concentrations >0.1M and EDTA at all concentrations masked Lead extractions (Godwin and Uzoukwu, 2012a) [5], all acids masked extraction of Nickel (Godwin *et al.*, 2012) [9] whilst masking of Iron occur mostly at high concentrations of acids (Godwin *et al.*, 2014) [2]. Masking in Cadmium extraction was only significant with CH₃COO⁻ at 1.0M (Godwin and Tella, 2019) [3]. Since most of the other results gave >90% extraction of these metals being studied with the ligand when masking occurs with a particular metal, there is a potential for separating these metals at these masked conditions as theoretically calculated; Separation Factors β_{XY} gave $\geq 1 \times 10^4$ under some conditions. The aim of the study was to determine the effect of a second metal on the theoretically calculated Separation Factors for the two metals under same condition and to ascertain the possibility of practically separating Lead, Nickel, Iron and Cadmium from a buffered aqueous solution containing any two of these metals using the Schiff base 4, 4'-(1*E*, 1*E'*)-1, 1'-(ethane-1, 2-diylbis(azan-1-yl-1ylidene)) bis (5-methyl-2-phenyl-2, 3-dihydro-1*H*-pyrazol-3-ol) (H₂BuEtP) alone and in the presence of (3-hydroxy-5-methyl-2-phenyl-2, 3-dihydro-1*H*-pyrazol-4-yl) butan-1-one (HBuP),

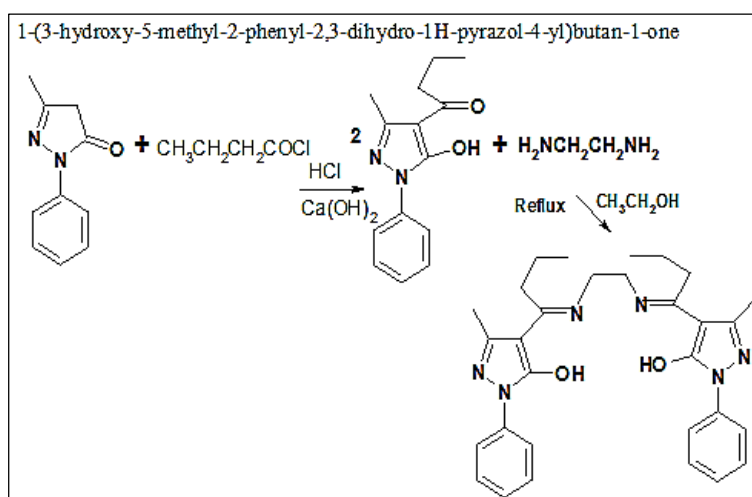


Fig 1: Reactions for the Synthesis of 1-(3-hydroxy-5-methyl-2-phenyl-2,3-dihydro-1*H*-pyrazol-4-yl)butan-1-one (HBuP) and 4,4'-(1*E*, 1*E'*)-1, 1'-(ethane-1,2-diylbis(azan-1-yl-1ylidene))bis(5-methyl-2-phenyl-2,3-dihydro-1*H*-pyrazol-3-ol) (H₂BuEtP)

Experimental

Twelve well labelled sets of 10mL volumetric bottles containing 50mgL⁻¹ each of a pair of the metals (Cadmium and Iron, Lead and Nickel) in 2mL aqueous media buffered to either pH 4.75 or 7.5 and containing varying concentrations of acids or anion at different concentrations were prepared. 2 mL chloroform solutions of 0.05M concentration of H₂BuEtP or 0.05M HBuP (9:1 ratio by volume) was pipetted into the aqueous phases. The immiscible phases were shaken mechanically for sixty minutes at a room temperature of 30 °C. A shaking time of sixty (60) minutes was found suitable enough for equilibration. The two phases were allowed to settle, separated and all organic phases removed carefully using separating funnel. Fresh organic phases added to the aqueous raffinate and process repeated based on calculated "n" number of batches needed to achieve 99.9%

extraction for the metal in the pair that has the higher Distribution Ratio D_X in the metal alone extraction with same organic phase. After the last batches of extractions, 0.2 mL of aqueous raffinate were then taken and analysed for each metal by difference between the concentration of metal ions in aqueous phases before and after the extractions using Atomic Absorption Spectrophotometer (AAS) for Cadmium (II), Nickel (II) and Lead (II), while concentration of Fe (II) in the aqueous raffinate were determined colorimetrically with a UV spectrophotometer at wavelength of 520nm. The colour development for Iron (II) determination was by addition of 0.1 mL of hydroxylamine hydrochloric acid, 0.1mL of 1, 10-phenanthroline and 0.1mL of sodium acetate. Distribution ratios D were calculated as the ratio of metal ion concentration in the organic phase (C_o) to that in the aqueous phase (C). Thus $D = C_o/C$. Separation Factors for each pair of

metal ion was calculated by using equation 1 and n number of batches needed to achieve 99.9% extraction of a particular metal ion was calculated by using equation 2.

$$\beta_{XY} = D_X/D_Y \quad \text{Equation 1}$$

$$C = C_{aq} \left[\frac{1}{D+1} \right]^n \quad \text{Equation 2}$$

The extraction parameters, Distribution Ratios, Percent Extractions and *n* batches of extractions needed to achieve 99.9% extraction of the metals in metal alone extractions and theoretically calculated Separation Factors β_{XY} are compared with experimental values from the bimetal extractions with the same aqueous conditions.

The data were statistically analysed using the R software package [R Development Core Team 2008] to test for significant difference between Distribution Ratios of the metal in extractions alone (D_X) and in the presence of a

second metal ($D_{X/Y}$) in the same organic phase. The *t* test statistics [Sprinthal, 2011]^[17] was used to test the hypothesis, if the two Distribution Ratios D_X and $D_{X/Y}$ were significantly different in these extractions.

The null hypothesis (H_0), which states that the two Distribution Ratios D_X and $D_{X/Y}$ of interest are not significantly different is rejected if the value of the test statistics is greater than the critical value and the alternative hypothesis (H_a), which states that the two groups of interest are significantly different is accepted. The *p* value was also used. If the *p* value is greater than the significant level $\alpha = 0.05$, the null hypothesis is accepted and we conclude that there is no significant difference between the groups of interest.

Results and Discussion

Table 1a: Extraction Parameters for Cadmium and Nickel in H_3PO_4 and H_2SO_4 at pH 4.75 in Ligand H_2BuEtP alone

Acid (M)														
H_3PO_4	% E_{Cd}	% E_{Ni}	D_{Cd}	D_{Ni}	% $E_{ECd/Ni}$	% $E_{ENi/Cd}$	$D_{ECd/Ni}$	$D_{ENi/Cd}$	$\beta_{TCd/Ni} \times 10^4$	$\beta_{ECd/Ni}$	<i>n</i> _{TCd}	<i>n</i> _{TNi}	<i>n</i> _{ECd}	<i>n</i> _{ENi}
0.001	97.4	0.1	37.2	0.001	95.5	82.2	21.2	4.63	3.72	4.58	2	5990	2	5
0.005	96.7	0.1	29.0	0.001	41.0	90.45	7.29	10.12	2.90	1.39	2	5990	9	4
H_2SO_4														
0.001	94.2	0.1	16.5	0.001	95.0	80.0	19.36	4.17	1.65	4.64	2	5990	2	5

Table 1b: Extraction Parameters for Cadmium and Nickel in H_3PO_4 and H_2SO_4 at pH 7.5 in Ligand H_2BuEtP alone

Acid (M)														
H_3PO_4	% E_{Cd}	% E_{Ni}	D_{Cd}	D_{Ni}	% $E_{ECd/Ni}$	% $E_{ENi/Cd}$	$D_{ECd/Ni}$	$D_{ENi/Cd}$	$\beta_{TCd/Ni} \times 10^4$	$\beta_{ECd/Ni}$	<i>n</i> _{TCd}	<i>n</i> _{TNi}	<i>n</i> _{ECd}	<i>n</i> _{ENi}
0.001	97.4	0.1	37.2	0.001	97.1	92.2	21.11	15.11	3.72	1.40	2	5990	2	3
0.005	96.7	0.1	29.0	0.001	96.0	86.25	25.0	6.27	2.90	3.99	2	5990	2	4
H_2SO_4														
0.001	94.2	0.1	16.5	0.001	95.7	93.2	22.38	13.69	1.65	1.64	2	5990	2	3

The Distribution ratios D_{Ni} in Table 1a and b for the extraction of Nickel alone in the presence of 0.001 M and 0.005 M of H_3PO_4 and 0.001M H_2SO_4 in aqueous phases buffered at pH 4.75 and 7.5 in chloroform solution of ligand H_2BuEtP alone all showed distribution ratio of 0.001, indicating that masking of Nickel occurred in these media for Nickel alone extractions for both pH 4.75 and 7.5 in these concentrations of H_3PO_4 and H_2SO_4 (Godwin *et al.*, 2012)^[9]. However, Nickel distribution ratios $D_{Ni/Cd}$ in the bimetal extractions in the presence of Cadmium for same concentrations of H_3PO_4 and H_2SO_4 using the same organic phase of chloroform solutions of 0.05 M H_2BuEtP alone had distribution ratios ranging from 4.17-15.11 (Godwin and Tella, 2019)^[3]. The distribution ratios $D_{ENi/Cd}$ were all statically significantly different from those for D_{Ni} and represented percentage extractions (%E) >80% as against <1% for all D_{Ni} in Nickel alone studies. This indicated that in the bimetal extractions, H_3PO_4 and H_2SO_4 had a releasing/salting out effect on both Cadmium and Nickel as against the masking effect they had in the Nickel alone extraction in both pH 4.75 and pH 7.5. The distribution ratios for Cadmium alone D_{Cd} under the same concentrations of H_3PO_4 and H_2SO_4 with the ligand H_2BuEtP were all in the range 16.5-37.2. Comparing distribution ratios for Cadmium alone D_{Cd} in same aqueous phases containing H_3PO_4 and H_2SO_4 with those in the bimetal extraction with Nickel $D_{Cd/Ni}$ using the same ligand H_2BuEtP alone organic phase as the extractant, showed that the values apart from that for H_3PO_4 at 0.005M were not significantly different. The values were all >19 except that at 0.005 M H_3PO_4 for pH 4.75 that was

7.29. All results indicated that both H_3PO_4 and H_2SO_4 had a salting out/releasing effect on Cadmium alone and in the presence of Nickel. Since Nickel that was masked in Nickel alone studies was remarkable extracted in the presence of a second metal leading to huge drop in experimental Separation Factors $\beta_{ECd/Ni}$ ranging from 1.19-4.64 compared to theoretical calculated Separation Factors $\beta_{TCd/Ni}$ that ranged from 1.65×10^4 - 2.90×10^4 and theoretically had potentials as separation methods for the separation of cadmium from nickel in a bimetal media containing H_3PO_4 and H_2SO_4 . The experimental results are indicating that in the bimetal extraction of Cadmium and Nickel in the presence of H_3PO_4 and H_2SO_4 at both pH 4.75 and 7.5, the presence of the second metal is enhancing the extraction of the metal that was originally masked. This could be by reducing the water molecules aquated around the metal ions and thereby increasing the dielectric constant of the aqueous media by the presence of a second metal ion (Uzoukwu, 2009)^[20]. The calculated number of batches of extractions *n* needed to extract 99.9% of each metal in Table 1 indicated that, the conditions are more suited for the bimetal extraction of Cadmium and Nickel with Cadmium extracted quantitatively (99.9%) after 2 extra batches in all concentrations of the two acids except for 0.005M H_3PO_4 at pH 4.75 that required 9 extra batches to extract 99.9% of Cadmium. Nickel required between 3-5 extra batches of extractions to achieve 99.9% extractions in all cases. pH 7.5 results were better than pH 4.75 as a bimetal extraction buffer medium for the two metals using the two acids with the ligand H_2BuEtP .

Table 2a: Extraction Parameters for Cadmium and lead in H₃PO₄ at pH 4.75 in Ligand H₂BuEtP alone

Acid (M)														
H ₃ PO ₄	% E _{Cd}	% E _{Ni}	D _{Cd}	D _{Ni}	% E _{ECd/Ni}	% E _{ENi/Cd}	D _{ECd/Ni}	D _{ENi/Cd}	β _{TCd/Ni} × 10 ⁴	β _{ECd/Ni}	n _{TCd}	n _{TNi}	n _{ECd}	n _{ENi}
0.001	96.7	0.1	29.0	0.001	95.4	88.2	20.1	8.13	2.90	2.41	2	5990	2	5
0.005	96.7	0.1	29.0	0.001	96.0	89.3	23.91	8.34	2.90	2.87	2	5990	2	4

Table 2b: Extraction Parameters for Cadmium and lead in H₃PO₄ at pH 7.5 in Ligand H₂BuEtP alone

Acid (M)														
H ₃ PO ₄	% E _{Cd}	% E _{Ni}	D _{Cd}	D _{Ni}	% E _{ECd/Ni}	% E _{ENi/Cd}	D _{ECd/Ni}	D _{ENi/Cd}	β _{TCd/Ni} × 10 ⁴	β _{ECd/Ni}	n _{TCd}	n _{TNi}	n _{ECd}	n _{ENi}
0.001	96.7	0.1	29.0	0.001	95.70	95.30	23.33	22.85	2.90	1.02	2	5990	2	2
0.005	96.7	0.1	29.0	0.001	95.01	90.69	19.41	10.12	2.90	1.39	2	5990	2	3

Data in Table 2a and b for the extraction of Cadmium and Lead in single metal extractions from aqueous media buffered at pH 4.75 and 7.5, containing 0.001M and 0.005M H₃PO₄ using chloroform solutions of 0.05M H₂BuEtP organic phases all showed that Lead was masked by the acid in Lead alone extractions with all distribution ratios D_{Pb} = 0.001 and percentage extraction % E = 0.1% (Godwin and Uzoukwu, 2012a) [5], while Cadmium was released/salted out by H₃PO₄ with all distribution ratios D_{Cd} = 29.0 and percentage extraction % E = 96.7% (Godwin and Tella, 2019) [3]. The calculated theoretical Separation Factor β_{TCd/Pb} of 2.90 × 10⁴ from the metal alone extractions indicated that 0.001 M and 0.005M H₃PO₄ in aqueous media buffered to pH 4.75 and 7.5 can be utilized in separating Cadmium from Lead using the ligand H₂BuEtP. However, data for the bimetal extraction of Cadmium and Lead showed remarkably increased extraction of Lead with distribution ratios D_{EPb/Cd} in the range 8.13-22.85 that were all statistically different from those for Lead alone D_{Pb}. The distribution ratios for Cadmium alone D_{Cd} 29 were

all statistically not different from those for Cadmium in the presence of Lead D_{ECd/Pb} that were in the range 19.41-23.91. This also indicated that in the bimetal media, the water molecule aquated to the metals is greatly reduced and the dielectric constant of the aqueous media remarkably increased with the resultant salting out/releasing of both metals (Uzoukwu, 2009) [20]. Thus, the experimental Separation Factors β_{ECd/Pb} ranging from 1.36-2.87 were far < 2.90 × 10⁴ the value for all theoretical Separation Factor β_{TCd/Pb}. The experimental Separation Factors β_{ECd/Pb} showed that ligand H₂BuEtP can be used for the bimetal extraction of Cadmium and lead from aqueous media containing 0.001 M and 0.005 M H₃PO₄ buffered to pH 4.75 and 7.5. The number of batches needed to obtain 99.9% extractions *n* for both metals (Table 2) showed that pH 7.5 was slightly better even though 2 extra batches of extractions are needed for Cadmium for both pHs, 4 extra batches are needed for Lead at pH 4.75 as against extra 2 at 0.001 M and 3 at 0.005 M for pH 7.5.

Table 3a: Extraction Parameters for Cadmium and Iron in Oxalate at pH 4.75 in Mixed Ligand H₂BuEtP/H BuP

ION(m)														
Oxalate	% E _{Cd}	% E _{Fe}	D _{Cd}	D _{Fe}	% E _{ECd/Fe}	% E _{FEi/Cd}	D _{ECd/Fe}	D _{FEFe/Cd}	β _{TCdFe} × 10 ³	β _{ECd/Fe}	n _{TCd}	n _{TFe}	n _{ECd}	n _{EFe}
0.1	90.3	0.1	9.27	0.001	93.3	85.8	14.55	6.04	9.27	2.41	2	5990	2	4

Table 3b: Extraction Parameters for Cadmium and Lead in Oxalate at pH 7.5 in Mixed Ligands H₂BuEtP/HBuP

ION(m)														
Oxalate	% E _{Cd}	% E _{Fe}	D _{Cd}	D _{Fe}	% E _{ECd/Fe}	% E _{FEi/Cd}	D _{ECd/Fe}	D _{FEFe/Cd}	β _{TCdFe} × 10 ³	β _{ECd/Fe}	n _{TCd}	n _{TFe}	n _{ECd}	n _{EFe}
0.1	90.3	0.1	9.27	0.001	95.44	91.19	20.93	10.35	9.27	2.02	2	5990	2	3

Extraction data in Table 3 for the extraction of Iron and Cadmium alone and in bimetal extractions containing the two metals from aqueous media containing 0.1M Oxalate ions buffered to pH 4.75 and 7.5 into chloroform solutions of 0.05 M H₂BuEtP and 0.05 M HBuP in a 9:1 volume ratio. The metals alone had Distribution ratios D_{Cd} and percentage extraction % E for Cadmium 9.27 and 90.3% respectively. This showed that at both pH 4.75 and 7.5, 0.1 M Oxalate ions salted out/released cadmium into the mixed ligands H₂BuEtP/HBuP organic phase. The distribution ratios D_{Fe} and percentage extraction % E_{Fe} for Iron alone extraction were 0.001 and 0.1% showing that Oxalate ions masked Iron extractions into the mixed ligands H₂BuEtP/HBuP organic phase (Godwin, *et al.*, 2014). Thus, calculated theoretical Separation Factors β_{TCd/Fe} using their distribution ratios D_{Cd} and D_{Fe} was 9.27 × 10³ and theoretically can be used for separating Iron from Cadmium. The distribution ratios and percentage extraction for the bimetal extractions for the two metals in aqueous media containing 0.1 M Oxalate ions at pH 4.75 and 7.5 into the mixed ligands H₂BuEtP/HBuP organic phases were D_{ECd/Fe} 14.55 and 93.3% for Cadmium and D_{FEFe/Cd} 6.04 and 85.8% for Iron at pH 4.75 and D_{ECd/Fe} 20.93

and 95.44% for cadmium and D_{FEFe/Cd} 10.35 and 91.19% for Iron at pH 7.5. The distribution ratios for bimetal extractions of Iron in the presence Cadmium D_{FEFe/Cd} for both pHs were both statistically significantly different from both for Iron alone D_{Fe}. The experimental Separation Factors β_{ECd/Fe} were both < 2.5 and thus suited only for bimetal extractions of Cadmium and Iron. pH 7.5 was also better than pH 4.75 as even though extra 2 extra batches of extractions *n* with fresh mixed ligands H₂BuEtP/HBuP organic phases are needed to obtain 99.99% extraction of Cadmium in both pHs, 4 and 3 extra batches of extraction *n* are needed for pH 4.75 and 7.5 to completely extract Iron.

Conclusions

1. Theoretically calculated separation factors based on distribution ratios for any pair of metals in single metal solvent extraction are greatly reduced in bimetal extraction for the two metals using same organic phase.
2. Bimetal media improved the salting out/releasing strength of acids and anions and favours simultaneous extraction of a pair of metals than separation of the two metals using the ligand H₂BuEtP.

- Reduction in water molecules aquated around the metals due to the presence of the second metal leading to increased dielectric constants of the aqueous media may be responsible for the improved extraction of a masked metal in metal alone extraction with the ligand H₂BuEtP.
- pH 7.5 was better than 4.75 as a buffer medium for the bimetal extraction of the pair of metals with H₂SO₄, H₃PO₄ and Oxalate ion using the ligand H₂BuEtP alone or in the presence of HBuP.

Recommendations

Since there was improved extraction of the masked metal in the metal alone extractions in the presence of a second metal leading to >90% extraction of both metals, we recommend that multi-metal extractions studies be undertaken with the ligand H₂BuEtP alone and in the presence of the synergist HBuP.

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