

Studies on the separation of some transition metals using trialkylimidazole as selective extractant

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Abstract. The possibility of Cu(II) ion separation from an equimolar Cu-Cd-Zn tertiary mixture by solvent extraction was investigated. The process was based on the use of 1,2,4-trimethylimidazole (TMI) and 1-decyl-2,4-dimethylimidazole (DDMI) as extractant. Dichloromethane was used as the solvent in the extraction process. The dissociation constant of extractants were found potentiometrically. Extraction data were used for finding stability constants and partition constants for the complexes being formed in the aqueous solution. For TMI, separation coefficients for Cu(II) with regard to zinc and cadmium are 2.9 and 1.3, respectively. For DDMI separation coefficients for Cu(II) with regard to zinc and cadmium are 1.4 and 1.6, respectively. Sparingly soluble in water 1-decyl-2,4-dimethylimidazole is a more useful extractant.

1 Introduction

Industrial wastewater containing heavy metals (eq. zinc, copper, cadmium) belongs to hazardous wastes. Wastewater discharge is a primary source of this metal release into the environment [1]. On the other hand, these metals are very important due to wide application in the manufacturing of alloys, batteries, pigments and metal plating [2]. Therefore, the recovery and recycling of heavy metals from various waste materials is necessary and very important to gain the metal values and protect the environment [2,3]. Moreover, the increasingly stringent regulations regarding the discharge of metals into the environment, and the increasing stress upon the recycling of effluents have focused the great interest on the development of new approaches for the recovery of metals from industrial wastes [4]. Cote scored copper, zinc and cadmium for strategic metals [5].

A survey of literature indicates that separation or removal of heavy metal ions from different aqueous solutions can be achieved by solvent extraction [6-9]. Several extractants have been proposed for separation of Cu(II), Zn(II), and Cd(II) from aqueous solutions, e.g., carboxylic acids [10], derivatives of phosphinic acid [11], derivatives of acid amides, trioctylamine [11], alkylphenylamines [12], complex oximes [13], crown ethers [14-16] derivatives of ketones [17-20], derivatives of pyridine [21, 22], as well as alkyl derivatives of imidazole [6, 23-25] and quaternary ammonium salt [26].

The possibility of using alkyimidazoles for solvent extraction of metals has been noted for a long time [27-29]. They are now a fairly well-known group of basic ligands (extractants) whose properties can be easily modified. The pK_a value of the imidazole molecule is 6.98 at 20°C [30]. The stability of its complexes with the

cations of the 1st row of transition metals is higher than ammonia due to the binding of $\pi_{M \rightarrow L}$ [31].

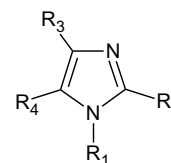


Fig. 1. General formula of alkyimidazole.

The alkyl group at position 1 (R₁ Fig. 1) slightly increases the basicity of the donor nitrogen atom as well as the stability of the complexes. Even small alkyl group at position 2 (R₂ Fig. 1) or 4 (R₃ Fig. 1) constitute a steric hindrance making it difficult to form of complexes with a rigid, hardly deformable coordination sphere with octahedral or flat symmetry. Although 2 or 4 substituted alkyimidazoles are stronger bases than 1-alkylimidazole their complexes have lower values of stability constants. The introduction of alkyl groups into the imidazole ring, irrespective of their location, reduces the water solubility in all homologous series of alkyimidazoles. Compounds containing a total of more than 5 carbon atoms in substituents are practically insoluble in water. They can be used for metal separation as extractants in hydrometallurgy or as a carrier in membrane techniques. So far, the extraction properties of 1-alkylimidazoles [6, 32, 33], 1-alkyl-2-methylimidazoles [7, 34, 35], 1-alkyl-2-ethylimidazoles [36], 1,2-dialkyimidazoles [23, 25] and 1-alkyl-4-methylimidazoles [25, 37] have been studied. On the other hand, our knowledge of donor and extraction properties of tri-substituted alkyimidazoles is very small.

The purpose of the present study was to determine the basicity, complexity and extraction properties of

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1,2,4-trimethylimidazole (TMI) and 1-decyl-2,3-dimethylimidazole (DDMI) and to compare their effectiveness in copper(II) ion separation from an equimolar Cu-Cd-Zn mixture. TMI and DDMI molecules differ in their hydrophobicity. Methylene chloride was used as the diluent in the solvent extraction process.

2 Experimental

2.1 Reagents

Inorganic chemicals: copper(II), zinc(II), cadmium(II), and potassium nitrates were of analytical grade and were supplied by POCh (Gliwice, Poland). Nitric acid was of analytical grade and were supplied by MERCK. Aqueous solutions were prepared with double distilled water (conductivity of 5 μ S/m).

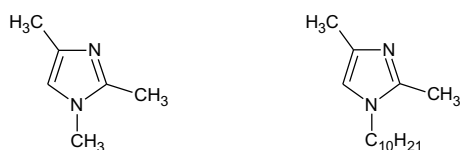


Fig. 2. Structure of the extrahents: 1,2,4-trimethylimidazole (TMI) and 1-decyl-2,4-dimethylimidazole (DDMI).

1,2,4-Trimethylimidazole (TMI) and 1-decyl-2,4-dimethylimidazole (DDMI) (Fig. 2) were synthesized at Medical University of Lodz, Poland.

2.2 Determination of dissociation constants (pK_a)

The dissociation constants of TMI was determined by potentiometric titration. Measurements of pH were made using PHM 250 pH-meter (Radiometer) equipped with a glass-calomel combination electrode C 2401-8 (Radiometr). Measuring cell was thermostated at $25^\circ\text{C} \pm 0.5^\circ\text{C}$. Two solution were prepared. One contained nitric acid (0.01 mol/dm^3), ligand (TMI or DDMI), and a proper quantity of potassium nitrate to ensure a constant ionic strength (0.5 mol/dm^3). Another solution was prepared in a similar manner but without ligand. Measurements were taken for two ligand (TMI or DDMI) concentrations (0.06 mol/dm^3 and 0.05 mol/dm^3) in the first solution. A 20 cm^3 volume of first solution was placed in the measuring cell, and the solution was titrated with the second. Three series of measurements were run.

2.3 Solvent extraction

Equal volumes of organic and aqueous phases (phase volume ratio $O/A = 1$) were mechanically shaken for 20 min at $25 \pm 2^\circ\text{C}$. $0.05\text{-}0.25 \text{ mol/dm}^3$ ligand (TMI or DDMI) in dichloromethane was used as the organic phase. After establishing equilibrium, the aqueous phase was separated from the organic phase. Equilibrium pH of aqueous phases was measured with a Radiometr PHM 250 pH-meter equipped with a glass-calomel combination

electrode C 2401-8 (Radiometr). Metal ions concentrations in aqueous phases were analyzed with BUCK Scientific 210 VGP spectrophotometer (AAS — Atomic Absorption Spectrometry). The concentrations of metal ions in aqueous phases were determined before and after solvent extraction for calculating extraction efficiency. The concentrations of metals in organic phases were deduced by mass balance. Each experiment was carried out three times and the error did not exceed 3%.

3 Results and discussion

3.1 Dissociation constant of ligands (TMI and DDMI)

The acid dissociation constants of the protonated TMI and DDMI molecules were calculated from the equation:

$$K_a = \frac{[H_3O^+][L]}{[HL^+]} = \frac{[H_3O^+](C_L - C_{HNO_3} + [H_3O^+])}{C_{HNO_3} - [H_3O^+]} \quad (1)$$

where $[H_3O^+]$, $[L]$, and $[HL^+]$ are equilibrium concentrations of the hydronium ion, the free ligand (TMI or DDMI) and its protonated form, respectively, where C_L and C_{HNO_3} are analytical concentrations of the ligand and HNO_3 in aqueous solution, respectively.

Table 1 presented the dissociation constants of the protonated TMI and DDMI together with the previously determined the dissociation constants of imidazole derivatives.

Table 1. Comparison of the dissociation constants pK_a of chosen alkylimidazole derivatives in aqueous solution ($I=0.5 \text{ mol/dm}^3 \text{ K(H)NO}_3$) at 25°C .

	pK_a	ref.
1,2,4-trimethylimidazole (TMI)	8.49	[this work]
1-decyl-2,4-dimethylimidazole (DDMI)	8.62	[this work]
imidazole	6.99	[30]
1-methylimidazole	7.06	[31]
2-methylimidazole	8.10	[38]
4-methylimidazole	7.80	[39]
1,2-dimethylimidazole (1,2-DMI)	8.21	[31]
1,4-dimethylimidazole (1,4-DMI)	7.72	[40]
2,4-dimethylimidazole (2,4-DMI)	8.57	[38]
4,5-dimethylimidazole (4,5-DMI)	8.19	[41]

The introduction of even a small alkyl group in positions 2 or 4 of the imidazole ring results in the molecule having a much higher basicity (Table 1). 2-Alkylimidazole and 4-alkylimidazole are harder bases in comparison with imidazole and 1-alkylimidazoles. 1,2,4-Trimethylimidazole (TMI) is a stronger base than the 1,2-dimethylimidazole (1,2-DMI), but 1-decyl-2,4-dimethylimidazole (DDMI) is the strongest base.

3.2 Solvent extraction

In the case where there is no phase change in volume, distribution ratio (D_M) was defined as follows:

$$D_M = \frac{C_{M(org)}}{C_{M(aq)}} = \frac{C_M^0 - C_M}{C_M} \quad (2)$$

where: C_M^0 and C_M denote analytical metal concentrations in the aqueous phase before and after attaining a partition equilibrium, respectively.

Based on analytical calculations for every equilibrium pH value for the aqueous phase, the distribution ratios (D_M) were calculated from formula (2) for each tested metal and then extraction curves were plotted ($\log D_M = f(\text{pH})$) (Figs. 3 and 4).

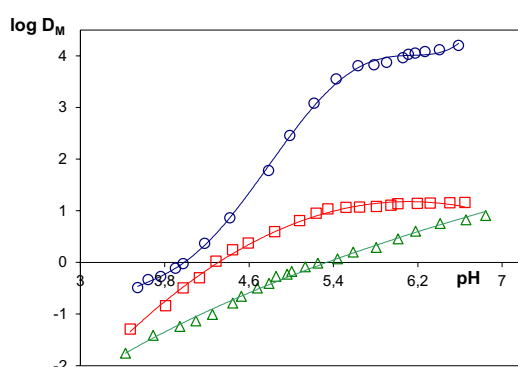


Fig. 3. Extraction curves of Cu(II) (○), Cd(II) (□), and Zn(II) (△) complexes with 1,2,4-trimethylimidazole (TMI) into dichloromethane.

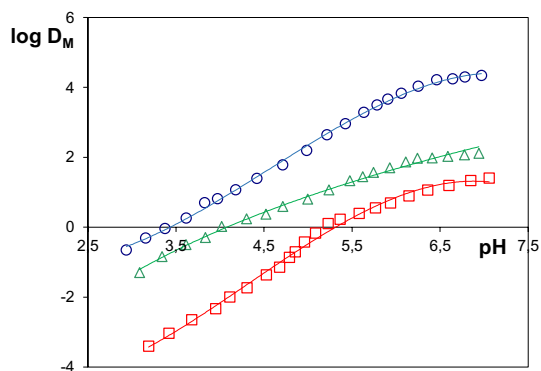


Fig. 4. Extraction curves of Cu(II) (○), Cd(II) (□), and Zn(II) (△) complexes with 1-decyl-2,4-dimethylimidazole (DDMI) into dichloromethane.

Fig. 3 and Fig 4 indicate that partition ratios for the test metal complexes with TMI and DDMI tend to increase with an increase in the pH values of the aqueous phase. For TMI (Fig.3), the order of extracted ions is Cu(II) > Cd(II) > Zn(II), but for DDMI (Fig.4) Cu(II) > Zn(II) > Cd(II).

In the case of Cu(II) and Cd(II) complexes with TMI (Fig.3) for pH > 5.8 (for Cu(II)) and 5.4 (for Cd(II)) the

values of D_M are constant and they do not depend on the increase in the extractant's concentration in the aqueous and organic phases. Best extracted were Cu(II) complexes with TMI, worst extracted were Zn(II).

In the case of DDMI, the extraction of Zn(II) and Cu(II) complexes is almost the same at lower pH values (to about pH=5), while for Cd(II) is much worse. Above pH=5.5, the values of D_M increase rapidly for Cu (II), but in case of Cd(II) and Zn(II) these values are approaching each other.

For TMI the values of $\text{pH}_{1/2}$, corresponding to 50% of the extraction of copper, cadmium and zinc, are equal to 4.0, 4.3 and 5.3, respectively. This means, they can be separated by partition in the liquid-liquid system, conducting a multistep extraction at a pH value which is optimum for a given metal (Fig. 3).

For DDMI (Fig. 4), 50% of the extraction of copper, cadmium and zinc, occurs at pH equal to 3.45, 5.4 and 4.05, respectively.

Distribution ratio (D_M) is related to stability constants of the complexes and their partition ratios by the following equation:

$$D_M = \frac{P_n \beta_n [L]^n + P_{n+1} \beta_{n+1} [L]^{n+1} + \dots + P_N \beta_N [L]^N}{\sum_{n=0}^{n=N} \beta_n [L]^n} \quad (3)$$

where: β_n are cumulative stability constants of the complexes in the aqueous phase, P_n are organic solvent/water partition ratios of the complexes, ($P_n = \frac{[ML_n]_{(org)}}{[ML_n]_{(aq)}}$), $[L]$ is the free ligand (TMI or DDMI) concentration (mol/dm^3) in the aqueous phase and n is the number of ligands molecules in the first metal(II) complex that is so hydrophobic that it freely passes into the organic phase.

Cumulative stability constants are described by the equations:

$$\beta_1 = \frac{[ML]}{[M][L]} \quad \beta_2 = \frac{[ML_2]}{[M][L]^2} \quad \dots \quad \beta_n = \frac{[ML_n]}{[M][L]^n} \quad (4)$$

The pooled D_M values and the corresponding sets of the independent variable $[L]$ were used for determination of the stability constants β_n and partition ratios P_n of the metal(II) complexes by using the equation proposed by Rydberg [42]:

$$\frac{[L]^n}{D_M} = \frac{\sum_{n=0}^{n=N} \beta_n [L]^n}{P_n \beta_n} \quad (5)$$

Equation (5) can be used for determination of the composition of the first complex being extracted from the aqueous phase and the stability constant, β_1 , of the first complex formed in the aqueous phase. To do this, the shape of the function $[L]^n/D_M = f[L]$ is monitored when varying the exponent n between 1 and 6. In this way, a bundle of curves were obtained of which only one was a straight line with a positive slope and b values. It is assumed that the n value is equal to the number of ligand molecules attached to the central ion in the first complex

being extracted. The method was used previously [6, 7, 23-25, 32-37]. The stability constants, β_n , obtained in this way are collected in Table 2 together with stability constants for 1,2-dimethylimidazole [31], and 1,4-dimethylimidazole [40].

Table 2. Comparison of the stability constants β_n of Cu(II), Zn(II), and Cd(II) complexes with 1,2,4-trimethylimidazole (TMI), 1-decyl-2,4-dimethylimidazole (DDMI), 1,2-dimethylimidazole (1,2-DMI), and 1,4-dimethylimidazole (1,4-DMI) at 25°C, ionic strength 0.5 mol/dm³ (KNO₃).

ligand/pK _a	metal ion	log β_1	log β_2	log β_3	log β_4	ref.
TMI 8.49	Zn(II)	2.03	4.17	6.27	9.51	[this work]
	Cu(II)	3.75	6.83	9.26	11.38	
	Cd(II)	2.34	3.79	5.32	6.95	
1,2-DMI 8.21	Zn(II)	1.92	4.32	7.11	9.00	[31]
	Cu(II)	3.70	6.80	9.18	10.80	
	Cd(II)	2.20	3.45	5.05	6.34	
1,4-DMI 7.72	Zn(II)	1.70	4.48	5.40	8.90	[40]
	Cu(II)	3.54	6.53	9.17	10.47	
	Cd(II)	2.05	3.93	5.11	5.81	
DDMI 8.62	Zn(II)	2.17	4.48	6.39	9.87	[this work]
	Cu(II)	3.95	7.18	9.42	11.69	
	Cd(II)	2.53	4.21	5.68	7.56	

A comparison of the values of stability constants in Table 2 indicates that stability for all complexes tends to increase with ligand's alkalinity and is the highest for Cu(II) complexes. In the case of TMI, stability constants for Zn(II) complexes are higher than those for Cd(II) complexes.

In aqueous solutions, cations of Zn(II), Cd(II) and Cu(II) exist in the form of octahedral aqua complexes $[M(H_2O)_6]^{2+}$. Owing to the effect of ligands (extractants), octahedral aqua complexes of certain cations tend to change their coordination number (c.n.) from 6 to 4 and they change their coordination sphere into flat or tetrahedral, depending on the structure of their d-electron layer. The process is illustrated by the equation.



Therefore, tetrahedral complex (c.n.=4) are formed in the solution in addition to octahedral ones (c.n.=6). Tetrahedral complexes are specific to Zn(II), and Cd(II). Also Cu(II) ions tend to change the symmetry of their coordination sphere, forming distorted tetrahedral or flat structures [43].

Changes in the coordination sphere lead to higher sums of complex stability constants (stability constants are the sum of the stability constants for the complexes with c.n.=4 and with c.n.=6) and higher susceptibility to extraction in the liquid-liquid system, because complexes with the c.n.=4, being less hydrated, pass more readily into the organic phase after at least part of the molecules of water, which is bound in the metal coordination sphere, is replaced by the molecules of the more hydrophobic ligand. For the fourth stage of complexation (n=4) this can be illustrated by the following equations:



wherein L is the extractant (TMI or DDMI) molecules.

Table 3. Comparison of the partition ratios P_n of Cu(II), Zn(II), and Cd(II) complexes with 1,2,4-trimethylimidazole (TMI), 1-decyl-2,4-dimethylimidazole (DDMI) and 1,4-dimethylimidazole (1,4-DMI) between the aqueous and the organic phases, at 25°C, ionic strength 0.5 mol/dm³ (KNO₃).

ligand/solvent	metal ion	P ₁	P ₂	P ₃	P ₄	ref.
TMI methylene chloride	Zn(II)		0.84	1.23	29.21	[this work]
	Cu(II)		1.26	1.87	84.72	
	Cd(II)		0.3	1.56	65.46	
1,4-DMI benzyl alcohol	Zn(II)				8.72	[40]
	Cu(II)		0.1	1.65	20.75	
	Cd(II)			1.54	110.7	
DDMI methylene chloride	Zn(II)		1.12	2.46	76.43	[this work]
	Cu(II)		0.98	2.15	106.8	
	Cd(II)		0.15	1.71	67.39	

The partition ratios between the aqueous and organic phases for the investigated metal complexes with TMI and DDMI, were found from Equation (5), and they are collected in the Table 3 together with partition ratios for the Cu(II), Zn(II) and Cd(II) complexes with 1,4-dimethylimidazole (1,4-DMI) [40].

The data in Table 3 indicate that in the process of extraction of the tested metal complexes with TMI and DDMI as well as 1,4-DMI [40], mainly the fourth complexes tend to pass into the organic phase. The partition constants P₄ (Table 3) well reflect susceptibility to extraction of the tested metal complexes with TMI and DDMI. Their high values are proof that, most probably, they are 4-coordination complexes of the formula $[ML_4](NO_3)_2$. Extraction order for depends on extractant, for TMI extraction order was Cu(II) > Cd(II) > Zn(II), but for DDMI – Cu(II) > Zn(II) > Cd(II).

For TMI separation coefficients Cu(II) with regard to Zn(II) and Cd(II), calculated as $S = P_{4(Cu)} / P_{4(M)}$, are 2.9 and 1.3, respectively. For DDMI separation coefficients $SCu(II)/Zn(II)$ decreased (1.39), but $SCu(II)/Cd(II)$ increased (1.58).

4 Conclusion

The results of the presented investigation prove that 1,2,4-trimethylimidazole and 1-decyl-2,4-dimethylimidazole in dichloromethane can be successfully used as the selective extractant for copper(II) from the Cu-Zn-Cd mixture. For TMI, separation coefficients for Cu(II) with regard to zinc and cadmium are 2.9 and 1.3, respectively. For DDMI separation coefficients for Cu(II) with regard to zinc and cadmium are 1.4 and 1.6, respectively. Sparingly soluble in water 1-decyl-2,4-dimethylimidazole is a more useful extractant. It can be used to separate Zn (II) from Cd (II) at a pH of 3 - 4.5.

Moreover, the investigation indicates that trialkylimidazole with all of the test metal cations tend to form 4-coordination complexes in addition to 6-coordination ones in the solution. This is possible because, in the case of Cu, Cd and Zn ions, a change of the coordination number takes place at various stages of complexation. Moreover, the configurational equilibrium tetrahedron \leftrightarrow octahedron for Zn(II) is clearly shifted more to the right than for Cd(II). This leads to an increase in all total stability constants β_n and an increase in the values of their partition ratios P_n .

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