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SIMULTANEOUS SEPARATION OF COPPER(II), SILVER(I) AND GOLD(III) USING PHTHALOYL-BIS-THIOSEMICARBAZONE BY FLOTATION TECHNIQUE

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ABSTRACT

Sensitive and selective flotation technique was investigated for separation of Cu(II), Ag(I) and Au(III) ions from aqueous media using phthaloyl-bis-(thiosemicarbazone), H2PhTSC, as chelating agent and oleic acid (HOL) as surfactant. The solution pH, chelating agent, analyte and surfactant concentrations as well as the effect of some foreign ions, which influence the separation process, were examined. Under the optimum conditions, the separation of about 100% of the analytes was attained. The procedure was successfully applied for the flotation of the analytes spiked to some natural water samples in addition to the recovery of silver and gold in pure metallic state. Moreover, the mechanism of flotation is suggested. A for the simultaneous method separation and determination of the analytes is elaborated, based on the addition of excess H₂PhTSC and floating the species with HOL at pH 2.5 - 5.0 for Ag(I), 1.0 - 6.0for Au(III) and pH 4.0 - 7.0 for Cu(II) ions.

Keywords: flotation, copper, silver, gold, technique, HOL

1 INTRODUCTION

Pre-concentration and separation techniques are necessary for reliable determination of traces metal ions. Numerous techniques for the separation and pre-concentration of metal ions including solvent extraction, [Saad, et al., (2007); Fu et al., (1995) and Satio, et al., (2000)] co-precipitation, [Arpadjan, et al., (1993)] ion exchange,

[Akatsuka, et al., (1995); Minamisawa, et al., (1996) and Matsubara, et al., (2000)] electro-osmosis, immobilizing reagents, [Ghazy, et al., (2003)] adsorption, [Kovalev, et al., (2000), Oh & Choi (2000) and Su, et al., (1995)] solid phase extraction [Saad, et al., (2008); Elci, et al., (2003); Kenduzler, et al., & Turker (2002); Liu, et al., (2000); Oleschuk & Chow (1998); Soylak, et al., (2000) and Hajkova, et al., (2000)] and flotation have been reported. Although some of these techniques may be tedious, having limiting concentration factors, lengthy and rigid conditions for separation by solid adsorbents, [Leyden & Wescheider (1981)] the flotation separation process is considered to be a promise technique for removing toxic metal ions from diluted aqueous solutions owing to its simplicity, rapidity, economy, good separation yield (10⁻⁶ - 10⁻² M), large possibility of application or species having different nature and structure, flexibility and friability of equipment and processing for recovery purpose. [Stoica, et al., (1998)] It is believed that this process will soon be incorporated as a clean technology to treat water and waste water. [Rubio, et al., (2002) and Capponi, et al., (2006)].

Although number of flotation procedures for separation and recovery of Cu(II), Ag(I) and Au(III) ions have been reported, [Kabil, et al., (1997); Ghazy, et al., (2006), Ghazy & El- Morsy (2008) and Mostafa, et al., (2006)] there is no work in the available literature on the application of phthaloyl-bis-(thiosemicarbazone), H_2PhTSC (Fig. 1) in this respect. Therefore, as a part of my interest in the pre-concentration and separation of toxic and precious metal ions, this study is reporting simple and economic processes for simultaneous separation and recovery of copper, silver and gold in pure metallic state under recommended conditions using phthaloyl-bis-(thiosemicarbazide), H_2PhTSC , as a chelating agent and oleic acid as a surfactant.

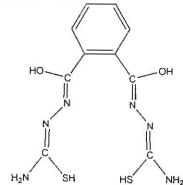


Fig.(1):Structure of phthaloyl-bis (thiosemicarbazide), H₂PhTSC.

EXPERIMENTAL 2

2.1 **Reagents and solutions**

Phthaloyl-bis-(thiosemicarbazone) (H2PhTSC) was synthesized as we have been reported (Abou Hussen A. et al 2005). It was prepared by refluxing diethylphthalate (11.1 g, 0.05 mol) in ethanol (30 ml) with thiosemicarbazide (9.1 g, 0.1 mol). The precipitate was filtered, washed with ethanol, re-crystalized and air-dried. The stock solution of H₂PhTSC $(1 \times 10^{-2} \text{ moll}^{-1})$ was prepared by dissolving the requisite amount in an aqueous - ethanol solution (1:1). Oleic acid (HOL) stock solution (6.36 x 10⁻² moll⁻¹) was prepared by dispersing 20 ml (food grade with sp.gr, 0.895, provided from J.T. Baker Chemical Co.) in one litre kerosene. Stock solutions of the analytes $(1 \times 10^{-2} \text{ moll}^{-1})$ were prepared from CuCl₂.2H₂O, AgNO₃ and HAuCl₄.3H₂O (analytical grade) in bi-distilled water. To study the interference effect, 0.1 moll⁻¹ stock solutions of Mn(II), Fe(III), Co(II), Ni(II), Zn(II), Cd(II), Hg(II), Al(III), Pb(II), Ba(II), Ca(II), Mg(II), K(I) or Na(I) as chlorides, sulphates, nitrates or acetates were applied.

2.2 Apparatus

pH were measured using Jeanway 3310 pH meter. IR spectra were recorded on Mattson 5000 FT-IR spectrophotometer using KBr disk method. The concentration of the analytes Cu(II), Ag(I) and Au(III) was determined using a Perkin- Elmer 2380 atomic absorption spectrophotometer with air-acetylene flame at 324.8, 328.1 and 242.8 nm, respectively. Elemental analyses (C, H, Cl, N and S) were performed at the Micro analytical Unit, Faculty of Science, Cairo University, Egypt. The flotation cell used was a cylindrical graduated tube of 16 mm inner diameter and 290 mm length with a stopper at the top and a stopcock at the bottom.

2.3 Procedure

Into a flotation cell, 0.5 ml of Cu(II), Ag(I) or Au(III) (1 x 10⁻³ moll⁻¹) was mixed with 1 ml of H₂PhTSC (1 x 10^{-3} moll⁻¹) and 5 ml bidistilled water. The pH was adjusted to the optimum value for each analyte by adding drops of HCl for Cu (II) and Au(III), and HNO₃ for Ag (I) and / or NaOH and then completed to 10 ml with bi-distilled water. The cell was shaken for few seconds to insure complete complexation. To the flotation cell 2 ml of HOL (1 x 10^{-3} moll⁻¹) was added and then

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inverted many times by hand to ensure complete separation of two phases. Meanwhile the stopper was removed to ensure passage of air. After 5 min. standing, the float was filtered off and the concentration of the analytes in both mother liquor and the float after dissolving in drops of concentrated HNO₃ was determined by flame atomic absorption spectrometry (FAAS). All measurements were carried out at 25 ± 0.5 °C. The separation efficiency was calculated using the relation:

 $F = (C_i - C_f / C_i) \times 100\%$ or $F = (C_s / C_i) \times 100\%$

where C_i , C_f and C_s denote the initial, final concentrations in the mother liquor and the concentration of the analyte in the float, respectively.

2.4 Simultaneous Separation

1 ml of H₂PhTSC (1 x 10^{-2} moll⁻¹) into a flotation cell was mixed with 0.5 ml of Cu(II), Ag(I) and Au(III) (1 x 10^{-3} moll⁻¹). pH was adjusted to 2.0 – 2.5 by adding drops of HNO₃ (0.1 moll⁻¹) then completed to 10 ml with bi-distilled water. The cell was shaken for few seconds to insure complete complexation. To the flotation cell 2 ml of HOL (1 x 10^{-3} moll⁻¹) was added and then inverted many times by hand to ensure complete separation of two phases. Meanwhile the stopper was removed to ensure passage of air. After 5 min., on standing, the concentration of Ag(I) and Au(III) in the scum were separated, stripped with HNO₃ and subjected to the flame atomic absorption spectrometry (FAAS). The mother liquor was transferred quantitatively to another flotation cell. The pH was raised to 5.5 – 7.0 by adding drop of NaOH (0.1 moll⁻¹). The concentration of Cu(II) in the scum was determined by flame atomic absorption spectrometry (FAAS) after treatment with concentrated HNO₃.

3 • RESULTS AND DISCUSSIONS

3.1 Effect of variables

In order to obtain the optimum conditions for the maximum flotation efficiency of Cu(II), Ag(I) and Au(III) ions using H₂PhTSC as a chelating agent and HOL surfactant, the different factors affecting the flotation process have been studied.

3.1.1. Effect of pH

The solution pH is a very important factor for metal-chelate formation and flotation processes. Therefore, the effect of the pH on the separation efficiency of Cu(II), Ag(I) and Au(III) ions with H₂PhTSC using HOL surfactant was studied in the range 1 - 8 by adding HCl for Cu(II) and Au(III), and HNO₃ for Ag(I), and / or NaOH solutions (Fig.2). The maximum separation efficiency (~100%) could be obtained in the pH range 4 - 7, 3 - 5 and 1 - 7 for Cu(II), Ag(I) and Au(III) ions, respectively. For subsequent experiments pH = 6 for copper, pH = 5 for silver and pH = 4 for gold were chosen as suitable pHs.

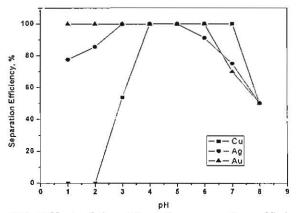


Fig. (2): Effect of the pH on the separation efficiency

3.1.1 Effect of chelating agent concentration (H₂PhTSC)

The amount of H₂PhTSC is another important factor that affects the separation efficiency. A series of experiments were conducted to investigate the relation between the floatability of Cu(II), Ag(I) and Au(III) ions $(1 \times 10^{-4} \text{ moll}^{-1})$ and the amount of H₂PhTSC using 2×10^{-3} moll⁻¹ HOL. Fig. 3 shows that the maximum separation efficiency (~100%) is obtained at 1:1 and 2:1 (analyte: H₂PhTSC) ratios for Cu(II), Ag(I) and Au(III) ions, respectively. Moreover, the excess amount of H₂PhTSC has no adverse effect on the flotation process and so the procedure can be applied to unknown samples containing Cu(II), Ag(I) and/ or Au(III) ions.

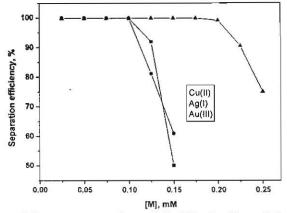


Fig.(4): Effect of the concentration of Cu(II), Ag(I) and Au(III) ions on the separation efficiency.

3.1.3 Effect of HOL concentration

To study the effect of surfactant (HOL) concentration on the floatability and separation of 5×10^{-5} moll⁻¹ of Cu(II), Ag(I) and Au(III) in presence of 1×10^{-4} moll⁻¹ H₂PhTSC, various experiments were carried out. The floation efficiency increases to maximum value (~100%) in the concentration ranges $5 \times 10^{-4} - 5 \times 10^{-3}$ moll⁻¹ of HOL for Cu(II) and Ag(I), and $1 \times 10^{-4} - 5 \times 10^{-3}$ moll⁻¹ for Au(III). Thus, 1×10^{-3} moll⁻¹ of HOL was fixed throughout all measurements for separation of Cu(II), Ag(I) and Au(III).

3.1.4 Effect of diverse ions

The concentration procedure for trace metals is strongly affected by the addition of various metal ions individually or in combination to the sample. Thus, the reliability of the proposed method was examined in the presence of different interfering ions in water samples. The possible interference of a number of cations and anions in the separation of 5×10^{-5} moll⁻¹ of Cu(II), Ag(I) and Au(III) were examined under the optimum conditions. The cations; Al(III), Pb(II), Ba(II), Mg(II), K(I), Na(I), Co(II), Ni(II), Zn(II), Cd(II) and Mn(II) individually or in combination have no pronounced effect on the separation efficiency of the studied analytes while Hg(II) and Fe(III) cations have a significant interference (Table 1). These effects can be completely eliminated by the addition of excess amount of H₂PhTSC (5×10^{-3} moll⁻¹). Moreover, the

applied procedure is suitable and applicable for the pre-concentration and separation of analytes under study from saline and complex matrix samples.

3.2 Applications

The proposed method was applied to natural water samples. The recovery of known amounts of Cu(II), Ag(I) and Au(III) added to tap water, river Nile, lake and midetrenian sea samples was examined with the above procedure. To 10 ml of clear filtered water samples, Cu(II), Ag(I) or Au(III) (0.05 mM) was introduced and 2 ml of H₂Phstc (1 mM) was added.' The above mentioned procedure of separation and determination were carried out. The data obtained (Table 2) demonstrate that the applied procedure for the separation of Cu(II), Ag(I) and Au(III) from different natural water samples is satisfactory.

3.2.1 Simultaneous Separation

The above procedure was successfully applied on three synthetic mixtures with different concentrations of Cu(II), Ag(I) and Au(III) (foreign ions free) and the obtained data are reported in Table 3.

3.2.2 Recovery of silver and gold

A simple economic procedure was proposed for recovering silver and gold from different sources. The procedure depends mainly on changing the different chemical forms of the metal ions into their M^{+n} -H₂Phstc complexes and fusing these complexes in the presence of Na₂CO₃ or borax followed by pouring the molten into a solution of 10% Na₂CO₃. Pellets of metals were obtained (Table 4).

Table (1): The effect of some foreign ions on the removal of Cu(II),Ag(I) and Au(III)(0.05 mM) using H2PhTSC (1 mM) andHOL (1 mM) at pH = 6, 5 and 4, respectively

Interfering ion	Concentration µg/ml	Recovery, % ^a			
		Cu(II)	Ag(I)	Au(III)	
Na(I)	5400	99.9	99.9	99.9	
K(I)	4000	99.9	99.9	99.9	
Mg(II)	1200	99.9	99.9	99.9	
Ca(II)	100	99.9	99.9	99.9	
Ba(II)	1350	99.9	99.9	99.9	
Mn(II)	500	99.9	99.9	99.9	
Fe(III)	500	99.9	99.9 ⁶	99.9 ⁶	
Co(II)	600	99.9	99.9	99.9	
Ni(II)	600	99.9	99.9	99.9	
Cu(II)	100	-	99.9	99.9	
Zn(II)	500	99.9	99.9	99.9	
Ag(I)	100	99.9	-	99.9	
Cd(II)	560	99.9	99.9	99.9	
Au(III)	100	99.9	99.9	-	
Hg(II)	300	99.9 ⁶	99.9 ^b	99.9 ⁶	
Al(III)	500	99.9	99.9	99.9	
Pb(II)	500	99.9	99.9	99.9	
Cr	4000	99.9	99.9	99.9	
SO4 ²⁻	950	99.9	99.9	99.9	
NO ₃	5400	99.9	99.9	99.9	
CH ₃ COO ⁻	500	99.9	99.9	99.9	

^a Mean of three replicates.^b addition of 5×10⁻² molL⁻¹ H₂PhTSC.

by pouring the molten into a solution of 10% Na₂CO₃. Pellets of metals were obtained. The results shown in Table 4 indicate that the procedure is

economic for silver and gold recovery (~ 100%) in pure metallic state [Mostafa, et al., (2006)].

	Removal, %			
water sample	Cu(II)	Ag(I)	Au(III)	
Tap water	99.9	99.8	99.9	
Distilled water <u>River water</u>	99.9	99.9	99.9	
Gamassa water	99.8	99.7	99.8	
El-Manzala water	99.8	99.8	99.7	
Ras- Elbar	99.9	99.7	99.8	
Suez water	99.8	99.6	99.4	
		new second s		

Table (2). Removal of 0.1 mM of Cu(II), Ag(I) and Au(III) H₂PhTSC (1 mM) and HOL(1 mM) at pH = 6, 5 and 4, respectively.

Table (3): Simultaneous separation of Cu(II), Ag(I) and Au(III) in synthetic mixture using H₂PhTSC (0.02 molL⁻¹)*

			Concentration,	µg/ml	
Mixture ion	Added	Found	Absolute error	Relative error	
Cu(II)	3.18	3.01	-0.17	-5.65	
Ag(I)	5.40	5.30	-0.1	-1.89	
Au(III)	9.85	9.44	-0.41	-4.31	
Cu(II)	6.35 -	6.18	-0.17	-2.75	
Ag(I)	10.79	10.55	-0.24	-2.27	
Au(III)	19.70	19.43	-0.27	-1.39	
Cu(II)	6.35	6.21	-0.14	-2.25	
Ag(I)	10.79	10.63	-0.16	-1.51	
Au(III)	9.85	9.80	-0.05	-0.51	

*mean of three measurements

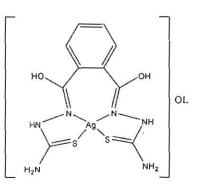
Table (4): Recovery of different concentrations of Ag(I) and Au(III) in	
the presence of H2PhTSC (0.02 molL^{-1}) and HOL (5 mM) at	
pH = 5 and 4, respectively.	

Analyte added(mg/L)	Analyte recovered (mg)		
	Ag	Au	
20	18.7	17.8	
40	39.4	39.1	
60	59.2	58.5	

3.2.3 Separation mechanism

The nature of bonding between the isolated species and HOL is suggested by elemental analyses and IR spectral data of the isolated complexes in the presence and absence of HOL. The IR spectra of isolated Ag(I) complex (Ag-H₂PhTSC) in the presence and absence of HOL are similar with extra bands in case of HOL due to the appearance of new bands at 1605 and 1430 cm⁻¹ attributed to vas(COO⁻) and Us(COO') [Mostafa (2007); Jin, & Ranford (2000); Wagner & Baran (2002); Szorcsik, et al., (2005); Duarte, et al., (1998); Girginova, et al., (2005) and Goher, et al., (1996)]. Also, the appearance of new bands at 2925 and 2855 cm⁻¹ due to vas(CH₂) and vs(CH₂), respectively [Anan, et al., (2011)]. The appearance of the bands at 1500, 1510 and 1385 cm⁻¹ are attributed to δ_{as} CH₃ and δ_{s} CH₃, respectively (Fraile J. M. Et al 2000) This feature is supporting the presence of OL as a counter ion replacing NO3 anion in the unfloated complex [Mostafa, et al., (2006)] (Fig. 2). In comparing the IR spectral data of Cu-H2PhTSC and Au-H2PhTSC complexes with the isolated complexes in the absence and presence of HOL, a new band near 1711 cm⁻¹ may be attributed to v(CO) of -COOH group is observed. In addition, a strong broad band in the 1800-2100 cm⁻¹ region due to the intermolecular hydrogen bonds [Mostafa & Bekheit (2000) and Abou El-Reash, et al., (1991)]. It is clear that the intensity of $\upsilon_{as}(NH_2)$ and $\upsilon_s(NH_2)$ is highly affected in the spectra of the complexes isolated in the scum which may be due to some kind of interactions [Mostafa & Abd El-Maksoud (1998) and Mostafa & Hadjiliadis (2008)]. This feature is acceptable as the unfloated Cu(II) and Au(III) complexes are not charged. The IR spectral data are in

agreement with the elemental analyses (Table 5) [Mostafa, et al., (2006)].



Scheme (1): Structure of floated species

Table (5): Elemental analysis H ₂ PhTSC and its Cu(II), Ag(I) and Au(III)	
complexes in the absence and presence of HOL*	

Compounds	C%	H%	N%	S%	Cl%
H ₂ PhTSC	38.52	3.79	26.70	20.54	
	(38.41)	(3.78)	(26.90)	(20.49)	
[Cu(PhTSC)]	32.03	3.40	22.10	17.20	
	(32.12)	(3.21)	(22.47)	(17.11)	
[Cu(PhTSC)]-HOL	50.98	6.23	12.55	9.83	
	(51.23)	(6.75)	(12.81)	(9.75)	
[Ag(H ₂ PhTSC)]NO ₃	29.19	3.20	19.51	14.89	
	(28.59)	(2.98)	(20.01)	(15.54)	
[Ag(H ₂ PhTSC)]OL	47.40	6.46	11.45	9.05	
	(47.93)	(6.60)	(11.98)	(9.12)	
[Au ₂ (PhTSC)Cl ₄]	14.20	1.25	9.76	7.55	16.40
	(14.19)	(1.18)	(9.93)	(7.56)	(16.78)
[Au2(PhTSC)Cl4]-HOL	29.89	4.00	7.39	5.57	12.80
	(29.79)	(3.90)	(7.45)	(5.67)	(12.58)

*Calculated data in parentheses.

CONCLUSION

Sensitive and selective flotation technique was applied for separation of Cu(II), Ag(I) and Au(III) ions from aqueous media using phthaloyl-bis-(thiosemicarbazone), H₂PhTSC, as chelating agent and oleic acid (HOL) as surfactant. Under the optimum conditions, the separation of about 100% of the analytes was attained. The procedure was successfully applied for the flotation of the analytes spiked to some natural water samples in addition to the recovery of silver and gold in pure metallic state. Simultaneous separation and determination of the analytes is was applied, by adding excess H₂PhTSC and floating the species with HOL at pH 2.5 – 5.0, 1.0 - 6.0 and 4.0 – 7.0 for Ag(I), Au(III) and Cu(II) ions, respectively.

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الملخص العربى

الفصل المتزامن للنحاس، الفضية و الذهب باستخدام فيثالويل بيس ثيوس يميكربازون وبتقنية التعويم أستهدف هذا البحث دراسة أنسب الظروف لفصل وتقدير أيونسات عناصسر النحاس الثنائية والفضبة الأحادية والذهب الثلاثي فرادي أو مجتمعين من المحاليل المائية. بطريقة التعويم وهي الرقم الهيدروجيني، تركيز المرتبط، تركيز العامل الــسطحي النــشط و تركيز الأيونات محل الدراسة. الدور الرئيسي الذي يلعبه المرتبط في عملية الفصل الكمي لهذه العناصر والذي وصل الى معدل فصل يساوى الوحدة . وقد له خط أن تركيز ١×٢٠ ⁻¹ مول لتر - (من أى من هذه العناصر يلزمه ١×١٠ ، مول لتر - (و١×١٠ ، مول لتر - (من المرتبط لعملية الفصل الكمي وباستخدام ١×١٠⁻⁷ مول لتر⁻¹ من العامل السطحي النشط. عند الــرقم الهيدروجيني ٢,٥ - ١,٠ ، ١,٠ - ١,٠ و ٧,٠ - ٧,٠ لأيونات الفضية والذهب و النحاس على التوالي. وقد بينت النتائج ان التداخلات المحتملة من العناصر المصاحبة وبتركيزات عالية نسبياً وباستخدام ١ × ١٠- مول لتر أ من المرتبط والعامل السطحي النشط لا تؤثر الا في حالة الحديد الثلاثي وقد تم التغلب عليه باستخدام ٥ ×١٠٠ ^{-٢} من المرتبط. تم تطبيق الطريقــة المقترحة لفصل كميات ضئيلةومضافة الى عينات مانية من مصادر مختلفة والتي تم فصلها فصلاً تاماً كما تم تطبيق الفصل المتزامن للعناصر الثلاثة مجتمعة عن طريق التغيير في الرقم الهيدروجيني الذي ضبط عند ١,٥ باستخدام ١,٠ مولار حمض النيتريك وتـم فـصل الفضة والذهب. برفع الرقم الهيدروجيني حتى ٦تم فصل النحاس . كما تم استرجاع الذهب و الفضة في صورتها الفازية النقية كما تم وضع ميكانكية لعملية الفصل بناءا على الدراسات الكيمائية والفيزيائية للمتر اكبات المتكونة في غياب ووجود العامل السطحي النشط.