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26th
*International
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Ecological
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EDITOR

Snežana Šerbula

12-15 June 2018, Hotel Jezero, Bor Lake, Serbia



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SYNTHESIS AND APPLICATION OF THIOCARBAMATES OBTAINED BY OXIDATIVE TREATMENT OF WASTE XANTHATE

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Abstract

In this paper, the optimal treatment of industrial waste consisting from xanthate and oxidation product, i.e. diisobutyl and diethyl dixanthogenate, was developed for production of flotoreagents. Waste dixanthogenate was generated during production and storage of flotation agents, i.e. potassium isobutyl (KiBuX) and potassium ethyl xanthate (KEtX), respectively. The process of waste xanthate treatment is based on the reaction of the nucleophilic heterolysis of the persulfide bond in the diisobutyl dixanthogenates by alkylamines in presence of various oxidizing agents (sodium hypochlorite, hydrogen peroxide, potassium persulfate) to produce N-alkyl-, N,N-dialkyl- and N-cycloalkyl-O-isobutyl thiocarbamate selective flotoreagents. Also, analogous methodology was applied for synthesis of N-alkyl and N,N-dialkyl-O-ethyl thiocarbamate from KetX using sodium hypochlorite at laboratory and semi-industrial level. The developed method provides the corresponding alkyl thiocarbamates in a high yield and purity. The flotation efficiency was analysed using the obtained thiocarbamate on a real sample of minerals in laboratory and industrial conditions.

Keywords: Alkyl xanthate, Alkyl thiocarbamate, Dixanthogenates, Flotation

INTRODUCTION

Thiocarbamates are derivatives of thiocarbamic acid, i.e. salts and esters of thiocarbamic acids [1,2]. Structural characteristics, such as the direct bond between the thioacyl group and the nitrogen, contribute to their pronounced biological activity [3]. These compounds have a wide application thus they are produced at industrial level and used as fungicides [4-6], bactericides [5-7], herbicides [8,9], germicides [10], pesticides [11-13], and insecticides [14-16]. Also, alkyl thiocarbamates are used as polymerization accelerators and selective flotoreagents [17].

Besides the known procedures of thiocarbamates syntheses [18-22], many others were developed by reacting mono and dithiocarbamic acid-*O,S*-diesters with primary or secondary amine [23], alkaline xanthate, amine and oxidizer [24], thiol and isocyanate in the presence of a catalyst [25], sodium or potassium alkyl xanthates with aliphatic amines and sulfur [26], xanthates and amines in the presence of nickel(II) sulfate heptahydrate catalyst [27], oxidation of amine salts of xanthic acid with hydrogen peroxide or sodium hypochlorite [28], and oxidation of amine salts of dithiocarbamic acids with ammonium peroxodisulfate [29].

In this paper the optimal laboratory synthesis of *N*-alkyl-, *N,N*-dialkyl- and *N*-cycloalkyl-*O*-isobutyl thiocarbamate selective flotoreagents from industrial waste xanthate and the corresponding amines in the presence of various oxidizing agents: peroxide, sodium hypochlorite and potassium peroxodisulfate were presented. Waste xanthate from production of potassium isobutyl xanthate (KiBX) and potassium ethyl xanthate (KEtX) consists of starting xanthate and dixanthogenates [30]. Innovative procedure relate to optimal technological treatment which provided efficient conversion to thiocarbamate products. Second part of the work was related to synthesis of the *N*-alkyl and *N,N*-dialkyl-*O*-ethyl thiocarbamate from diethyl dixanthogenates in presence of sodium hypochlorite at laboratory and semi-industrial level. Moreover, the comparative study on flotation efficiency of the synthesized and commercial thiocarbamates for copper isolation from real mineral copper ore sample was performed.

MATERIALS AND METHODS

Synthesis of isobutyl thiocarbamate from KiBX and amines in the presence of potassium peroxodisulfate (Method 1)

Synthesis of isobutyl thiocarbamates was performed according to literature procedure [33]. The yield and purity of the products are determined by the GC method and are given in Table 1.

In a manner analogous to the procedure described above, the synthesis of thiocarbamate in the presence of hydrogen peroxide (Method 2) and sodium hypochlorite (Method 3) as an oxidizing agents was carried out [33], and the yields and purity of the products, determined by the GC method, are given in Table 1.

Synthesis of ethyl thiocarbamates from waste diethyl dixanthogenates

Synthesis of thiocarbamates from waste dixanthogenates, separated from waste xanthates, was carried out by reaction of the corresponding alkyl amines in the presence of sodium hypochlorite [34]. All other ethyl thiocarbamates are synthesized in an analogous manner to the procedure above, using the appropriate amines under reaction conditions presented in Table 2.

RESULTS AND DISCUSSION

Synthesized *N*-alkyl, *N,N*-dialkyl- and *N*-cycloalkyl-*O*-isobutyl thiocarbamate are characterized by FTIR, ¹H and ¹³C NMR results, and obtained results are in accordance to literature data [30,33]. The purity is determined by the GC method and confirmed by elemental microanalysis. The yields and purity of the synthesized thiocarbamate, obtained by methods 1, 2 and 3, are shown in Table 1.

Based on the yields of the reactions products (Table 1), the most significant oxidation agent is potassium peroxodisulfate, and the yields are relatively similar and somewhat higher in oxidative processes than in those obtained by applying the conventional synthesis process of aminolysis of sodium isobutyl xanthogenacetate [30].

Table 1 Yields and purity of synthesized isobutyl thiocarbamate

Compound	Yield [%]			GC purity [%]		
	H ₂ O ₂	NaOCl	K ₂ S ₂ O ₈	H ₂ O ₂	NaOCl	K ₂ S ₂ O ₈
iBuOC(S)NHEt	87.2	85.1	90.3	98.5	98.3	98.6
iBuOC(S)NHPr	88.6	86.5	91.7	97.3	97.5	97.2
iBuOC(S)NHnBu	89.5	86.8	92.6	99.0	99.1	99.2
iBuOC(S)NHsBu	89.3	87.2	92.4	99.4	99.3	99.1
iBuOC(S)NHiPr	87.1	85.0	90.2	99.2	99.0	99.2
iBuOC(S)NHiBu	93.6	92.5	96.7	97.0	97.2	97.8
iBuOC(S)NHiPent	94.1	92.0	97.3	97.5	97.7	97.9
iBuOC(S)NHcPr	77.4	75.6	80.2	97.8	98.0	98.6
iBuOC(S)NHcPent	83.6	82.4	86.7	98.0	98.1	98.5
iBuOC(S)NHcHeks	87.7	85.6	90.8	97.0	97.2	97.9
iBuOC(S)N(Et) ₂	93.8	91.7	96.5	97.4	97.6	97.5
iBuOC(S)N(Pr) ₂	95.2	94.0	98.0	98.0	98.1	98.8
iBuOC(S)N(Bu) ₂	95.9	95.1	98.2	97.0	97.2	97.9

Waste diethyl dixanthogenates treated with various alkylamines in the presence of sodium hypochlorite oxidant gave ethyl thiocarbamate product (Table 2).

Table 2 Yields and purity of *N*-alkyl and *N,N*-dialkyl-*O*-ethyl thiocarbamate

Compound	Reaction time [h]	Temperature [°C]	Yield [%]	GC purity [%]
EtOC(S)NHMe	2.0	30 - 45	89.2	97.9
EtOC(S)NMe ₂	2.0	30 - 45	90.0	97.8
EtOC(S)NHEt	2.0	30 - 45	88.0	97.0
EtOC(S)NEt ₂	2.0	30 - 45	88.8	97.6
EtOC(S)NHPr	2.0	30 - 45	88.2	98.0
EtOC(S)NPr ₂	3.0	40 - 50	84.5	98.4
EtOC(S)NHi-Pr	3.5	40 - 55	82.1	99.6
EtOC(S)Ni-Pr ₂	4.5	40 - 55	75.0	98.9

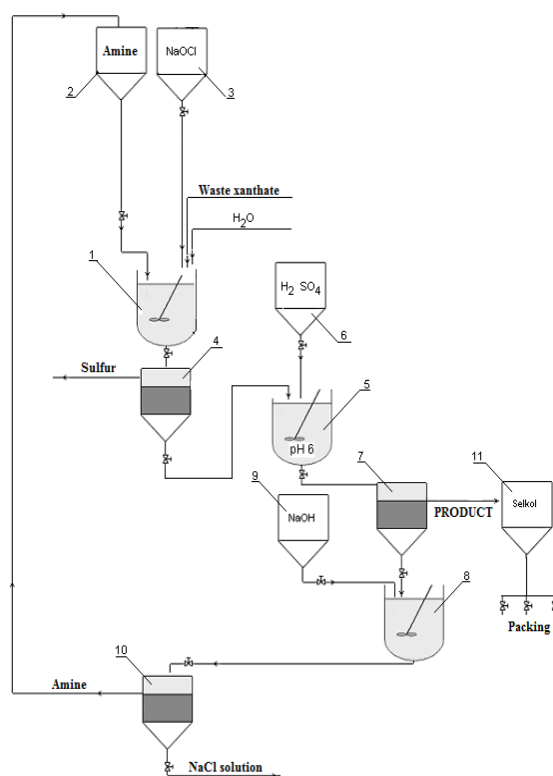
Based on the results presented in Table 2 high yields and purity of *N*-alkyl and *N,N*-dialkyl-*O*-ethyl thiocarbamate were obtained, and depends on the structure of the amines used. Voluminosity of di-*n*-propyl, isopropyl, and di-isopropyl group contributes significantly to steric repulsion which prevents amines to performe an effective nucleophilic heterolysis of S-S bonds in the first step of the reaction. Amine structure has essential impact on the nucleophilicity of amines [31]. Lower yields were obtained for *N,N*-di-propyl-*O*-ethyl thiocarbamate (84.5%), *N*-isopropyl-*O*-ethyl thiocarbamate (82.1%), while lowest for *N*-diisopropyl-*O*-ethyl thiocarbamates (75.0%). MS, FTIR, ¹H and ¹³C NMR data are in accordance with literature [31]. The amount of sulfur produced after the filtration of the reaction mixture corresponds to the theoretical stoichiometric value in relation to the starting quantities of the reactants.

In accordance to optimal laboratory synthesis semi-industrial production was carried out at H. I. Župa Kruševac plant. It was found that the reaction products are not present in waste water, while the concentration of dixanthogenates is below the maximum contamination limit, Table 4 [32]. Necessary wastewater treatment is also very simple. An innovative method could be widely used for the synthesis of thiocarbamate starting from various raw materials: ammonium salts and alkaline salts of *O*-alkyl xanthic acid [33], as well as waste or commercial diethyl dixanthogenate [34]. The results of the implementation of the defined method at the semi-industrial level are shown in Table 3.

Table 3 Results of semi-industrial synthesis of *N*-ethyl-, *N*-propyl and *N,N*-dipropyl-*O*-ethyl thiocarbamate

Thiocarbamates	Reactants								Reaction conditions		By-product	Product		
	Amine		H ₂ SO ₄		Sodium ethyl xanthate		NaOCl		t	T	Sulfur	Yield	GC	
	kg	kmol	kg	kmol	kg	kmol	m ³	kmol	h	°C	kg	kg	%	%
EtOC(S)NHEt	35.4	0.55	28.1	0.28	144	1.0	0.66	1.5	2.5	30.0	25.0	126.5	95.0	99.0
EtOC(S)NH(nPr)	33.2	0.55	28.1	0.28	144	1.0	0.66	1.5	2.8	30.0	24.0	135.2	92.0	98.6
EtOC(S)N(nPr) ₂	54.4	0.55	28.1	0.28	144	1.0	0.66	1.5	4.0	30.0	20.0	161.0	85.2	98.1

The technological scheme of the semi-industrial process for the synthesis of thiocarbamate is presented in Figure 1. Results of determination of isobutyl dixanthogenate in wastewater in the process of semi-industrial production are presented in Table 4.

**Figure 1** Technological scheme of the semi-industrial synthesis of ethyl thiocarbamate**Table 4** Concentrations of ethyl dixanthogenate in waste water

Compound	Sample amount [cm ³]	Concentration of ethyl dixanthogenate	
		c · 10 ³ [mmol dm ⁻³]	c · 10 ² [mg dm ⁻³]
EtOC(S)NHEt	70.00	21.36	58.00
EtOC(S)NHnPr	85.00	20.09	53.80
EtOC(S)NHPr	85.00	19.98	54.31
EtOC(S)NHBu	80.00	18.45	50.29

The product obtained by the defined procedure was examined in the flotation process on a real mineral sample. The results of comparative studies on flotation efficiency, synthesized

versus commercial, of copper ore sample from the Elacite-Bulgaria are presented in Tables 5 and 6.

Table 5 Flotation results obtained using commercial *N*-ethyl *O*-isobutyl thiocarbamate

	W, [g]	w, [%]	Cu, [%]	Cu, [g]	ICu, [%]
Entrance	996.9	100	0.454	4.5284	100
BCCu	36.9	3.70147	11.350	4.1881	92.4845
KKCu	14.5	1.4545	1.030	0.1493	3.2980
UKCu	51.4	5.1560	8.438	4.3375	95.7820
Tailings	945.5	94.8440	0.020	0.1909	4.2175

*W- weight of ore sample, w- mass % of ore sample, ICu-copper content BCCu-basic concentrate, KKCu- prolonged flotation, UKCu-total flotated copper, Tailings- copper content in tailings.

Table 6 Flotation results obtained using synthesized *N*-ethyl *O*-isobutyl thiocarbamate

	W, [g]	w, [%]	Cu, [%]	Cu, [g]	ICu, [%]
Entrance	1000.6	100	0.460	4.6088	100
BCCu	40.5	4.0475	10.820	4.3821	95.0794
KKCu	12.2	1.2192	0.810	0.0988	2.1441
UKCu	52.7	5.2668	8.502	4.4809	97.2230
Tailings	947.9	94.7332	0.013	0.1279	2.7765

Based on the results shown in Tables 5 and 6, it can be seen that synthesized flotoreagents are more efficient flotation reagent. Analogous comparative study was carried out with real copper ore sample from JAMA - Bor mine, and obtained results are given in Tables 7 and 8.

Table 7 Flotation results obtained by using commercial *N*-ethyl *O*-ethyl thiocarbamate

	T, [g]	t, [%]	Cu, [%]	Cu, [g]	ICu, [%]
Entrance	990	100	0.657	6.5072	100
UKCu	195	19.6969	3.123	6.0906	93.5981
Tailings	795	80.3030	0.052	0.4165	6.4018

*Kcu – flotated copper by basic flotation, Tailings– copper content in tailings.

Table 8 Flotation results obtained using synthesized *N*-ethyl *O*-ethyl thiocarbamate

	T, [g]	t, [%]	Cu, [%]	Cu, [g]	ICu, [%]
Entrance	995	100	0.696	6.9346	00
UKCu	210	21.1001	3.1385	6.5908	95.0418
Tailings	785	78.8912	0.044	0.3438	4.9581

The obtained results on the flotation efficiency related to the real copper ore sample from JAMA-Bor mine, Tables 7 and 8, showed higher flotation efficiency of synthesized thiocarbamate comparing to commercial one.

CONCLUSION

Synthesis of *N*-alkyl-, *N,N*-dialkyl- and *N*-cycloalkyl-*O*-isobutyl thiocarbamate from amine salts of xanthic acid obtained from KiBX separated by waste xanthate treatment with potassium peroxodisulfate, hydrogen peroxide and sodium hypochlorite. Highest yields in

presence of $K_2S_2O_8$ (80.2-98.2%), lower in the presence of H_2O_2 (77.4-95.9%), and the lowest in the presence of $NaOCl$ (75.6-95.1%). The process for the synthesis of *N*-alkyl and *N,N*-dialkyl-*O*-ethyl thiocarbamate from waste diethyl dixanthogenates and amines in the presence of sodium hypochlorite was developed at the laboratory level and applied at the semi-industrial level. The structures of all synthesized thiocarbamates were confirmed by FTIR, 1H and ^{13}C NMR spectroscopy, as well as MS spectrometry. The purity was determined using GC chromatography and elemental analysis. This new environmentally acceptable process represents a favorable possibility for existing methods, and offers a significant contribution to environmental protection. The results of comparative studies related to flotation efficiency from copper ore samples showed higher percentage of floated copper using synthesized thiocarbamates in comparison to commercial one.

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