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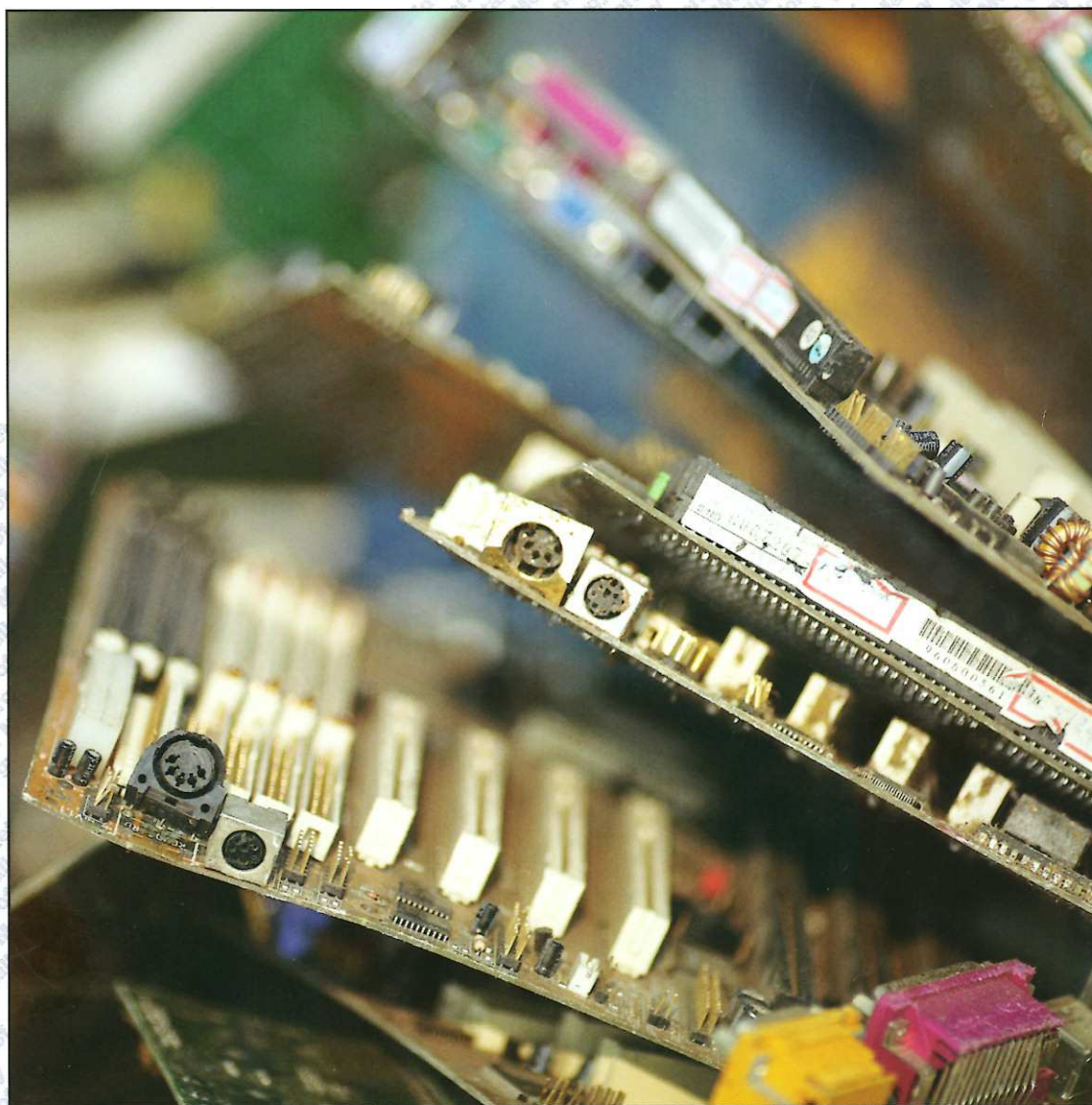
# ERZMETALL

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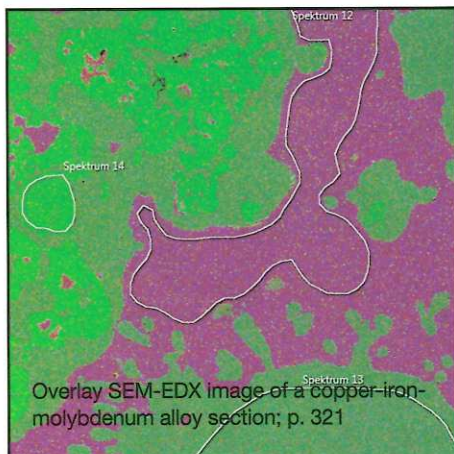


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# ERZMETALL World of Metallurgy

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# Cobalt Precipitation from Leachate Originated from Leaching of Spent Li-ion Batteries Active Mass – Characterization of Inputs, Intermediates and Outputs

Zita Takacova, Vladimir Dzuro, Tomas Havlik

The paper is focused on the experimental study of cobalt precipitation from leachate originated from leaching of active mass of spent lithium batteries (LiBs). The work contains results of preliminary experiments with following precipitating agents – NaOH, Na<sub>2</sub>CO<sub>3</sub> and oxalic acid. During these experiments, high efficiency of cobalt precipitation was achieved, however co-precipitation of other accompanying metals was also observed. Oxalic acid was determined as the most suitable agent for cobalt precipitation. During experiments with oxalic acid, the effect of its concentration, temperature and precipitation time on the process efficiency was investigated. Optimum precipitation conditions were determined as follows: 3× stoichiometric excess of oxalic acid, 80 °C, 1 hour, 500 rpm, where almost

80 % of cobalt was precipitated as cobalt oxalate. In this work, AAS, XRD and SEM-EDX analysis were used for analysis of input and output samples. Despite the theoretical study, it was found that selective cobalt precipitation is practically not possible. By calcination at 400 to 800 °C, cobalt oxalate was transferred into Co<sub>3</sub>O<sub>4</sub> with purity to 78 %, which cannot be considered sufficient. It is recommended to incorporate solvent extraction or other refining process for accompanying metals recovery before precipitation step.

Keywords:

Spent Li-ion batteries (LiBs) – Cobalt – Hydrometallurgy – Leaching – Precipitation – Calcination

## Die Fällung von Cobalt aus der nach der Laugung der aktiven Masse verbrauchter Lithium-Ionen-Akkus entstandenen Lösung – Charakterisierung von Ausgangsmaterial, Zwischenprodukten und Produkten

Diese Arbeit befasst sich mit der experimentellen Untersuchung der Cobaltfällung aus der nach der Laugung der aktiven Masse verbrauchter Lithium-Ionen-Akkus entstandenen Lösung. Sie enthält Ergebnisse vorausgehender Untersuchungen mit den Fällungsagentien NaOH, Na<sub>2</sub>CO<sub>3</sub> und Oxalsäure. In diesen wurde eine hohe Effizienz der Cobaltfällung erreicht, jedoch wurde auch die Mitfällung von Begleitmetallen beobachtet. Oxalsäure wurde als geeignetstes Mittel zur Cobaltfällung bestimmt. In den Experimenten mit Oxalsäure wurde die Abhängigkeit der Prozesseffizienz von deren Konzentration, Temperatur und der Fällungszeit untersucht. Als optimale Fällungsbedingungen wurden dreifacher stöchiometrischer Oxalsäureüberschuss, 80 °C, eine Stunde und 500 U/min bestimmt; dabei wurden ca. 80 % des Cobalts als Cobaltoxalat gefällt. Für die Analyse von

Ausgangsmaterial und Produkten wurden Atomabsorptionsspektrometrie (AAS), Röntgendiffraktometrie (XRD) und Rasterelektronenmikroskopie mit energiedispersivem System (SEM-EDX) eingesetzt. Entgegen den theoretischen Untersuchungen zeigte es sich, dass eine selektive Cobaltfällung praktisch nicht möglich ist. Durch Kalzinierung bei 400 bis 800 °C wurde Cobaltoxalat in eine Co<sub>3</sub>O<sub>4</sub>-Phase mit einem Gehalt von 78 % Co<sub>3</sub>O<sub>4</sub> überführt, was als unzureichend anzusehen ist. Es wird empfohlen, Lösungsmittelextraktion oder ein anderes Raffinationsverfahren für die Begleitmetalle vor der Fällungsstufe einzubinden.

Schlüsselwörter:

Verbrauchte Lithium-Ionen-Akkus – Cobalt – Hydrometallurgie – Laugung – Fällung – Kalzinierung

## Précipitation de cobalt dans le lixiviat produit pendant la lixiviation de la masse active des batteries Li-ion – caractérisation des produits de départ, intermédiaires et finaux

## Precipitación de cobalto desde lixiviado originando de la lixiviación del material activo de acumuladores Li-ion gastados – caracterización productos de entrada, intermedios y salida

This is a peer-reviewed article.

## 1 Introduction

Currently, LiBs represent a mobile source of electricity in many electrical devices. Their consumption is constantly rising because of the expansion of information technologies as well as hybrid and electric vehicles production. Increasing consumption of LiBs will result in long-term increasing amount of spent LiBs. In addition, the European Union sets targets for the collection and recycling of spent batteries that need to be met. From that reason, their successful recycling is highly actual issue.

The main interest components for spent LiBs recycling is cobalt present in cathode active material at a content of approximately 20 wt.-% [1]. The possibilities of the cobalt recovery from spent LiBs can be divided into pyrometallurgical and hydrometallurgical treatment. Current research in laboratory scale focuses mainly on the sophisticated hydrometallurgical processing – leaching and metals recovery from a leachate.

Leaching of spent LiBs active mass focuses on determination of suitable leaching agent, temperature, L:S ratio and other parameters for effective recovery of Co, lithium and other present metals. The leaching is mostly carried out in acidic leaching agents such as HCl, H<sub>2</sub>SO<sub>4</sub>, HNO<sub>3</sub> [1-8]. The most common leaching agent is represented by H<sub>2</sub>SO<sub>4</sub> at various concentrations with addition of H<sub>2</sub>O<sub>2</sub> as a reducing agent [1, 2, 4, 5, 9-13]. Cobalt can be obtained from the leachate by precipitation with various precipitating agents. Using ammonium oxalate as precipitating agent was studied by [15], oxalic acid by [14, 15, 17], NaOH by [18] and KOH by [19].

In this work, the following precipitating agents were used: NaOH, Na<sub>2</sub>CO<sub>3</sub> and oxalic acid. The aim was to determine an effect of precipitating agent and pH on precipitation efficiency as well as to determine the most suitable precipitating agent. Such comparison of various precipitation agents has not yet been published. Oxalic acid was selected to examine the effect of temperature, concentration, and precipitation time on process efficiency. Part of the work is dealing with a calcination of obtained precipitate at 400, 600 and 800 °C, in order to transfer the obtained oxalate to the oxidic form. There is a lack of published information about influence of temperature on calcination efficiency of the obtained product and its detailed analysis.

## 2 Experimental

### 2.1 Analytical methods

The chemical composition of inputs, intermediates and products was determined by AAS method on Varian Spectrophotometer AA20+. Qualitative XRD analysis was performed on PANalytical X'PERT PRO-360. SEM-EDX analysis was performed using MIRA3 TESCAN instrument.

### 2.2 Material

The input leachate was obtained by leaching the active mass in H<sub>2</sub>SO<sub>4</sub> with the addition of 10 vol.-% H<sub>2</sub>O<sub>2</sub>. Active mass, with a particle size of +0 –0.71 mm, was coming from mechanical pre-treatment of spent LIBs from cell phones consisting of grinding, milling and sieving. The chemical composition of the active mass is shown in Table 1. The XRD pattern of the active mass is shown in Figure 1. SEM-EDX analysis of the active mass was performed in different spectrum (Figure 2) at 406× magnification. Figure 3 shows

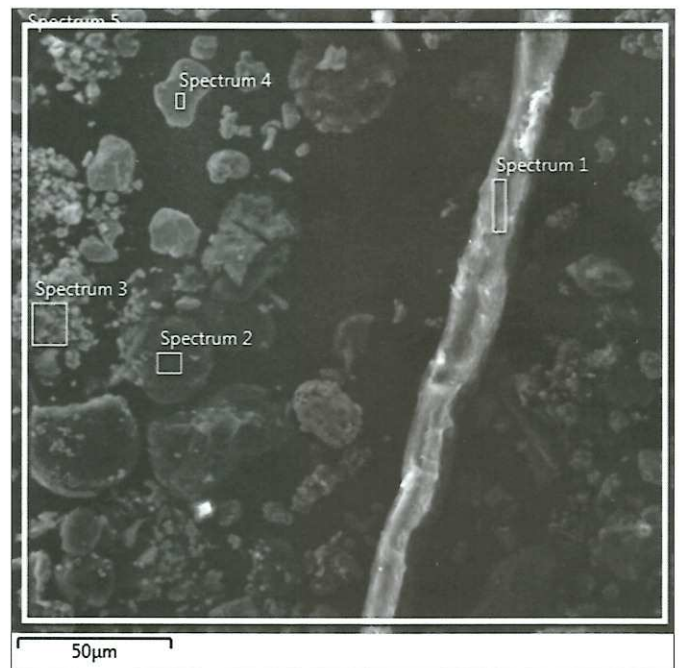


Fig. 2: Spectrum of active mass indicated for SEM-EDX analysis

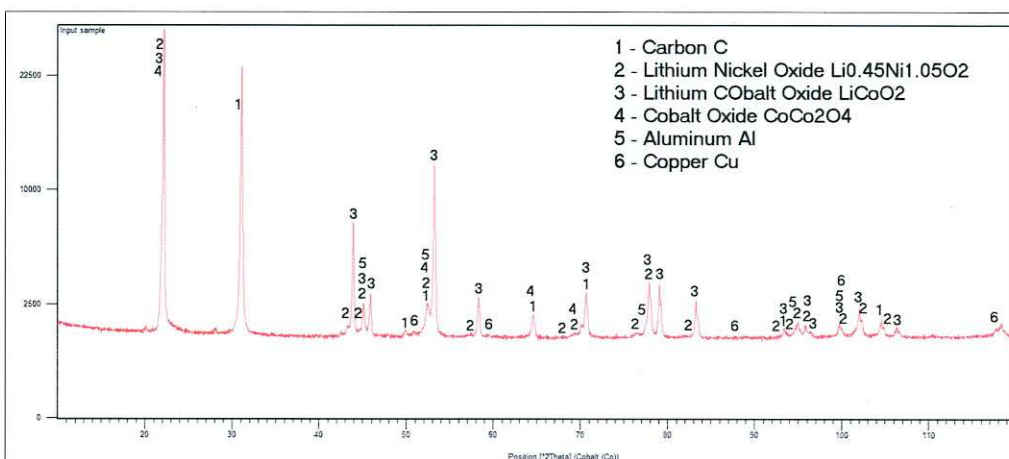


Fig. 1: XRD pattern of the active mass

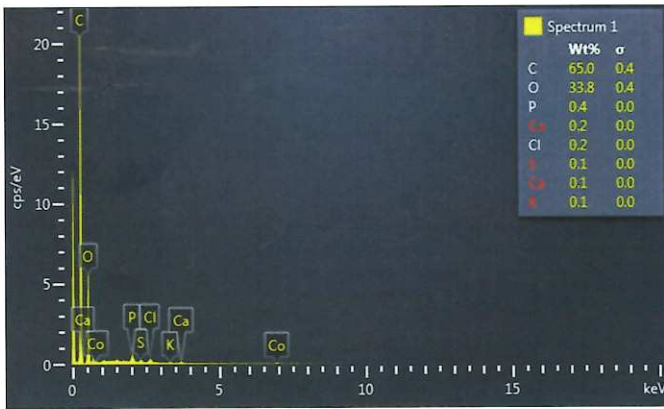


Fig. 3: Chemical composition of Spectrum 1, SEM-EDX analysis

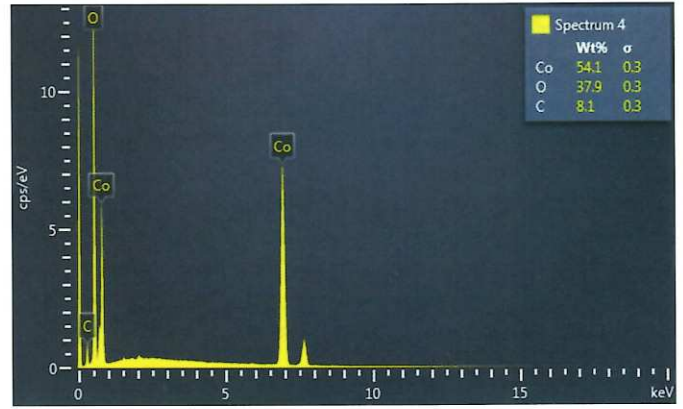


Fig. 4: Chemical composition of Spectrum 4, SEM-EDX analysis

the chemical composition of the area designated as Spectrum 1 and Figure 4 shows the chemical composition of Spectrum 4.

Table 1: Chemical composition of the active mass [%]

Active mass (fine fraction +0 -0.71 mm)	Co	Li	Ni	Cu	Al	Mn	Fe
	22.43	3.65	1.54	1.33	0.72	1.49	1.27

Analytical results confirmed a high content of Co – 22.43 wt.-% as  $\text{LiCoO}_2$  and  $\text{Co}_3\text{O}_4$  in the analysed active mass. Li content in active mass was 3.65 wt.-%. Other metals – Mn, Ni, Cu, Al and Fe – occur in the active mass up to 1.5 wt.-%. These metals were identified as  $\text{Li}_{0.45}\text{Ni}_{1.05}\text{O}_2$ , metallic aluminium and copper and are present as residual electrodes and Al-shells, which cannot be removed by mechanical pre-treatment and their presence is unfavourable. Mn and Ni occur as a component of the cathode active material, alternating the more common  $\text{LiCoO}_2$  into  $\text{LiMO}_2$ , where  $M = \text{Co, Mn, Ni}$  [20].

Leaching conditions (2 M  $\text{H}_2\text{SO}_4$  + 10 vol.-%  $\text{H}_2\text{O}_2$ , L:S = 10, 1 hour, 80 °C, 300 rpm) were determined based on the literature mentioned in the introduction, where this combination has proven to be one of the most powerful

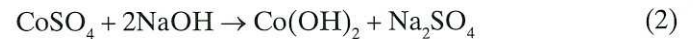
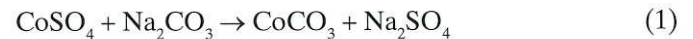
methods for converting the maximum amount of cobalt into the leachate. Table 2 shows the chemical composition of obtained leachate at pH = 0.28.

Table 2: Chemical composition of the leachate [µg/ml]

Co	Li	Ni	Cu	Al	Mn	Fe
12680	2625	1167	695	610	1357	422

### 3 Theoretical study of precipitation

Cobalt precipitation from sulfate medium with selected precipitants ( $\text{NaOH}$ ,  $\text{Na}_2\text{CO}_3$ ,  $\text{H}_2\text{C}_2\text{O}_4 \cdot 2\text{H}_2\text{O}$ ) should be performed according to following reactions:



Fraction diagrams (Figures 5 to 7), constructed by software Hydra Medusa [21], show the behaviour of cobalt and other accompanying metals (Mn and Ni) in the sulfate medium in the presence of chosen precipitating agents. Real concentration of metals in the leachate and the concentration of precipitants in 1.5× stoichiometric excess were used.

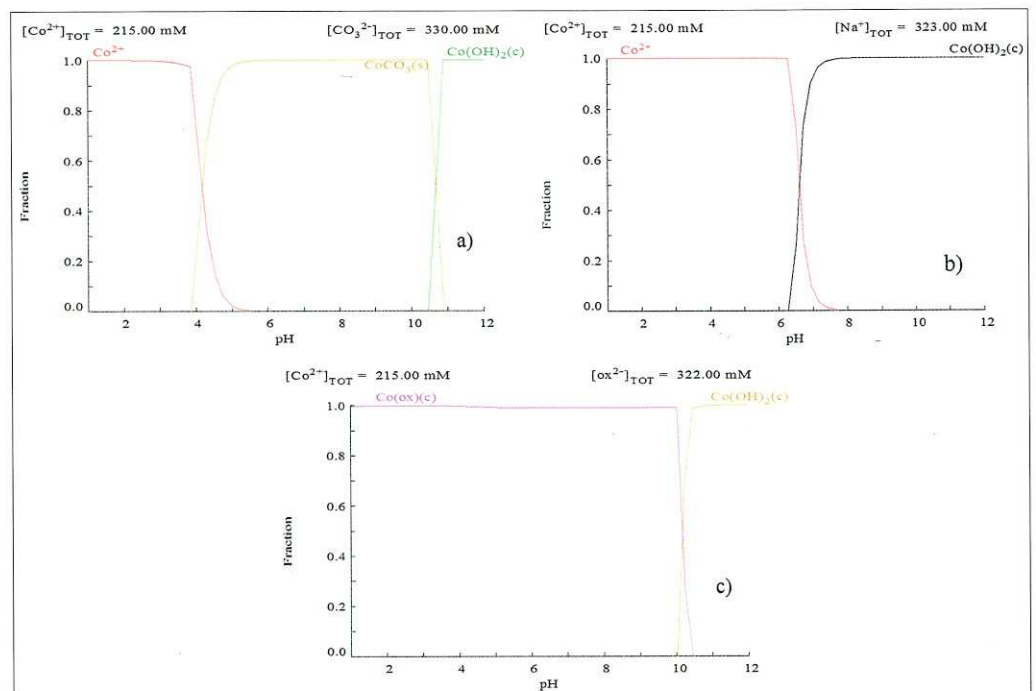


Fig. 5: Fraction diagrams of Co in the presence of a)  $\text{Na}_2\text{CO}_3$ , b)  $\text{NaOH}$ , c) oxalic acid in 1.5× stoichiometric excess

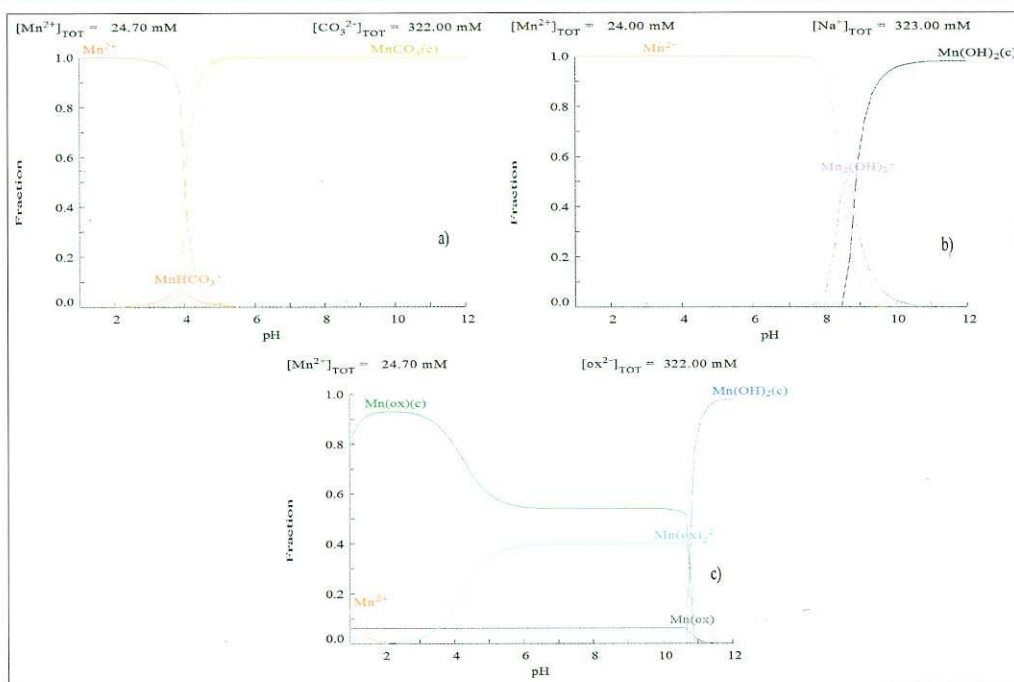


Fig. 6: Fraction diagrams of Mn in the presence of a)  $\text{Na}_2\text{CO}_3$ , b)  $\text{NaOH}$ , c) oxalic acid in 1.5 $\times$  stoichiometric excess

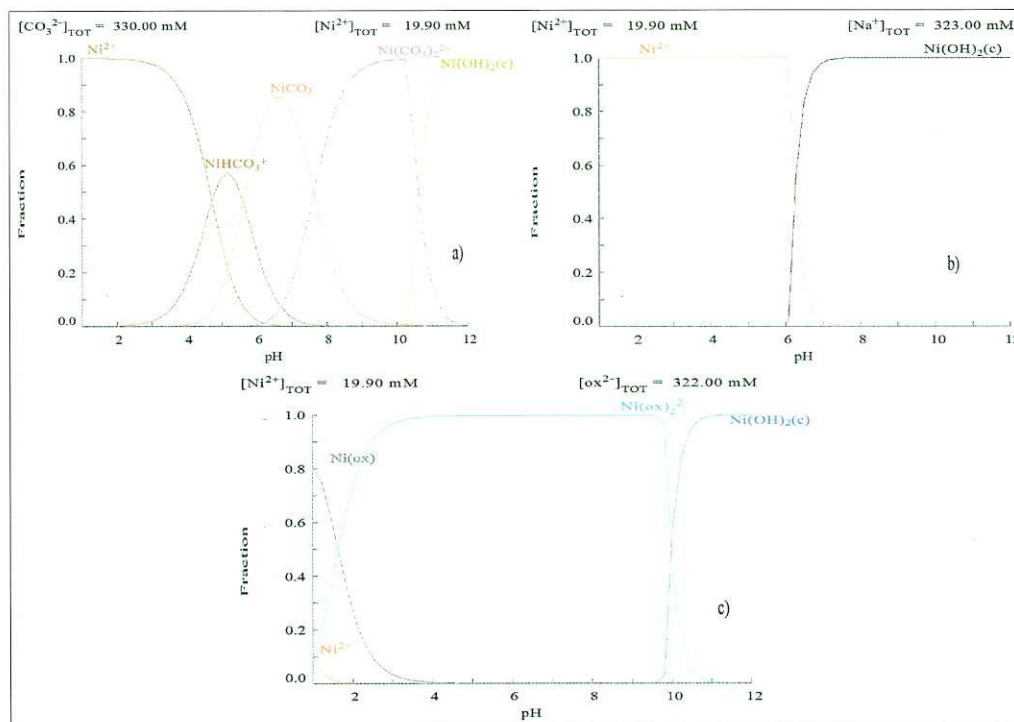


Fig. 7: Fraction diagrams of Ni in the presence of a)  $\text{Na}_2\text{CO}_3$ , b)  $\text{NaOH}$ , c) oxalic acid in 1.5 $\times$  stoichiometric excess

As it can be seen in Figure 5, by using  $\text{Na}_2\text{CO}_3$ , a cobalt carbonate should be formed from  $\text{pH} = 4$ . In case of  $\text{NaOH}$ ,  $\text{Co}(\text{OH})_2$  formation starts at  $\text{pH} = 6$ . Oxalic acid acts as precipitating agents in acidic area. Theoretically, 100 % of cobalt in this concentration can be converted to oxalate in the acidic area.

Fraction diagrams for other metals indicate their co-precipitation with Co as a function of pH (except of using oxalic acid). Figure 6 shows that Mn co-precipitation can be expected from  $\text{pH} = 4$  by using  $\text{Na}_2\text{CO}_3$  and from  $\text{pH} = 8$  by using  $\text{NaOH}$ . Figure 7 shows that Ni co-precipitates by using  $\text{Na}_2\text{CO}_3$  in the narrower range of pH and theoretically selective precipitation could be possible. By using  $\text{NaOH}$ ,

Ni is precipitated from  $\text{pH} = 6$ , similarly to Co. By using oxalic acid, Ni precipitates within acidic pH range but it can also be present in ionic form at  $\text{pH} = 2$ . Lithium should not be co-precipitated, but it should exist in ionic form in all used precipitants.

#### 4 Preliminary experiments

Preliminary experiments were carried out in standard laboratory apparatus. Theoretical study indicates significant effect of pH on Co precipitation and the co-precipitation of other metals by using  $\text{NaOH}$  and  $\text{Na}_2\text{CO}_3$  as precipitation agents. For that reason, the effect of pH on precipitation efficiency by using  $\text{NaOH}$  and  $\text{Na}_2\text{CO}_3$  at  $\text{pH} = 6, 7.5, 8.5$

was experimentally verified. Using oxalic acid, the effect of pH was not monitored and the pH ranged from 0.28 to 0.5 during experiments.

Preliminary experiments were performed under following conditions: 1.5× stoichiometric excess of precipitation agent, 20 °C, 300 rpm, 30 min. After precipitation, the solution was filtered; the precipitate was washed with distilled water and dried at 105 °C to constant weight. Analysis of obtained precipitates was carried out. The efficiency of precipitation  $\eta$  was calculated by Equation (4)

$$\eta = \frac{(c_0 \cdot V_0 - c_1 \cdot V_1)}{c_0 \cdot V_0} \cdot 100 \% \quad (4)$$

where  $c_0$  = input metal concentration in the leachate,  $V_0$  = input volume,  $c_1$  = output metal concentration in the leachate,  $V_1$  = output volume.

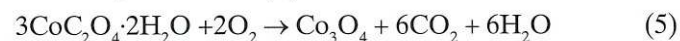
Based on the results, the most suitable precipitating agent was selected. The selected agent was used for further experiments in order to determine the effect of concentration, temperature and time on the process efficiency.

#### 4.1 Oxalic acid as a precipitating agent – influence of temperature and concentration on cobalt precipitation efficiency

An advantage of the using oxalic acid as a precipitating agent for Co precipitation is the possibility of precipitation in the acidic pH range, i.e. without pH adjustment, so that the leachate is not contaminated with added metal ions, e.g.  $\text{Na}^+$ . Following conditions and their influence on the process efficiency were investigated: oxalic acid in 1.5, 2.0, 2.5 and 3.0× stoichiometric excess and temperature (20, 40, 60, 80 °C). Experiments were running for 1 hour at 500 rpm. A determination of time dependence was performed by 4-hours experiments under following conditions: oxalic acid in 1.5 and 3.0× stoichiometric excess, 80 °C, 500 rpm.

#### 4.2 Calcination of the precipitate

Prior the calcination, all obtained precipitates were mixed and homogenized. The main purpose of the calcination was conversion of cobalt oxalate to the oxidic form ( $\text{Co}_3\text{O}_4$ ), according to reaction (5):



Calcination was carried out at 400, 600 and 800 °C for 4 hours in laboratory electric furnace.

## 5 Results and discussion

### 5.1 Preliminary experiments

Co precipitation efficiency from leachate by using  $\text{Na}_2\text{CO}_3$  and NaOH at pH = 6, 7.5 and 8.5 and by oxalic acid is shown in Figure 8. The precipitation efficiency of accompanying metals under the same conditions is given in Table 3. Chemical composition of obtained precipitates is given in Table 4.

The maximum cobalt precipitation efficiency (100 %) can be achieved at pH = 8.5 by using  $\text{Na}_2\text{CO}_3$  and NaOH. By oxalic acid, 80 % efficiency of Co precipitation was achieved. According to the fraction diagrams (see Figure

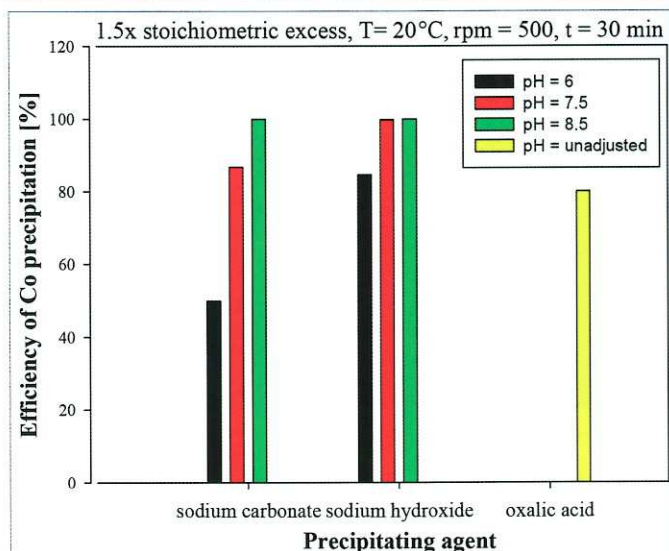


Fig. 8: Co precipitation efficiency – effect of precipitating agent and pH

Table 3: Precipitation efficiency  $\eta$  of accompanying metals [%]

Precipitating agent, pH	Li	Mn	Ni
$\text{Na}_2\text{CO}_3$ , pH = 6	27.31	63.06	69.38
NaOH, pH = 6	31.11	64.88	91.93
$\text{Na}_2\text{CO}_3$ , pH = 7.5	60.92	83.42	90.24
NaOH, pH = 7.5	66.70	97.85	99.81
$\text{Na}_2\text{CO}_3$ , pH = 8.5	76.89	99.98	99.93
NaOH, pH = 8.5	71.05	99.81	99.90
$\text{C}_2\text{H}_2\text{O}_4 \cdot 2\text{H}_2\text{O}$ , pH = 0.5	24.13	43.44	73.29

Table 4: Metal content in precipitates [%]

Precipitating agent, pH	Co	Li	Mn	Ni	Na
$\text{Na}_2\text{CO}_3$ , pH = 6	20.35	0.12	0.53	2.65	5.94
NaOH, pH = 6	24.60	0.14	0.78	2.25	4.41
$\text{Na}_2\text{CO}_3$ , pH = 7.5	15.96	0.36	0.96	1.36	9.26
NaOH, pH = 7.5	14.94	0.36	1.17	1.02	10.38
$\text{Na}_2\text{CO}_3$ , pH = 8.5	18.26	0.30	1.98	1.36	10.6
NaOH, pH = 8.5	12.40	0.42	1.18	0.87	11.06
$\text{C}_2\text{H}_2\text{O}_4 \cdot 2\text{H}_2\text{O}$ , pH = 0.5	24.3	0.04	0.40	1.28	-

5a), pH = 6 should be appropriate for Co precipitation by  $\text{Na}_2\text{CO}_3$ , but only about 50 % Co was precipitated at this pH. On the contrary, by NaOH Co precipitation should only begin from pH = 6, but at this pH nearly 90 % of Co was precipitated, as is shown in Figure 8.

In addition to Co precipitation, the efficiency of co-precipitation of the accompanying metals – Li, Mn and Ni was investigated. Lithium should not be precipitated in a given concentration according to the fraction diagrams in either case, but almost 80 % of Li was co-precipitated at pH = 8.5 by NaOH and  $\text{Na}_2\text{CO}_3$  as well. By oxalic acid, approximately 25 % of Li was co-precipitated. The efficiency of Mn and Ni precipitation was in  $\text{Na}_2\text{CO}_3$  65 % and 70 % even at the lowest pH = 6. Study has confirmed that Mn and Ni cannot be maintained in solution without co-precipitation when cobalt is precipitated by  $\text{Na}_2\text{CO}_3$ . By NaOH, 65 % of Mn and 90 % of Ni were already precipitated at pH = 6. However, theoretically, Mn precipitation by NaOH should start

at pH = 8.5 and Ni precipitation from pH > 6. At higher pH values (7.5 and 8.5), up to 100 % of these metals were co-precipitated by both precipitating agents. It has been found, that in the presence of Mn and Ni it is not possible to selectively precipitate Co from the sulfate leachate neither by NaOH nor Na<sub>2</sub>CO<sub>3</sub> at studied concentrations of these metals (about 1.17 to 1.35 g/l).

The low co-precipitation efficiency of the accompanying metals was observed when oxalic acid was used as the precipitating agent. In case of NaOH and Na<sub>2</sub>CO<sub>3</sub>, higher pH values resulted in higher concentrations of the accompanying metals in the precipitates. In addition, Table 4 shows that at pH = 7.5 to 8.5 formed precipitates contained up to 9 to 11 % of Na. For this reason, oxalic acid was labelled as the most suitable precipitating agent for cobalt precipitation when the Co content in precipitate was relatively high (24.30 %) with low content of lithium (0.04 %), manganese (0.40 %) and nickel (1.28 %).

## 5.2 Co-precipitation by oxalic acid – effect of temperature and concentration

Table 5 shows the efficiency of cobalt precipitation by using oxalic acid with 2.5 and 3× stoichiometric excess at 20 to 80 °C together with Co content in the precipitates. Figure 9 shows the time dependency of Co precipitation at 60 and 80 °C with 1.5 and 3.0× stoichiometric excess of oxalic acid.

As it can be seen in Table 5, temperature and concentration of oxalic acid have significant effect on the Co precipi-

Table 5: Results of Co precipitation by oxalic acid

Temperature [°C]	Stoichiometric excess of oxalic acid	Precipitation efficiency [%]	Co content in precipitate [%]
20	2.5×	19.21	25.8
20	3.0×	31.00	26.45
40	2.5×	29.21	25.40
40	3.0×	41.13	26.35
60	2.5×	53.99	25.50
60	3.0×	72.39	26.10
80	2.5×	57.68	22.90
80	3.0×	78.69	26.55

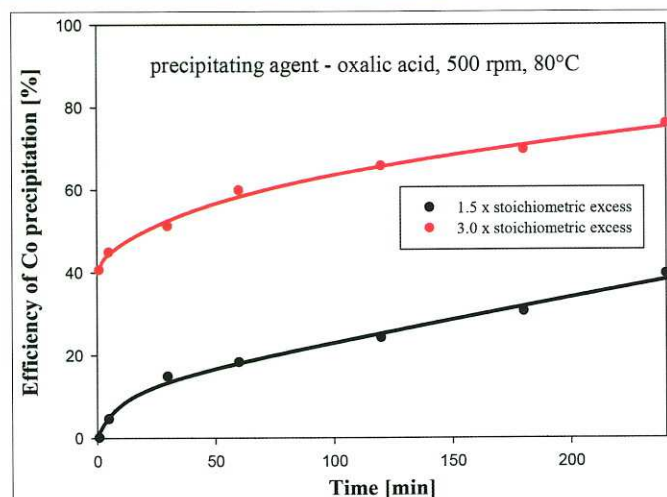


Fig. 9: Time dependency of Co precipitation

tation, with maximum precipitation efficiency (78 %) at 80 °C using 3.0× stoichiometric excess of oxalic acid. Under these conditions, the cobalt content in the precipitate was 26.55 %, which theoretically corresponds to the 82.4 % cobalt oxalate content.

The time of precipitation positively influences the efficiency of the process and further increasing of Co precipitation by extending of precipitation time can be expected.

## 5.3 Analysis of obtained precipitate

The chemical composition of the precipitate before calcination is shown in Table 6. Its XRD pattern is shown in Figure 10 and results from its SEM-EDX analysis are shown in Figure 11.

The precipitate contained 25.17 % of Co as C<sub>2</sub>CoO<sub>4</sub>·2H<sub>2</sub>O. The presence of two moles of water in the cobalt oxalate compound was also confirmed by DTA analysis. It was found from XRD pattern and AAS analysis that copper is present in precipitate as C<sub>2</sub>CuO<sub>4</sub> in relatively high con-

Tab. 6: Chemical composition of precipitate [%]

Co	Li	Mn	Ni	Cu	Fe	Al
25.17	0.02	0.32	1.46	3.48	0.29	0.086

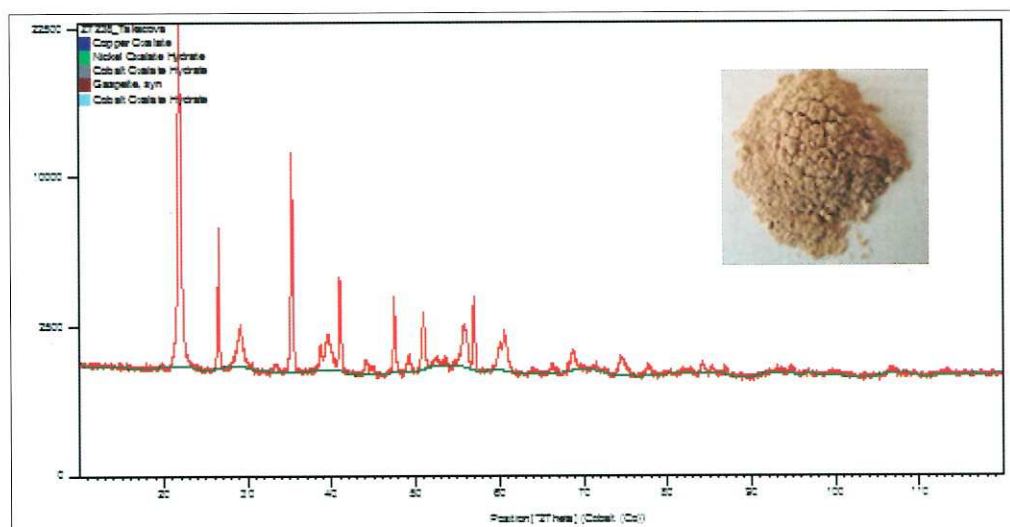


Fig. 10: XRD pattern of precipitate intended for calcination



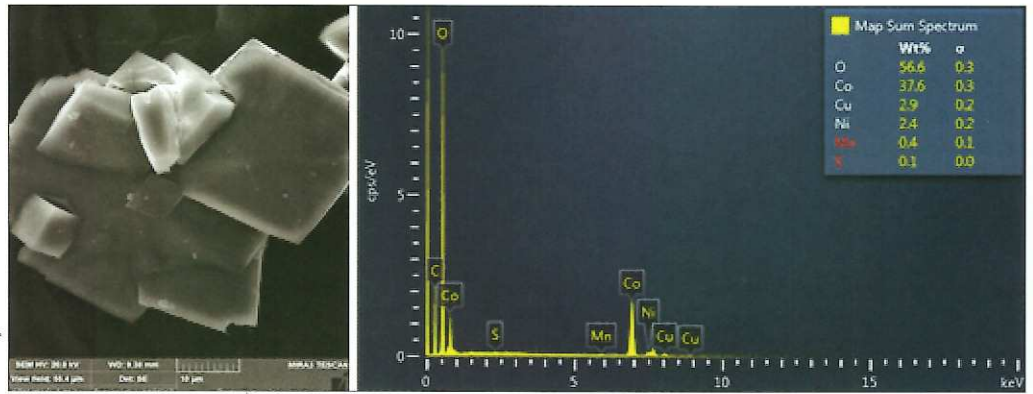


Fig. 11: Results from SEM-EDX analysis of precipitate

tent (3.48 %). The second impurity was nickel (1.46 %) as  $C_2NiO_4$ . Copper and nickel were also determined by SEM-EDX analysis (2.4 to 2.9 %).

### 5.3 Calcination of the precipitate

The effect of calcination temperature on weight loss of precipitate together with the expression of cobalt content after calcination is shown in Figure 12. Table 7 shows the chemical composition of obtained calcinates.

XRD pattern of calcinate coming from calcination at 600 °C and at 800 °C is shown in Figures 13 and 14. The

Tab. 7: Chemical composition of calcinates [%]

Calcination temperature [°C]	Co	Li	Mn	Ni	Cu	Fe	Al
400	56.25	0.04	0.65	3.425	8.59	0.20	0.20
600	57.35	0.04	0.68	2.970	7.49	0.21	0.20
800	54.05	0.04	0.62	3.170	7.77	0.18	0.18

SEM-EDX analysis of the calcinate coming from calcination at 800 °C is shown in Figure 15.

During calcination of the precipitate at 400 to 800 °C, the highest weight loss ca. 71 wt.-% was achieved at 800 °C. According to the results from AAS, content of cobalt and other accompanying metals has doubled. After calcination, a change in the phase composition of the precipitate was expected by removing volatile precipitate components such as water and  $CO_2$ . The Co content in calcinate ranged from 54 to 57 %. After calcination at 600 °C (see Figure 14), new cobalt phases such as  $Co_3O_4$ ,  $Cu_{0.72}Co_{2.28}O_4$  and other oxidic phases of the accompanying metals, especially copper, were identified. Copper content in the calcinate has increased to 7.5 to 8.6 % and the nickel content to 2.9 to 3.4 % depending on the used temperature. A relatively high copper and nickel content was also confirmed by SEM-EDX analysis (7.1 % Cu and 3.7 % Ni). In the calcinate coming from calcination at 800 °C (see Figure 15), CoO was also identified. CoO formation is possible, according to [22], at temperatures below 200 °C and above 900 °C. That

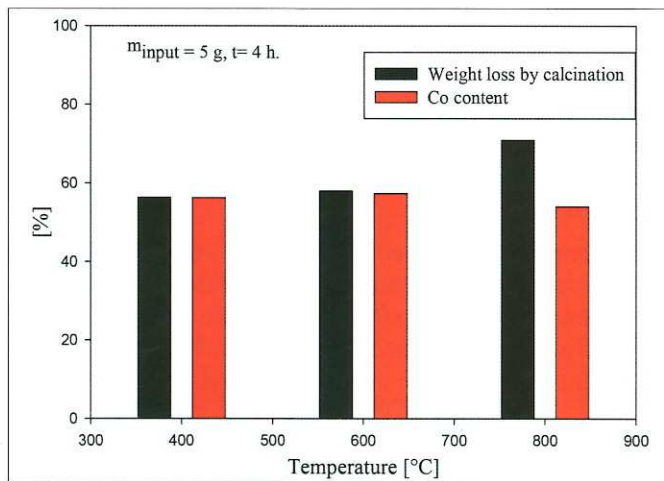


Fig. 12: Effect of calcination temperature on weight loss of precipitate

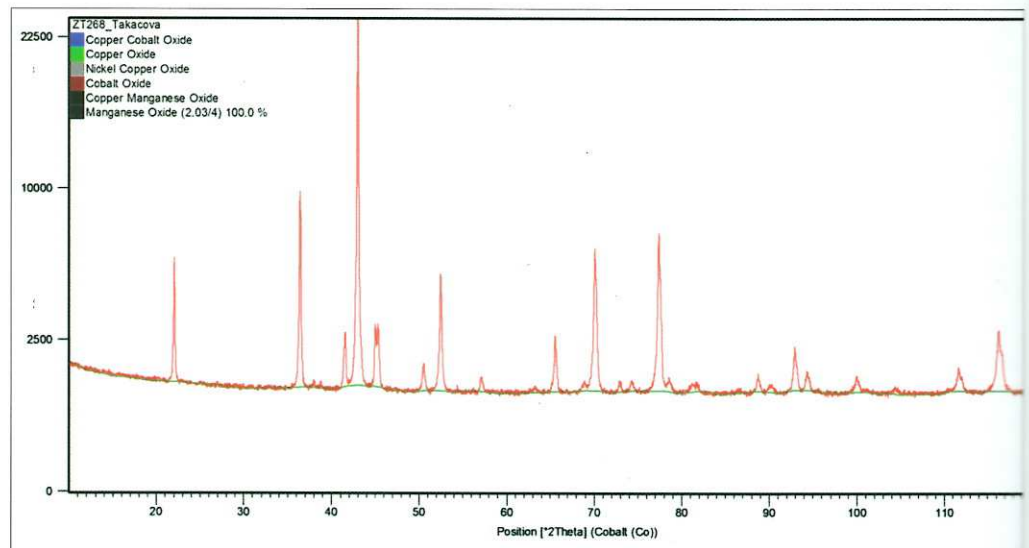


Fig. 13: XRD pattern of calcinate from calcination at 600 °C

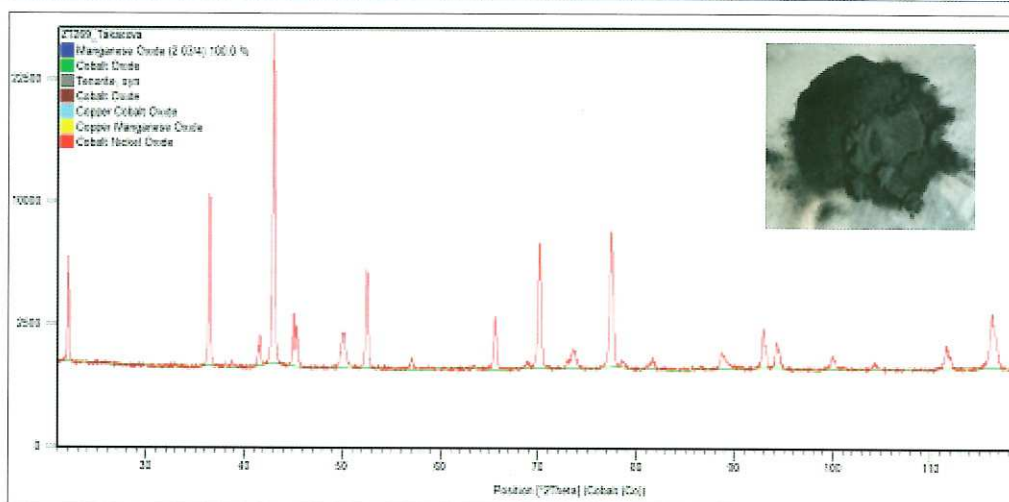


Fig. 14:  
XRD pattern of calcinate from calcination at 800 °C

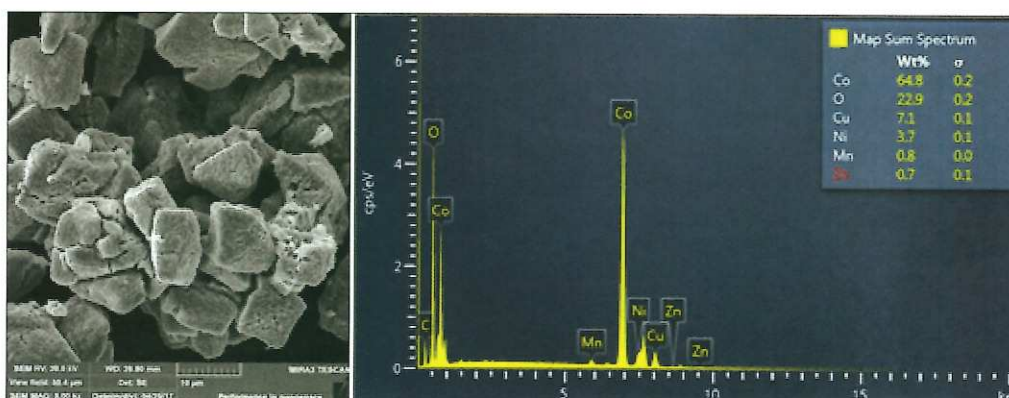


Fig. 15:  
Results of SEM-EDX analysis of calcinate from calcination at 800 °C

author [22] also reports that commercially produced  $\text{Co}_3\text{O}_4$  can contain up to 20 % of  $\text{CoO}$ .

The resulting product from calcination was Co in oxidic form, mainly as  $\text{Co}_3\text{O}_4$  with purity of 73.6 to 78.11 %, with the addition of other phases. In order to obtain a product with higher purity, it would be appropriate to remove copper and nickel from leachate before Co precipitation, e.g. by solvent extraction.

## 6 Conclusion

Following statements can be concluded from this study:

- A theoretical study indicated possibility of selective Co precipitation by selected precipitating agents and suitably chosen pH.
- Preliminary experiments found that most of the Mn and Ni and 80 % of Li were co-precipitated with 90 to 100 % of Co at pH = 7.5 to 8.5 by using  $\text{Na}_2\text{CO}_3$  and NaOH despite the theoretical assumption.
- In terms of low co-precipitation of the accompanying metals, oxalic acid can be considered the most suitable precipitating agent.
- High excess of oxalic acid is necessary for achieving high precipitation efficiency – with 3× stoichiometric excess maximally 80 % of Co was precipitated at 80 °C during 1 hour.
- It has also been confirmed that precipitation of cobalt in oxalic acid significantly depends on the temperature.

- Extending time of the precipitation to 4 hours increases Co precipitation efficiency constantly, but it is necessary to assess the economy of this process.
- A relatively high content of copper and nickel was identified in the obtained precipitate in the form of oxalates.
- By calcination,  $\text{Co}_3\text{O}_4$  can be obtained, but at the higher temperature (800 °C)  $\text{CoO}$  was identified as well. Resulting purity of the product obtained after calcination ( $\text{Co}_3\text{O}_4$ ) was 78 %.
- Suggestions for further study – solvent extraction of accompanying metals before Co precipitation in order to achieve high purity of obtained product.

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