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Crystallization of $Li_2SO_4{\cdot}H_2O$ salt from an aqueous solution obtained by solvent extraction of lithium ions using the new extractant Mextral 3938HDC

Agnieszka Sobianowska-Turek [1,2], Michał Zygmunt [2], Agnieszka Fornalczyk [2,3], Tang Qiming [4]

[1] Department of Water and Wastewater Management and Waste Technology, Wrocław University of Science and Technology, Wrocław, Poland

[2] Elemental Strategic Metals Sp. z o.o, Grodzisk Mazowiecki, Poland

[3] Department of Production Engineering, Silesian University of Technology, Katowice, Poland

[4] Kopperchem Institute of Technology, Changshou District, Chongqing, China

Corresponding author: agnieszka.fornalczy@polsl.pl (Agnieszka Fornalczyk)

Abstract: The implementation of innovative industrial processes for the recovery of critical metals from solutions originating from the processing of both primary raw materials (ores) and secondary raw materials (waste) is one of the most important challenges facing the developing world. Production of battery grade purity materials such as lithium salts, which are used for the production of new lithium-ion cells is the key to the sustainable development of hybrid and electric cars, submarine drives or spacecraft. This article presents preliminary results of research on the solvent extraction of lithium from a solution of lithium(I) carbonate(IV) using the extractant called Mextral 3938HDC from Kopper Chemical Industry Corp., Ltd. It has been found that the efficiency of lithium ion extraction from the lithium carbonate solution is 68.9%.

Keywords: solvent extraction, lithium, Mextral 3938HDC

1. Introduction

Nowadays, lithium has become a key element for the further dynamic development of global electrification in the automotive industry (Domingues and de Souza, 2024; Yang et al., 2024; Peng et al., 2024). The analysis of market data shows that in the years 2020–2050 the demand for lithium used for the production of NMC (lithium-nickel-cobalt-manganese) cells may increase even 18–20 times (Xu et al., 2020), which will directly translate into the need to recover this element in various chemical forms from polymetallic waste, using different, often previously unused chemical metallurgy methods (Siekierka et al., 2022; Ochromowicz et al., 2024; Leszczyńska-Sejda et al., 2024a). The following unit processes can be used for lithium recovery: membrane methods (Yu et al., 2024), flotation methods (Feng et al., 2025), adsorption methods (Li et al., 2025) and solvent extraction (Kanagasundaram et al., 2024). The development of the latter is a consequence of the industrial scale implementation by Aqua Metals, Inc. (2024), Botree Tech. (2024), Li-Cycle (2024), Brunp Recycling (2024), GEM Co., Ltd. (2024) or Ganpower Technology Co., Ltd. (2024) the lithium cell recycling technology based on solvent extraction of copper, nickel, cobalt and manganese from solutions obtained after acidic single-stage and/or multi-stage reductive leaching of battery masses originating from mechanical processing of waste lithium-ion cells (Leszczyńska-Sejda et al., 2024b; Willner et al., 2024). In the latter processes the most commonly obtained products are the following sulphate salts: $NiSO_4{\cdot}6H_2O$, $CoSO_4{\cdot}7H_2O$ and $MnSO_4{\cdot}H_2O$. Until the end of last year, there were no organic solvents available on the market dedicated to direct solvent extraction of lithium from aqueous solutions. In 2024 two companies: Solvay (2024a) and Kopper Chemical Industry Corp., Ltd. (2024a) presented in their commercial offer, respectively, CYANEX®936P (Solvay, 2024b) and Mextral 3938HDC (Kopper Chemical Industry Corp. 2024b) extractants. According

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to the manufacturers, these substances form complexes with lithium in a wide range of Li+ ion concentrations in aqueous solution according to the reaction:

$$Li^+_{(H_2O)} + RH_{(org)} = RLi_{(org)} + H^+_{(H_2O)} \tag{1}$$

with better separation of Li^+ ions from Na^+ from aqueous alkaline solutions, free from divalent Ca^{2+} and Mg^{2+} ions (Cohen et al., 2022).

The application of a single solvent extraction process of lithium from aqueous solutions requires in earlier stages recovery of valuable metals such as nickel, cobalt and manganese. Application of the similar methods of metals recovery might be beneficial in the industrial scale recycling of used and/or waste Li-ion cells of various types and kinds. Since in most batteries technologies lithium element is used in the form of carbonate salts - Li_2CO_3, this salt is obtained from lithium-ion battery masses in the process of co-precipitation from aqueous solutions using sodium(I) carbonate(IV) followed by multi-stage purification of the product until a substance of high purity is obtained (Battaglia et al., 2022). Implementation of a comprehensive technology for the recovery of all payable metals using one method may allow the recycler to generate savings both in terms of machinery and the acquisition of qualified staff. This article presents preliminary results of solvent extraction of lithium from a synthetic solution of lithium(I) carbonate(IV) using Mextral 3938HDC from Kopper Chemical Industry Corp., Ltd. Followed by crystallization of $Li_2SO_4 \cdot H_2O$ salt. Kopper Chemical Industry Corp., Ltd. is a manufacturer of reagents and chemical materials used in broadly understood chemical metallurgy for the recovery of critical metals from solutions by flotation and/or solvent extraction methods (Kopper Chemical Industry Corp., Ltd. 2024a). Table 1 presents the most important results of studies conducted using Mextral-type organic compounds for solvent extraction of copper, cobalt, nickel, lithium from various solutions and/or industrial wastewater.

Table 1. Summary of solvent extraction test results using Mextral-type chemicals

Extractant	Extracted metal	Research solution	Highlights	Literature reference
Mextral 272P Mextral 5640H	Co, Ni	Acidic solution leaching of Li-ion battery masses	- A combined hydrometallurgical process was proposed. - Fe, Mn, Ni and Li were selectively precipitated and recovered. - Cu was selectively extracted and recovered by Mextral®5640H. - Nickel loaded Mextral®272P was employed to recover Co. - All metals can be effectively separated and recovered.	Chen et al., 2015
Mextral EOL	Li	Wastewater from the boron industry	- A synergistic solvent extraction (SSX) system was implemented for extracting lithium. - Statistical modelling was used to obtain regression models. - Optimization and validation results were obtained. - Stripping studies were conducted with water and HCl acid solutions. - - The SSX system has been proven to be an effective method for lithium extraction.	Çelebia et al., 2019
Mextral 84H Mextral 54-100	Cu	Alkaline solution from PCB waste leaching	- Three options, i.e. hydrazine reduction, sulphide precipitation, and solvent extraction, are evaluated. - >95% Cu could be recovered as Cu_2O or native Cu at different Cu/hydrazine molar ratios. - >99% Cu was recovered as CuS by sulphide within 5 min. - Pb, Sn and Zn were co-precipitated considerably (>65%), making CuS purity reduced. - Two extractants, Mextral 84H and Mextral 54–100, were compared during solvent extraction.	Li et al., 2021

mixture: Versatic 10 Mextral 984H Mextral DT100	Cu, Co, Ni, Cd	Synthetic wastewater from the metallurgical industry	- A carboxylic acid synergistic system was proposed. - It was determined that the utilization of Versatic 10 or Mextral 984H alone cannot separate desirable metals from undesirable ones. - The extraction order of metals with a mixture of Versatic 10 acid and Mextral 984H in Mextral DT100 was Cu ≫ Ni > Co > Cd ~ Zn ≫ Ca > Mg. - - The Versatic 10 acid/Mextral 984H system produced significant synergistic shifts for nickel, cobalt, zinc, and cadmium, but antagonistic shifts for magnesium and calcium.	Qiu et al., 2017
mixture: Versatic 10 Mextral 84H P204	Co	Synthetic cobalt solution	- Versatic 10 has negligible effect on cobalt extraction and the poisoning of Mextral 84H by cobalt. - P204 significantly inhibits the poisoning of Mextral 84H by cobalt. - The mechanism of poisoning Mextral 84H by cobalt based on coordination structure and hybridization was proposed. - - The reasons for inhibition of the poisoning Mextral 84H by cobalt with P204 were analyzed.	Sun et al., 2018
Mextral 84H	Ni	Synthetic nickel solution	- P204 produced a significant synergistic effect on nickel while Versatic 10 didn't. - The formation of NiL_2 and $Ni(H_2A_2L_2)$ in different organic systems were proposed. - The structures of nickel extracts were proposed. - - The reasons for the synergistic effect on nickel were discussed.	Sun et al., 2019
Mextral 84H Mextral 54-100	Cu	Synthetic copper solutions	- The recovery of Cu from alkaline glycinate leach solutions is demonstrated. - Mextral 54–100 (1-benzoyl-2-nonyl ketone) was used as one solvent extractant. - Mextral® 84H (2-hydroxy-5-nonylacetophenone oxime) was used as another extractant. - Isotherms are presented for the extraction of Cu from alkaline glycinate solutions. - - Isotherms are presented for the stripping of Cu from the extractant using acidic electrolyte.	Tanda et al., 2017
Mextral 6103H	Co	Solution after leaching Cu-Co ores	- Synergistic solvent extraction was employed for the separation of metal ions. - Co(II) was selectively extracted by Mextral 6103H-naphthenic acid-isooctanol system. - Mextral 6103H had an evident synergistic effect on naphthenic acid. - A process was proposed to recover cobalt form the pre-treated leaching solution of copper-cobalt ores. - - The purity of Co(II) in the stripping solution was higher than 99.8%.	Xu et al., 2021
mixture: Versatic 10, Mextral 6103H Aliquat 336	Co, Ni	Nickel laterite leach solutions	- New synergistic system containing Versatic 10 acid/Mextral 6103H/Aliquat 336 was proposed. - A synergistic extraction system was employed for the selective separation of Ni and Co from other metals. - Aliquat 336 could significantly improve the nickel stripping kinetics. - - 99% nickel and 97% cobalt were extracted via a two-stage semi-continuous extraction.	Xu et al., 2022a

Mextral 6103H Mextral DT100	Co, Ni	Synthetic laterite leach solution	- Synergistic system containing Mextral 6103H/naphthenic acid/TOPO was proposed. - A synergistic system was employed for the separation of Ni and Co from other metals. - TOPO could significantly improve the nickel stripping kinetics. - - 97% nickel and 99% cobalt were extracted via a two-stage of extraction.	Xu et al., 2022b

2. Materials and methods

2.1. Research materials

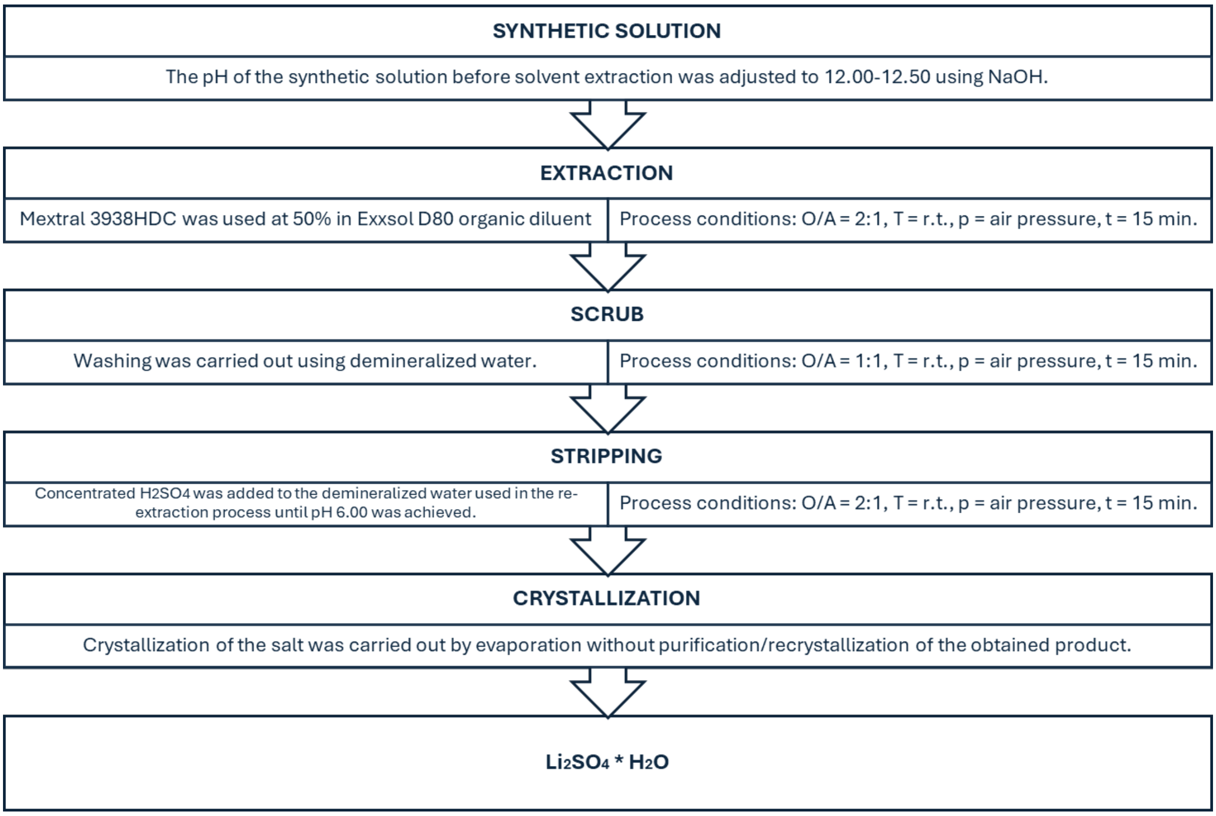
A 2.0 dm^3 aqueous solution of lithium(I) carbonate(IV) with a pH of about 11, prepared by the R&D Team of Elemental Strategics Metals Sp. z o.o., was used in the study. The solution was prepared using a a minimum content of 99.0% Li_2CO_3 from Chempur (2024a). To test the pH of the solutions at each stage of solvent extraction, a pH meter from Elmetron, Series 410 was used. Corrections of the pH of the tested solution to a value between 12.00 and 12.50 necessary for efficient solvent extraction were made using a 30% aqueous solution of sodium(I) hydroxide with a minimum content of 99.0% NaOH from Chempur (2024b). The final pH of the tested solution was 12.31, and no precipitation or turbidity of the solution was observed during pH correction. The contents of the cations in the tested solution were: 1.41 g/dm^3 Li^+ and 3.01 g/dm^3 Na^+. Solvent extraction studies were performed using commercial extractant Mextral 3938HDC purchased directly from Kopper Chemical Industry Corp., Ltd. The organic diluent used in the studies was Exxsol D80 from Exxon Mobil (2024). The re-extraction process was carried out using aqueous solutions prepared from a chemical reagent with a minimum content of 98.0% H_2SO_4 from Chempur (2024c). The prepared Li_2CO_3 solution was a representation of the real solution obtained by acidic reductive leaching of the battery mass from waste lithium-ion cells, from which valuable metals Cu, Ni, Co, Mn were first recovered and which was subjected to purification from interfering ions before a single operation of Li solvent extraction. The aqueous phase in the washing processes was demineralized water. All experimental work on a laboratory scale, including quantitative and qualitative analyses of solutions and solid samples, was commissioned and performed in accordance with the research concept and under the substantive supervision of the R&D Team of Elemental Strategic Metals Sp. z o.o. in the Łukasiewicz Research Network - Institute of Non-Ferrous Metals in Gliwice (2024), which has the necessary research infrastructure.

2.2. Research methodology

Solvent extraction studies of lithium from the synthetic solution were carried out using the Mextral 3938HDC extractant according to Fig. 1 below. The process was carried out for an alkaline solution (pH 12.00 - 12.50), with the organic to inorganic phase ratio (O/A) of 2:1 (extraction, stripping) and 1:1 (scrub), normal pressure, room temperature (r.t.) and time of 15 minutes for individual solvent extraction units.

3. Results and discussion

For the solvent extraction tests, 1.0 dm^3 of the following organic solution was prepared: 50% v/v Mextral3938HDC, 50% v/v Exxsol D80. 0.5 dm^3 of the test solution was introduced into a 2.0 dm^3 beaker equipped with a mechanical stirrer with an anchor rod, followed by 1.0 dm^3 of the previously prepared organic phase. After 15 minutes of mixing, both phases were transferred to a 2.0 dm^3 separator. The phases separated quickly for up to 2 minutes. After phase separation, 0.5 dm^3 of aqueous solution (raffinate after Li extraction) and 1.0 dm^3 of organic solution (Li extract) were obtained. Additionally, the pH of the aqueous phase after extraction was measured, which was 11.58. In the next stage, the organic phase after lithium extraction was directed to the washing stage using a phase ratio of O/A – 1:1 and using demineralized water. 1.0 dm^3 of demineralized water was introduced into a beaker with a volume of 2.0 dm^3, equipped with a mechanical stirrer with an anchor rod, and then 1.0 dm^3 of the


SYNTHETIC SOLUTION

The pH of the synthetic solution before solvent extraction was adjusted to 12.00-12.50 using NaOH.

EXTRACTION

Mextral 3938HDC was used at 50% in Exxsol D80 organic diluent

Process conditions: O/A = 2:1, T = r.t., p = air pressure, t = 15 min.

SCRUB

Washing was carried out using demineralized water.

Process conditions: O/A = 1:1, T = r.t., p = air pressure, t = 15 min.

STRIPPING

Concentrated H2SO4 was added to the demineralized water used in the re-extraction process until pH 6.00 was achieved.

Process conditions: O/A = 2:1, T = r.t., p = air pressure, t = 15 min.

CRYSTALLIZATION

Crystallization of the salt was carried out by evaporation without purification/recrystallization of the obtained product.

$Li_2SO_4 * H_2O$

Fig. 1. Scheme of laboratory scale production of $Li_2SO_4{\cdot}H_2O$

organic phase (Li extract) obtained after lithium extraction. After 15 minutes of mixing, the phases were transferred to a separatory funnel with a volume of 2.0 dm^3. The phases separated relatively slowly for about 10 minutes. After phase separation, 1.0 dm^3 of an aqueous solution with a pH of 12.08 and 1.0 dm^3 of an organic solution were obtained. The collected organic phase was directed to the Li re-extraction stage. A phase ratio of O/A – 2:1 was used. In a 2.0 dm^3 beaker equipped with a mechanicalstirrer with an anchor rod, 0.5 dm^3 of demineralized water was added, followed by 1.0 dm^3 of washed Li extract. During mixing, the pH of the solution was lowered using a concentrated solution of sulfuric acid (VI). After adding just a few drops of H_2SO_4 solution, the pH of the aqueous phase decreased from above 11.00 to 5.50. After 15 minutes of mixing, the phases were transferred to a 2.0 dm^3 separator. The phases separated for up to 3 minutes. After phase separation, 0.5 dm^3 of aqueous solution and 1.0 dm^3 of organic solution were obtained. After each stage of the solvent extraction process shown in Fig. 2, 0.1 dm^3 of the obtained aqueous solution was collected and a quantitative and qualitative chemical analysis was performed for the content of lithium and sodium ions using the ICP-OES method, using the Agilent 5800 series

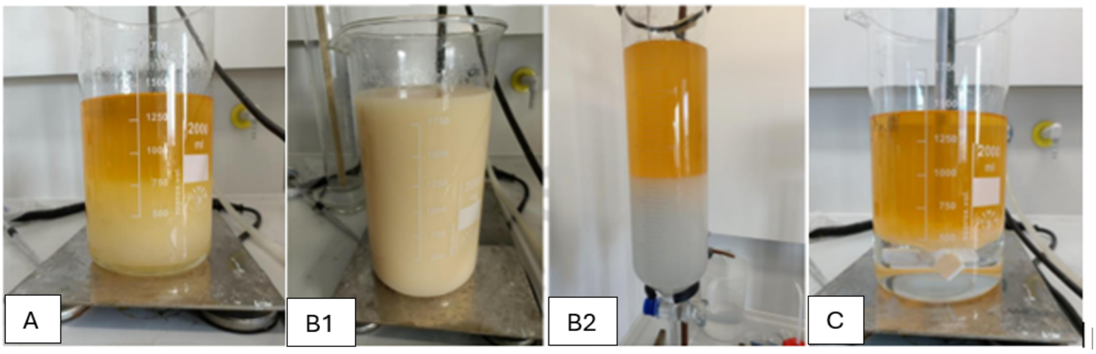


Fig. 2. The course of the lithium solvent extraction process using Mextral 3938HDC extractant: A – view of the phases during the Li extraction process, B1 and B2 – view of the phases during and after the Li extractant washing process, C – view of the phases after the Li re-extraction process

Tables 2 and 3 present the results from all stages of Li and Na extraction along with the balance of the studied process. The degrees of metal solvent extraction (EM, %), leaching after the metal solvent extraction process (WM, %) and the degree of metal re-extraction (RM, %) were calculated using the following formulas:

$$EM=((([M]_{aq0}\cdot V_{aq0})-([M]_{aq1}\cdot V_{aq1}))/([M]_{aq0}-V_{aq0}))\cdot 100\% \tag{2}$$

$$WM=(([M]_{aq2}\cdot V_{aq2})/(([M]_{aq0}\cdot V_{aq0})-([M]_{aq1}\cdot V_{aq1})))\cdot 100\% \tag{3}$$

$$RM=((M_{aq3}\cdot V_{aq3})/(([M]_{aq0})\ \cdot V_{aq0})-([M_{aq1}\cdot V_{aq1})-([M]_{aq2}\cdot V_{aq2})))\cdot 100\% \tag{4}$$

where: EM – metal extraction degree, %; WM – metal leaching degree, %; RM – metal re-extraction degree, %; $[M]_{aq0}$ – metal concentration in the water phase before extraction, g/dm^3; $[M]_{aq1}$ – metal concentration in the water phase after extraction, g/dm^3; $[M]_{aq2}$ – metal concentration in the water phase after leaching, g/dm^3; $[M]_{aq3}$ – metal concentration in the water phase after re-extraction, g/dm^3; V_{aq0} – water phase volume before extraction, dm^3; V_{aq1} – water phase volume after extraction, dm^3; V_{aq2} – water phase volume after leaching, dm^3; V_{aq3} – water phase volume after re-extraction, dm^3.

Table 2. Results of quantitative and qualitative chemical analyzes of the solutions obtained in the test, along with the calculated degrees of extraction, washing and re-extraction of lithium and sodium

Water solution	Volume of aqueous solution, dm^3	Concentration in aqueous solution, g/dm^3		Process degree, %	
		Li	Na	Li	Na
Synthetic solution	0.5	1.41	3.01	-	-
		Extraction			
Raffinate	0.5	0.48	4.07	66	-35
		Wash			
After washing	1.0	0.078	0.067	17	-13
		Re-extraction			
Re-extract	0.5	0.78	0.077	101	-6.0

Table 3. Mass balance of the conducted solvent extraction test of lithium and sodium

Water solution	Mass, g	
	Li	Na
	Entry	
Synthetic solution	0.705	1.505
	Exit	
Raffinate	0.240	2.035
Wash	0.078	0.067
Re-extract	0.390	0.039
Balance sheet compliance, %	100.4	142.2*

* balance > 100% Na extraction is due to the use of sodium hydroxide to correct the pH of the test solution

After Li re-extraction, lithium salt crystallization was performed. For this purpose, 450 cm^3 of the solution obtained after re-extraction was concentrated until dry mass was obtained. 3.7929 g of white, fine-crystalline precipitate was obtained, a photo of which is presented in Fig. 3. The content of lithium and sodium in the precipitate obtained in this way was analysed and it was subjected to XRD analysis (Fig. 3). The obtained results of Li and Na content in the precipitate and the calculated values of Li and Na recovery from the input solution are presented in Table 4.

Table 4. Results of analyses of the obtained lithium salt

Material	Type of material	Element content, %		Recovery from the input solution, %	
		Li	Na	Li	Na
Obtained precipitate	Examined	11.53	0.11	68.9	8.3

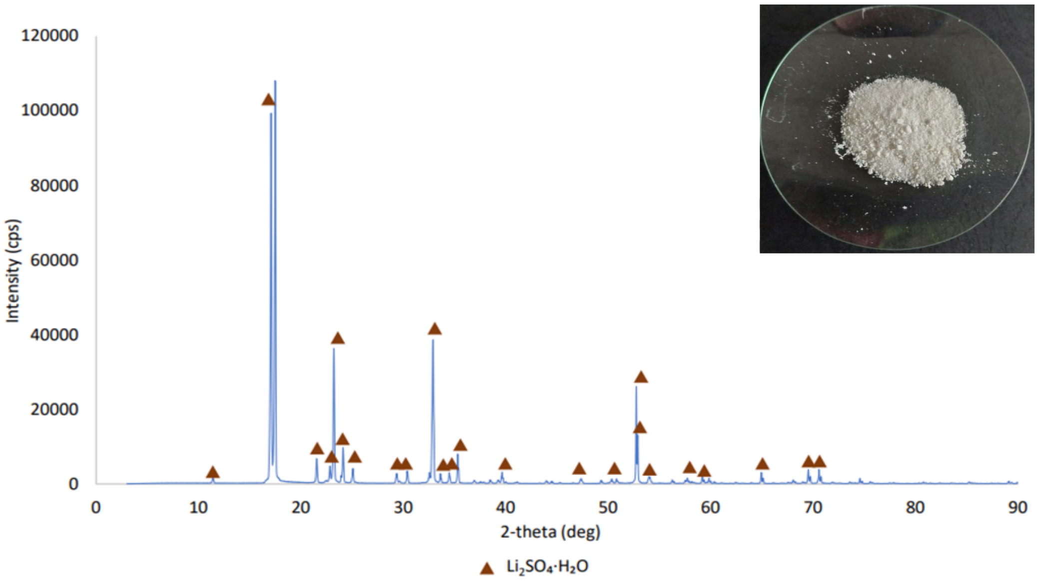


Fig. 3. Results of XRD analyses of the obtained lithium salt product

4. Conclusions

This article presents preliminary results of solvent extraction of lithium from lithium(I) carbonate (IV) solution using Mextral 3938HDC substance from Kopper Chemical Industry Corp., Ltd. The organic phase obtained in the process (Li extract), after washing with water, was re-extracted using demineralized water with the addition of concentrated H_2SO_4 solution. From the solution obtained after re-extraction, $Li_2SO_4{\cdot}H_2O$ salt was crystallized as the end product of the process. The obtained results of lithium ion extraction efficiency from lithium carbonate solution amounting to 68.9% are sufficient to conduct process tests on a larger scale. And they are almost 25% higher than the published experimental results of solvent extraction of lithium from synthetic liquors after processing LIBs using CYANEX®936P, in which the authors of the work obtained 44% Li extraction for 10% extractant concentration in contact with a solution at pH 2 (Solis et al., 2022). But 26.1% lower when the solvent extraction process using CYANEX®936P is carried out from a synthetic solution containing lithium ions at pH 12 for a concentration of 8% v/v of the organic to inorganic phase (O/A), where only one stage was required in accordance with the development of the McCabe-Thiele diagram (Solis and Yezica, 2022).

Further research on the solvent extraction process using the extractant Mextral 3938HDC from Kopperchem Chemical Industry Corp. may change the current approach to designing new technological processes for the recovery of raw materials from the battery mass resulting from the mechanical processing of used and/or waste lithium-ion cells. This change will be related to the fact that the entire technology of recycling and recovery of copper, cobalt, nickel, manganese and lithium metals from solutions after acidic reductive leaching of battery masses will be able to be an automated extraction process completed by the crystallization of salts, which are desirable commercial products with high market prices 7 143 $/MT $CoSO_4$, 4 018 $/MT $NiSO_4$, 10 119 $/MT Li_2CO_3 and 9 875 $/MT $LiOH{\cdot}H_2O$ (Mineral Prices, 2025).

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