

## Modelling, simulation and technical assessment of spent Li-ion batteries recycling plant

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### Abstract

The literature clearly indicates that both academia and industry are strongly committed to develop comprehensive processes for spent Li-ion batteries (LIB) recycling. In this regard the current study comes with an original contribution by providing a quantitative assessment of a large scale recycling plant designed for the treatment of the complete spent LIB. Besides the basic process concept (Case I) the assessment also considered the case study with thermal integration, involving the combustion of H<sub>2</sub> generated in the process (Case II), and the one with recycling of CO<sub>2</sub> and water into the process (Case III). Also, the impact on the technical performances of different reducing agents and the addition of CO<sub>2</sub> capture subsystem to the spent LIB recycling plant were quantified. Process flow modeling software was used to evaluate the contribution of all process steps and equipments to the overall energy consumption and to the mass balance data required for the technical assessment of the large-scale recycling plant. To underline the advantages and identify the optimal novel process concept several key performance indicators were determined such as recovery efficiency, specific energy/material consumptions, and specific CO<sub>2</sub> emissions. The results revealed that the thermally integrated process with H<sub>2</sub> combustion, recirculation of CO<sub>2</sub> and water is the most promising large scale configuration of the spent LIB recycling plant even if CO<sub>2</sub> capture is applied.

### 1. Introduction

Based on the current tendency it is estimated that on a global scale the LIB industry will reach in 2026 the capacity of 1000 GWh corresponding to almost USD 140 billion which is almost four times the value reported in 2019 and only half of the capacity predicted for 2030 (Abdelbaky, et al. 2021; Deshwal, et al. 2022; Shi, et al. 2023). It is undeniable that LIB production capacity is on an ascendant trajectory, increasing from year to year in correlation with other industrial sectors, which boosts energy and raw material consumption to a level that clearly cannot be economically sustained by current supplies without a proper recycling (Henckens 2021; Miao, et al. 2023). From the demand and price evolution of key energy transition minerals it can be noticed that in just five years the market increased with 200% to 320 billion USD last year (IEA 2021; IEA 2023a). Clearly, the gap between demand and supply deepens and production costs will significantly increase due to the increase of raw materials uncertainty and unavailability.

However, the biggest risk for LIB production is not related to the unforeseen market impact of natural resources availability dynamics, but continues and unpredictable decline of ore quality which involves more waste, higher emissions, and all together increasing exploration costs (Ciez and Whitacre 2019; Henckens 2021). For instance, Jose-Luis et al. reported a decline in copper ore grade with an average of 25% in Chile between 2003 and 2013 leading to an increase of energy costs with almost 50% (Jose-Luis, et al. 2019). Other publications indicate the decrease in copper and nickel ore quality with almost 28% between 2010 and 2017, accompanied by an increase of tailings and waste rock output with 13% and 37% respectively (Azadi, et al. 2020). Increasing processing cost of lower grade ores means that raw material costs will make up a larger share of the total cost of LIB productions despite the fact that cost dropped from 1100 USD/kWh in 2010 to 150-180 USD/kWh in 2022 (Ciez and Whitacre 2019; Hoarau and Lorang 2022; IEA 2023b). For instance, the share of cathode material in battery costs increased significantly from under 5% in 2015 to 20% in 2021 and in just one year reached 40%. This is the most obvious for cobalt rich LIB (Thompson, et al. 2021) but in general the active cathode material accounts for an average of 34% of the whole battery cell costs and an average of 50% (Thompson, et al. 2021) of the total material costs exceeding greatly the cost associated with other battery components (Gaines, et al. 2021; Thompson, et al. 2020; Wu, et al. 2023).

Different assessments revealed that the cathode material is not only the costliest component in newly manufactured LIB but at the same time it is the most important part of spent LIB because it has the highest recycling potential from both technical and economic points of view (Dobó, et al. 2023). So, when it comes down to sustainability of large scale LIB production, recycling and recovery it is mandatory in order to cover the necessities of the present without compromising the future (Goyal, et al. 2023). According to Costa et al. and Dewulf et al. the cost benefit of LIB recycling goes beyond just conserving primary reserves of critical minerals because the 51% decrease of virgin materials use includes the reduction of energy vectors associated with LIB production as well, and it can prevent 10 to 30% of the production related environmental impact (Costa, et al. 2021; Dewulf, et al. 2010).

Therefore, recycling spent LIB can contribute to environmental preservation, inhibit the depletion of primary resource, diminish the amount of waste, and ultimately yield economic advantages thus promoting a sustainable development (Costa, et al. 2021; Kong, et al. 2023). Still, meeting this ambitious target requires more than a series of policies, measures and potential secondary resources; it needs a massive research and innovation effort to ensure the development and large scale deployment of ground-breaking, cost effective and eco-friendly spent LIB recycling technologies. Unfortunately, current recycling of spent LIB is performed in existing large-scale industrial processes, initially developed for the extractive cobalt or nickel metallurgy, which are normally adjusted but not dedicated to LIB recycling (Liu, et al. 2019). It is well known that recycling companies have difficulties to meet the stipulated 50% mass recovery by the Directive 2012/19/EU, especially in the case of Li, not to mention the fact that the applied pyrometallurgical options are highly energy and cost intensive (Guo, et al. 2017; Makuza, et al. 2021). Another important problem is related to the superficial and incomplete processing of spent LIB in many studies, focusing only on some material fractions or process steps, without offering a comprehensive techno-economic assessment (Cabral-Neto, et al. 2023).

To fill the existing gap and promote the deployment of industrial scale spent LIB recycling technologies the current study defines an overall process and identifies the technical performances not for individual subsystems, operating at laboratory scale, but for integrated large scale recycling plant that includes all essential subsystems. This approach leads to overall conclusions regarding the technical performance variations for the integrated industrial scale recycling plant and provides the necessary data for the future economic and environmental assessments of the process.

**Keywords:** spent Li-ion batteries, recycling, modeling, simulation.

## **2. Plant configuration and model assumptions**

### **2.1. Description of the mathematical models developed for the recovery process of metals from spent LIB**

Fig. 1. presents the mathematical model developed for the recycling process of spent LIB with a processing capacity of 1314 kg/h. As can be seen, the mathematical model involves a sequential processing of spent LIB leading to the recovery of nickel and lithium in the form of carbonates and manganese and cobalt in the form of sulfates. In addition, the recycling plant generates by-products that are used in process integration or can be valorized together with the main products. The process is divided into the following subsystems:

**1. The mechano-thermal treatment of spent LIB** is the first stage of the process and its main purpose is the disassembly and sorting of spent LIB into different fractions of materials that are prepared through thermal treatments for the chemical dissolution of the components of interest. The material obtained in the grinding and sieving step is subjected to thermal treatment in two stages: (i) at a temperature of 370 °C occurs the separation of the electrolyte and its treatment; (ii) and at the temperature of 797 °C the plastic fractions are converted by pyrolysis to combustible products with the parallel partial carbothermic reduction of oxide materials. In addition this technological stage involves the separation of magnetic metals (Ni, Fe, Co) from non-magnetic materials which are then transferred to the corresponding subsystems for their processing.

**2. Dissolution and purification.** This subsystem is designed for the processing of the non-magnetic material stream obtained in the mechano-thermal treatment of spent LIB. The dissolution of oxides takes place in an acidic environment and in the presence of a reducing agent (oxalic, formic, citric acid, H<sub>2</sub>O<sub>2</sub>) depending on the case study, followed by the separation, in an alkaline environment, of the solution rich in sulfates of Mn, Ni, Co from other secondary products.

**3. Separation and recovery of manganese.** In this subsystem, the selective extraction of  $\text{MnSO}_4 \cdot x\text{H}_2\text{O}$  takes place in an alkaline environment and in the presence of the solvents kerosene, respectively Di-(2-Ethyl Hexyl) phosphoric acid. To minimize the consumption of solvents, the subsystem involves their regeneration in sulfuric acid medium and their recirculation in the process.

**4. Dissolution of magnetic metals.** The flow of magnetic metals (Ni, Fe, Co) is treated with sulfuric acid in adiabatic conditions to obtain the corresponding sulfate solutions respectively to produce  $\text{H}_2$  usable for thermal energy generation in the process.

**5. Separation and recovery of cobalt.** The extraction process is similar to the one implemented in the case of  $\text{MnSO}_4 \cdot x\text{H}_2\text{O}$  with the difference that Di-(2-Ethyl Hexyl) phosphoric acid is replaced by CYANEX which presents selectivity for  $\text{Co}^{2+}$ . In addition, obtaining solid  $\text{CoSO}_4$  also involves a crystallization-recrystallization and filtration step.

**6. Separation and recovery of nickel.** From the aqueous solutions of sulfates resulting in the last two stages, a solution of  $\text{NiSO}_4$  is obtained, by crystallization-recrystallization respectively filtration, which is treated with a solution of  $\text{Na}_2\text{CO}_3$  in order to precipitate and separate solid  $\text{NiCO}_3$ .

**7. Separation and recovery of lithium.** In the last subsystem of the process,  $\text{Li}_2\text{CO}_3$  is obtained through the following two consecutive steps: (i) obtaining the  $\text{LiOH}$  solution by treating the exhausted solution from nickel recovery subsystem with  $\text{Ca}(\text{OH})_2$  and mixing the filtrate with the  $\text{LiOH}$  solution from dissolution and purification subsystem; (ii) carbonation of  $\text{LiOH}$  solution followed by crystallization and filtration of  $\text{Li}_2\text{CO}_3$ .

For the evaluation of the technical performances of the spent LIB recycling plant the following case studies were considered:

- **Case I** - thermally non-integrated process - to generate the thermal energy requirement, combustion of additional amounts of  $\text{CH}_4$  was considered
- **Case II** - thermally integrated process - with  $\text{H}_2$  combustion
- **Case III** - thermally integrated process with recirculation of  $\text{CO}_2$  and water into the process.

## 2.2. Description of the mathematical model developed for the $\text{CO}_2$ capture process

Considering the importance of  $\text{CO}_2$  capture in the industrial sector, the recycling plant of spent LIB was coupled with a post-combustion  $\text{CO}_2$  capture based on amine absorption process. According to Fig. 2. the  $\text{CO}_2$  capture process involves the following three major steps: (i) absorption of  $\text{CO}_2$  in the lean amine solution at a temperature of approximately 35–55 °C and a pressure of 1.05 bar; (ii) The rich amine- $\text{CO}_2$  stream is pumped and heated to a temperature of about 100–120 °C using the hot mass of solvent from the bottom of the desorption column. After preheating, the  $\text{CO}_2$ -rich stream is passed to the desorption column where the solvent regeneration is done by using the thermal energy provided by the recycling plant; (iii) the  $\text{CO}_2$  stream is dried and compressed in 4 stages up to the storage pressure of 122 bar.

## 2.3. Methodology and basic assumption

The evaluation of the technical performances for the recovery processes of metals from spent LIB based on the defined mathematical models were carried out by simulating and optimizing the processes using process flow modeling software CHEMCAD specific to chemical engineering. Considering the operating conditions and the physico-chemical properties of the compounds involved, the Soave–Redlich–Kwong (SRK) thermodynamic model with Boston–Mathias modifications was used. In the simulations, chemical and phase equilibrium conditions based on the Gibbs free energy minimization model were considered. The pressure loss in the heat exchangers was considered 1%, the minimum temperature difference for the thermal integration of 10 °C and the pressure drop in the barometric condenser of 46 mbar. In order to identify the optimal systems for metal recovery from spent LIB, the technical performances of the thermally integrated/non-integrated technological variants, respectively with and without  $\text{CO}_2$  capture, were compared in stationary conditions. In addition, the impact of the type of reducing agent ( $\text{H}_2\text{O}_2$ ,  $\text{C}_6\text{H}_8\text{O}_7$ ,  $\text{HCOOH}$  and  $\text{H}_2\text{C}_2\text{O}_4$ ) on the performance of the spent LIB recycling plant was assessed.

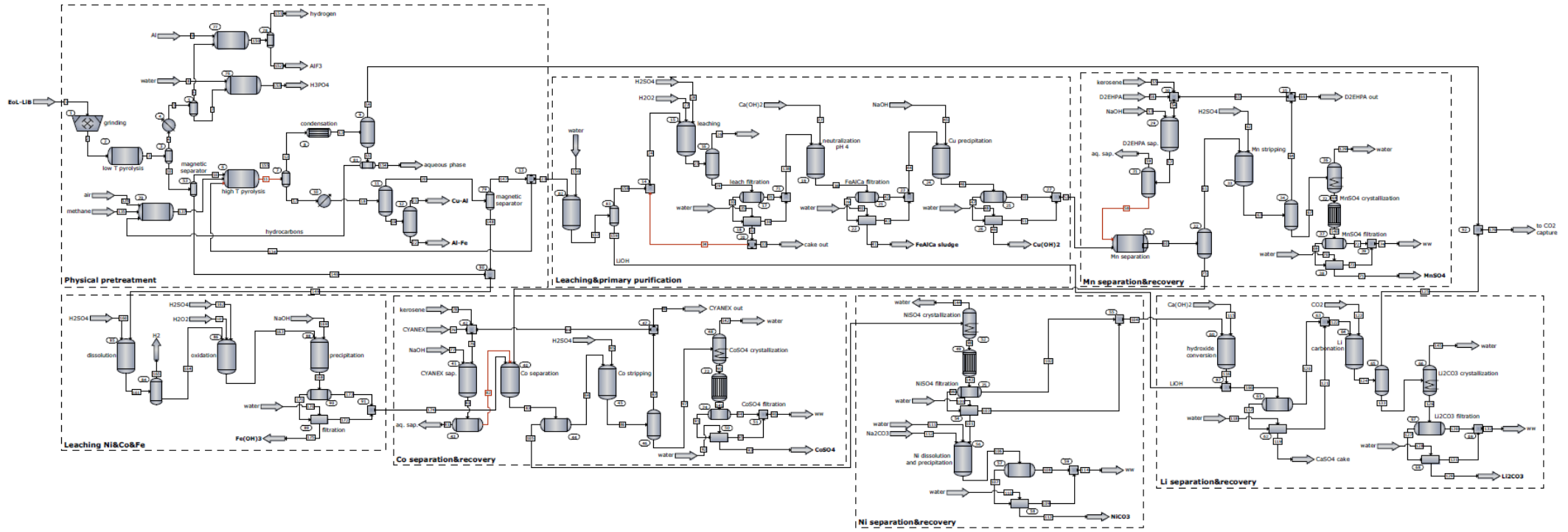


Fig. 1. Process flow diagram of the spent Li-ion batteries recycling plant.

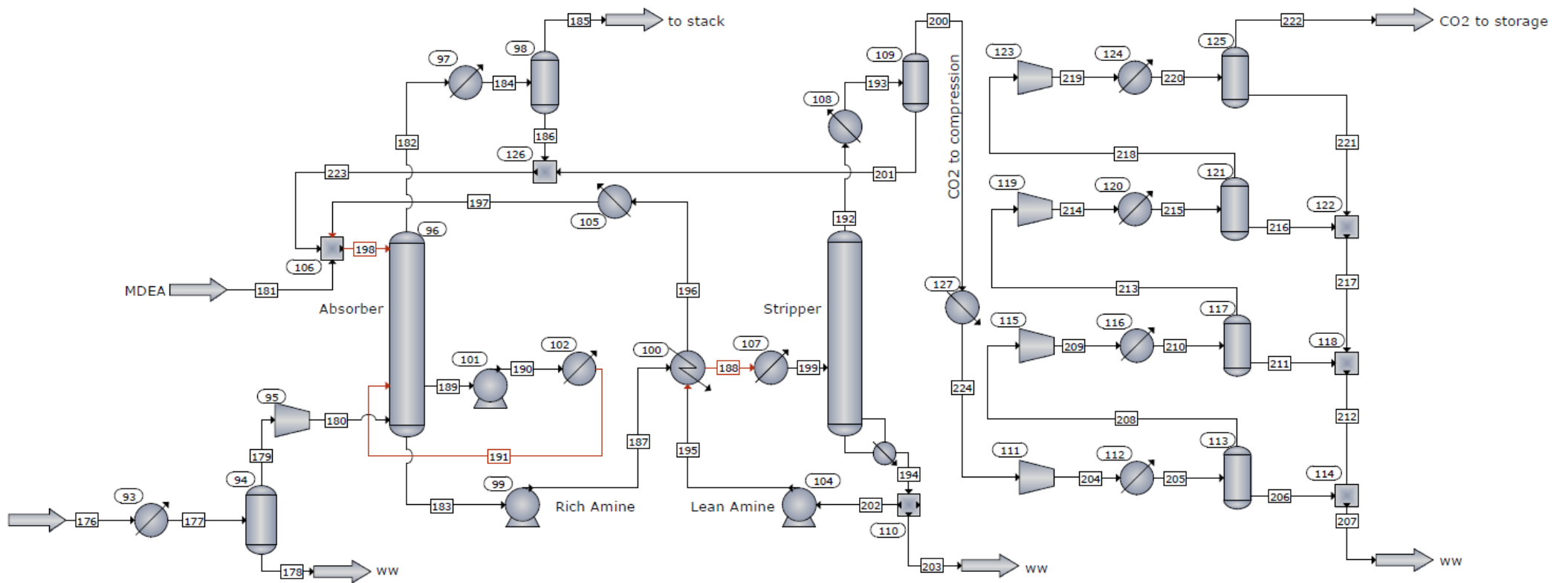


Fig. 2. Process flow diagram for the CO<sub>2</sub> captures process.

### 3. Results and discussions

Based on the material balance data, the recovery yield and production rate for the main products of the spent LIB recycling process were determined. Considering that the values of these performance indicators are very similar, almost identical for the evaluated technological variants, only the average values are presented in Table 1. The results obtained indicate a high performance because, apart from the recovery yield of cobalt, they exceed 80% and in some situations even 90%.

**Table 1. Recovery yields and production rates for the main products of the spent LIB recycling process.**

Product	MnSO <sub>4</sub> xH <sub>2</sub> O	CoSO <sub>4</sub> x7H <sub>2</sub> O	NiCO <sub>3</sub>	Li <sub>2</sub> CO <sub>3</sub>	Fe(OH) <sub>3</sub>	Grafit	Al	Cu
<b>Production rate, kg/h</b>	206.63	299.03	208.76	131.81	98.77	189.71	296.25	101.5
<b>Recovery yield, %</b>	84.09	79.26	82.61	94.84	98.21	87.50	93.24	90.91

Clearly, achieving these performances, regardless of the particularities of the technological variants, involves the supply of raw materials in different amounts and ratios to the process. According to the data in Table 2, the specific consumption of raw materials in kg/kg LIB are the highest for the thermally non-integrated process (I) in which the combustion of additional amounts of CH<sub>4</sub>, to generate the thermal energy requirement, involves the use of an increased consumption of air. Almost 80% of the total specific consumption of raw materials is represented by the air used in the combustion process of CH<sub>4</sub>. In comparison, the thermally integrated process (II) which uses less CH<sub>4</sub> and implicitly air, is characterized by a specific air consumption of approx. 5 times lower than for (I) which also led to a 250% lower total specific consumption of raw materials. As can be seen, the performances are even higher for the integrated thermal process with recirculation of water and CO<sub>2</sub> into the process (III) which requires a total specific consumption of raw materials almost 300% lower than (I).

**Table 2. Specific consumption of raw materials in kg/kg LIB for the spent LIB recycling process.**

Raw material	H <sub>2</sub> O	CH <sub>4</sub>	H <sub>2</sub> SO <sub>4</sub>	Na <sub>2</sub> CO <sub>3</sub>	NaOH	air	Ca(OH) <sub>2</sub>	CO <sub>2</sub>	TOTAL
<b>Consumption, kg/h</b>	2544	120	1019.7	211.98	424.76	3500	137	98.5	-
<b>Case I</b>	1.94	0.09	0.78	0.16	0.32	11.39	0.10	0.07	<b>14.85</b>
<b>Case II</b>	1.94	0.09	0.78	0.16	0.32	2.66	0.10	0.07	<b>6.17</b>
<b>Case III</b>	1.10	0.09	0.78	0.16	0.32	2.66	0.10	0	<b>5.22</b>

Regarding the specific consumption of raw materials in kg/kg product, for different subsystems of the spent LIB recycling process, the results in Table 3 indicate the highest values for the subsystem used in the dissolution of magnetic metals (4) followed by the subsystems defined for separation and recovery of Ni (6) and finally for the mechano-thermal treatment of spent LIB (1). Since the values are relatively close for the case studies involving different reducing agents, Table 3 presents only the data for the one with oxalic acid. In contrast the subsystem for the dissolution and purification of oxide materials (2), respectively the one defined for obtaining Li<sub>2</sub>CO<sub>3</sub> (7) involve the lowest specific consumption of raw materials.

**Table 3. Specific consumption of raw materials in kg/kg product for different subsystems of the spent LIB recycling process using oxalic acid.**

No. subsystem	1	2	3	4	5	6	7
<b>TOTAL, kg/h</b>	4959.5	801.86	727.10	1145.69	693.60	861.98	272.50
<b>W, kg/kg</b>	3.77	2.08	3.52	4.61	2.32	4.13	2.07

On the other hand, the performances are completely different from the point of view of the specific thermal energy consumption determined on the basis of the energy balance data. Most importantly the results in Table 4 indicate that among all the subsystems of the spent LIB recycling plant, only the dissolution and purification subsystem is an energy generator even for the non-thermally integrated process. Unlike the specific consumption of raw materials, this time the subsystem used for the separation and recovery of cobalt ensures the lowest performances because it consumes more than 50% of the thermal energy provided for (I). In second place are the subsystems operated in the separation and recovery of nickel and manganese. It is important to mention

that the thermal integration of the process massively reduces the consumption of thermal energy and CH<sub>4</sub>, leading to 10 times lower values. Even if the specific consumption of CH<sub>4</sub> does not decrease to zero for the type (II) process, it is worth noting that the system can provide 3.8 MJ of thermal energy with each kg of recycled LIB, and at a rather high potential (380 °C).

**Table 4. Thermal energy balance for the recycling process of spent LIB using oxalic acid.**

No. subsystem	Parameters of energy flows	Thermal energy generated			Thermal energy consumed			TOTAL consumption, MJ/h	Specific consumption, MJ/kg	Equivalent consumption, kg CH <sub>4</sub> /h
		1	2	3	1	2	3			
1	T, °C	120	40	380	370	797				
	Q, MJ/h	-488	-51	-11269	1173	76		1249	0.95	25
2	T, °C	60								
	Q, MJ/h	-1704						0	0	0
3	T, °C	24			24	40	25			
	Q, MJ/h	-1687			1469	271	419	2159	10.45	43
4	T, °C	32			40					
	Q, MJ/h	-1713			700			700	2.82	14
5	T, °C	24			24	40	25			
	Q, MJ/h	-1395			1384	5057	582	7023	23.49	140
6	T, °C	40	110		110					
	Q, MJ/h	-258	-583		2129			2129	10.20	43
7	T, °C	35			93					
	Q, MJ/h	-112			387			387	2.94	8
<b>Case I</b>								<b>13646</b>		<b>273</b>
<b>Case II</b>	T, °C	380			370	797				
	Q, MJ/h	-3781			1173	76		<b>1249</b>	<b>0.95</b>	<b>25</b>

The thermally non-integrated process (I) stands out as the least efficient in terms of total and specific CO<sub>2</sub> emissions, respectively the average energy consumption of the CO<sub>2</sub> capture process for the recycling of spent LIB. As can be seen from Table 5, in this case study approximately 1.5 times more CO<sub>2</sub> is generated than in (II) and 3 times more than in process (II) which involves H<sub>2</sub> combustion and partial reuse of CO<sub>2</sub> generated. Regarding the energy consumption for CO<sub>2</sub> capture, apart from the non-thermally integrated process, for all other technological variants it can be covered by the thermal energy generated in the recycling process of spent LIB. Moreover, the process remains a thermal energy generator even with CO<sub>2</sub> capture producing 1.5 GJ/h for process type (II), ~2 GJ/h for process (II) with H<sub>2</sub> combustion and 2.3 GJ/h for process type (II) with H<sub>2</sub> combustion and partial reuse of CO<sub>2</sub> in the process, the last technological option being the most efficient in terms of recycling spent LIB, regardless of the reducer used.

**Table 5. Total and specific CO<sub>2</sub> emissions, respectively the average energy consumption of the CO<sub>2</sub> capture process for the recycling of spent LIB.**

Process type	Case I		Case II		Case II with H <sub>2</sub> combustion		Case II with H <sub>2</sub> combustion and partial reuse of CO <sub>2</sub> in the process	
	Total, kgCO <sub>2</sub> /h	kg CO <sub>2</sub> /kg LIB	Total, kgCO <sub>2</sub> /h	kg CO <sub>2</sub> /kg LIB	Total, kgCO <sub>2</sub> /h	kg CO <sub>2</sub> /kg LIB	Total, kgCO <sub>2</sub> /h	kg CO <sub>2</sub> /kg LIB
H <sub>2</sub> O <sub>2</sub>	1402	1.07	652	0.50	516	0.39	417	0.32
C <sub>6</sub> H <sub>8</sub> O <sub>7</sub>	1418	1.08	667	0.51	517	0.39	418	0.32

<b>HCOOH</b>	1425	1.08	675	0.51	525	0.40	426	0.32
<b>H<sub>2</sub>C<sub>2</sub>O<sub>4</sub></b>	1449	1.10	698	0.53	548	0.42	449	0.34
<b>CO<sub>2</sub> capture energy consumption, GJ/h</b>	4.73		2.24		1.75		1.42	

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## Conclusions

The obtained results proved that the developed, modeled and simulated a recycling plant is adequate and efficient for the sequential processing of spent LIB allowing the recovery of nickel and lithium in the form of carbonates and manganese and cobalt in the form of sulfates. Additional by-products were also generated such as CaSO<sub>4</sub>, Fe<sub>2</sub>(SO<sub>4</sub>)<sub>3</sub> that can be valorized in other industrial sectors. It was found that in the best operating conditions the recovery rate of critical materials is over 80% and the purity of the obtained main products was more than 99 % which makes them suitable for new LIB production or other industrial use. Based on the energy balance data it can be concluded that beyond its technical potential the process remains a thermal energy generator even with CO<sub>2</sub> capture, producing 1.5 GJ/h for thermally integrated process, ~2 GJ/h for thermally integrated process with H<sub>2</sub> combustion and 2.3 GJ/h for thermally integrated process with H<sub>2</sub> combustion and partial reuse of CO<sub>2</sub>. As an overall conclusion it can be stated that the technological option with H<sub>2</sub> combustion and partial reuse of CO<sub>2</sub> is the most efficient in terms of recycling spent LIB regardless of the reducer used and/or CO<sub>2</sub> involvement.

Given the current findings, future studies may rethink and redesign some key steps and investigate other case studies as well to improve the technical performances of the spent LIB recycling plant. Moreover, it is necessary to assess the economic potential and apply a LCA for the developed conceptual recycling plant in order to reach global conclusion in comparison to other processes.

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## Data availability

All data generated or analyzed during this study are included in this publish article.

## Declarations

**Ethical approval:** Not applicable.

**Consent to participate:** Not applicable.

**Consent for publication:** Not applicable.

**Competing interests:** The authors declare no competing interests.



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