



D7.8 WORKING PAPER ON IDENTIFIED TECHNOLOGY GAPS AND INNOVATION POTENTIALS

Lithium, cobalt, nickel and graphite: technological gaps in the EU supply chain and innovation potential

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ABSTRACT

The current working paper examines the technological gaps and respective innovative potentials in the supply chain of lithium, cobalt, nickel and graphite. These materials have a significant role for the green energy transition. Initially, the available primary and secondary resources in the European Union are evaluated according to their: (a) amount, (b) quality and (c) exploitation feasibility. The most efficient industrial processing practices that can be adapted or optimized are identified. Finally, novel technological practices that have been tested at pilot or semi-industrial scale are proposed for the processing of currently no-exploited primary and secondary resources.

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INTRODUCTION

The Climate Law set by European Commission defines a legally binding target of net zero greenhouse gas emissions by 2050. The law also sets the intermediate target of reducing net greenhouse gas emissions by at least 55% by 2030, compared to 1990 levels (**climate.ec.europa.eu**). The productivity increase of specific critical raw materials (CRM), by primary or secondary resources, is necessary for the transition to the green energy era. Lithium, cobalt, nickel and graphite are involved in a number of crucial green-energy applications. Li-ion batteries for electric vehicles (EVs), apart of lithium, contain graphite in their anode and cobalt at various complex phases. SmCo₅ and Sm₂Co₁₇ alloys consist strong permanent magnets in electric vehicles (EVs) motors presenting extremely resistant to demagnetization. Stainless steel is widely used for the construction of wind turbine system. Graphite is the main material for the construction of bipolar plates in fuel cells, while it is contained in electrodes of vanadium redox flow batteries used for large scale energy storage (**innovationnewsnetwork, 2022; ifpenergiesnouvelles, 2021; elements.visualcapitalist, 2021**), (**Table 1**).

	APPLICATIONS				
CRM	Li-ion battery	samarium– cobalt magnets	stainless steels i wind turbines	Vanadium n redox flow batteries	Hydrogen fuel cells
Lithium	+				
Cobalt	+	+			
Nickel	+		+		
Graphite	+			+	+

Table 1. The most significant green energy applications involving the use of Li, Co, Ni and graphite.

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The precise forecast concerning the supply/demand balance and the price evolution for Co (cobalt), Ni (nickel) and Li (lithium) in medium term is challenging due to various uncertainties in green energy sector, nevertheless, it is generally accepted (**Goldman Sachs, 2022a; Goldman Sachs, 2022b**) that the EVs market expansion will lead to the necessity for larger produced amounts for these metals (**Figure 1**). Taking into account these conditions, new routes and innovative technologies should be developed in the European industry aiming to the exploitation of primary and secondary resources of lithium, cobalt, nickel and graphite.



Figure 1. Demand increase forecast for nickel, cobalt and lithium by the EVs industry (left) and balance of global nickel supply/demand (right) until 2030 (Goldman Sachs, 2022a; Goldman Sachs, 2022b).

1. PRIMARY RESOURCES: TECHNOLOGY GAPS AND INNOVATION POTENTIALS

1.1 LITHIUM

Currently, lithium production by primary resources is not taking place in EU. About 18.000 tonnes of Li₂CO₃ and 5.000 tonnes of LiOH were imported in 2021. Lithium carbonate is mainly imported from Chile, while lithium hydroxide in mainly imported from Switzerland (at the processing stage) and Russia (**Eurostat, 2021**). The mining and metallurgical processing of lithium from hard rocks (i.e. spodumene and various Li-bearing mica-group species) is promising. The EU has a number of Li resources that it is expected to exploit in the short term (during the next few years). **Table 2** summarizes the techno-economical characteristics of the most significant expected lithium projects in EU. Cinovec in Czech Republic consists a world-class deposit (reserves are estimated at >50 million tonnes with > 0.4 wt.% Li₂O, while the indicated + inferred resources are estimated to be 660 million tonnes). The project is expected to provide 360,000 t/a of mica concentrate to produce both LiOH and Li₂CO₃ (**europeanmet.com**). Six more deposits with a Li carbonate equivalent ranged between 0.27 and 1.6





million tonnes have been exhaustively defined. Their respective metallurgical projects are focused to the production of LiOH end product or to a Li-rich concentrate. The feasibility study for all described projects has been completed or it is expected to be completed shortly.

Table 2. Mature mining/metallurgical projects of lithium in EU and their techno-economical characteristics (data were collected by the official web addresses of respective Companies) (FS: feasibility study, PFS: pre-feasibility study).

Company	European Metals	Infinity Lithium	Imerys	Savanna	Bacanora	Keliber	European Lithium
Project	Cinovec	San Jose	Beauvoir	Mino do Baroso	Zinnwald	Keliber	Wolfsberg
Country	Czech Republic	Spain	France	Portugal	Germany	Finland	Austria
Mineral	Zinnwaldite	Zinnwaldite	Mica	Spodumene	Zinnwaldite	Spodumene	Spodumene
Li ₂ O(%)	0.42	0.86	0.9	1.06	0.76	1.16	1.0
Resources (Li carbonate equivalent) in Mt	7	1.6	1.0	0.7	0.38	0.29	0.27
Product	Li ₂ CO ₃ /LiO H	LiOH	LiOH	Spodumene	LiOH	LiOH	LiOH
Production per year	≈30000	15000	34000	175000	12000	15000	10000
STATUS FS	In progress	In progress	2024/ 2028 Project begins	Scoping Study 2019	2023	2019	PFS 2018

The metallurgical processing of Li bearing hard rocks is well established and comprises the main steps in case of spodumene: (a) calcination of a-spodumene to b-spodumene at 1100 °C, (b) sulphuric acid digestion at high temperature (250 °C), (c) neutralization/purification of the leachate and (d) precipitation of Li as carbonate using Na₂CO₃. The processing of Li-micas (i.e. lepidolite) comprises: (a) roasting with Na₂SO₄ at 1000 °C, (b) water leaching of LiKSO₄ and Li₂NaK(SO₄)₂ phases and (c) precipitation with Na₂CO₃ (Liu et al. 2023).

The high-energy consuming calcination, of both a-spodumene and micas, and the use of dense H_2SO_4 in case of b-spodumene leaching, are the steps that mostly effect on the sustainability of the whole metallurgical process. Recently, a number of metallurgical companies work on the development of methodologies presenting a low environmental footprint that could replace the calcination or/and H_2SO_4 leaching stages. The following cases that have been tested at pilot scale should be mentioned:

1. The Outotec lithium hydroxide process

Aims on the replacement of the sulfuric acid leaching of b-spodumene. The process concept is based on a two-stage alkaline leach process. Lithium is first extracted from the silicate mineral in a pressure leaching stage using soda ash. Lithium carbonate (intermediate product) and zeolite analcime





(commercial by-product) are formed. At the second step, Li_2CO_3 is converted to the LiOH end product via leaching with $Ca(OH)_2$ and crystallized as LiOH.H₂O (**Figure 2**). Overall, the yield from concentrate for lithium leaching extraction typically exceeds 90% (**metso, 2019**).





2. LieNa process (Lithium Australia & ANSTO)

The technique developed by Lithium Australia and ANSTO based on the direct processing of aspodumene with Na_2CO_3 to form a synthetic lithium sodalite. Most of the initial lithium amount is concentrated into the formed sodalite, which is recovered via a simple, solid/liquid separation step. The lithium within the sodalite is weakly bound, so an exchange reaction that substitutes H⁺ for Li⁺ allows it to be recovered from the solid by leaching in weak acid (**Figure 3**) instead of using dense H₂SO₄ (**lithium-au.com**).







Figure 3. LieNa process for the direct processing of a-spodumene (lithium-au.com/about-liena/).

The adaptation of the above mentioned, or similar, technologies by the European Lithium extractive industry, which is expected to be established shortly, will render European lithium projects as more sustainable as the environmental footprint of Li extraction will be lower in comparison to Li mining/metallurgical projects outside EU, while its recovery yields will remain at high levels.

The exploitation of lithium-containing geothermal water in EU constitutes an additional option for Li recovery. The existence of geothermal fluids rich in Li in France and Italy has been described since the early 90es. Lithium concentration in these waters reach 0.04 mol/L (**Pauwels et al. 2009**). Recently, projects for the exploitation of lithium geothermal brines/waters deposits in Germany and France are under development. The Upper Rhine valley geothermal deposit presents total inferred mineral resources of 2.484 million tonnes of brine, at a lithium grade of 181 mg/l Li. Vulcan Energy Resources Company proceeded at tests indicating the recovery of both lithium and geothermal energy by injecting brine deep underground following a carbon-neutral extraction process (**Vulcan Energy, 2021**). Ageli project in Alsace, France, which is being developed by Eramet in partnership with Électricité de Strasbourg aims to combine geothermal energy (decarbonized heat and power production by pumping hot brine deep underground) with one of the world's most efficient battery-grade lithium extraction processes, patented by Eramet. Production is scheduled to start before the end of the decade (**eramet.com**). The industrial exploitation of Li-containing geothermal waters is currently at preliminary stage, therefore respective technological gaps have not been specified.

1.2 COBALT

In 2022 the global terrestrial cobalt resources are about 25 million tons, the vast majority of which are in copper deposits in DRC and Zambia; nickel-bearing laterite resources are in Australia and Cuba meanwhile magmatic nickel-copper sulfide deposits are in Australia, Canada, Russia, and the United States (**Cobalt Market Report 2022**). Currently, Europe accounts only for the 2% of mined and 12% of global refined cobalt (**Cobalt Market Report 2022**). In 2018, Finland accommodated around 1.65% of the world's cobalt production capacity (around 3kt/yr by both primary and secondary resources) and the 2% in 2020. Finland disposes the largest cobalt resources in EU estimated at 312.200 tonnes. Other This project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement No 958211





European resources are in Sweden and Spain, 1.446 t and 5.700, respectively (**Alves Dias et al. 2018**). Finally, in Greece there are cobalt resources in laterites of about 90.300 tonnes, however at very low concentrations (0.05 wt.%) in laterite ores (**Horn et al. 2021; Stankovic et al. 2022**). In 2022, 57% of Co has been used for the production of Li-ion batteries. It is estimated that, by 2029, the global industry will require an additional 100kt, with total consumption of nearly 300kt so the production from primary and/or secondary resources should increase (**Raabe, 2023**).

Sulfide ores

Sulfide ores account approximately 85% of global cobalt resources. The most significant EU cobalt sulphide deposits exist in Finland and Sweden (**Figure 4**). The main type of Co-sulphide deposits, by a geochemical point of view, in these countries are (**Horn et al. 2021**):

- Black-shale hosted deposits (Operating mine with production of Ni (nickel), Zn (zinc), Co (cobalt) and Cu (copper) in Sotkamo, Finland)
- Magmatic-Ni-Cu-PGE (platinum group metals) deposits [Operating mine with Ni, Cu, PGE, Au (gold) and Co production in Kevitsa, Finland]
- Volcanogenic massive sulfide deposits (Operating mine with Cu, Zn, Co and Au production in Kylylahti, Finland)









In Europe, currently Co primary production is derived from three mines in Finland: Sotkamo, Kevista and Kylylahti, representing the three above mentioned of sulphide type deposits. The Finnish company Terrafame extracts Ni, Co, Cu, and Zn from sulfide minerals contained in the black schist ore in Sotkamo using heap bioleaching technology. The company claims that the average CO₂ emissions are only 1.75 kg per kg of nickel sulfate produced. This is a result of the use of heap bioleaching technology, which consumes less electricity and heat. The bioleaching process is technically possible by creating optimal This project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement No 958211





conditions for the natural growth of the bacteria. The primary heap comprises of two heap pads, a lower and an upper section and a corridor between them, pipelines and fixed conveyor lines. From the top, the heap is irrigated with leaching solution, which is collected from the bottom of the heap. Several parameters are monitored and changed to optimize the metals recovery. The primary heap pad is constructed as a dynamic pad while the secondary leaching pads are constructed on top of waste rock dumps (**Pakostova et al. 2017; Riekkola-Vanhanen et al. 2013; terrafame.com, 2022**).

Cobalt-containing concentrates, after the flotation ore enrichment, from Kevitsa and Kylylahti are metallurgically processed in the smelting facilities of Harjavalta and Rönnskär operated by Boliden Company. Cobalt is extracted as by-product via a complex pyro and hydro metallurgical treatment. The first step comprises the flash smelting of the concentrate (Direct Outokumpu, now Outotec process) to the formation of initial low Ni-Co content matte, which is further refined/enriched for the production of the high Ni-Co content electric furnace matte and slag. The electric furnace matte is hydrometallurgically processed (pressure leaching) for the production of copper, Ni, Co and PGM. Cobalt is received under powder form via solvent extraction (Figure 5) Boliden (Boliden, 2018; Svens, 2013).



Figure 5. Flowsheet of the metallurgical processing of magmatic and volcanogenic Co-containing sulfide ores in Harjavalta facilities in Finland (**Svens, 2013**).

The cobalt amount losses during the steps of mining, beneficiation and matte formation, consists a weakness point of the established processing of Cu-Ni-Co sulfides, while there are no available data in the literature concerning the weaknesses of industrial bioleaching. It has been estimated that Ni-Co mining sulfide tailings present nickel and cobalt concentration at the range of 0.2 and 0.01 %, respectively. Low-scale tests have shown that recovery of nickel and cobalt (\approx 91% and \approx 55% yields) can be achieved from this type of tailings using mixed nitric–sulphuric acid solutions at ambient temperature and atmospheric pressure (**Xie et al. 2005**). Furthermore, recently the application of





bioleaching for the recovery of cobalt by sulfide tailing have shown even better results (87% yield) (Mäkinen et al. 2020).

The efficient separation of Ni-Co phases (i.e. pentladite) from other sulfide gangue minerals and copper phases represents a technological challenge. Novel industrial practices comprise the developed of a complex multiple stage flotation system with separate copper and Ni/Co circuits as has been proposed in case of Hautalampi Nickel-Cobalt-Copper Project, Finland (**mining-technology.com, 2023**).

Low–nickel matte is the main intermediate product of nickel sulfide ore in traditional pyrometallurgical smelting, during this process, the valuable metals Ni, Cu, and Co have been enriched. In the traditional smelt route, the low–nickel matte are further processed in blowing converter to reduce the iron content and other impurities forming the high nickel–copper matte. However, through this process, almost 70 wt % Co is lost. Efforts are focused on the direct hydrometallurgical processing of the initial low Ni-Co content matte. The sulphation roasting (up to 700 °C) of the low content Ni-Co matte followed by water leaching was attempted permitting high recoveries of cobalt and nickel (95% and 94%, respectively) (**Sun et al. 2020**).

1.3 NICKEL

Nickel extraction by sulfides is performed via the Direct Outokumpu process as it was already described in the chapter 1.2. In most of cases cobalt and nickel were co-extracted. Nickel in EU was additionally extracted in Greece by nickeliferous laterites until 2022.

The main laterite deposits in Greece are Kastoria, Agios Ioannis, and Evia, owned by the Larco Mining and Metallurgical Company (Bruno Diaz et al. 2019; Economou-Eliopoulos M., 2023).

<u>Greek laterite deposits – Beneficiation.</u> The beneficiation of Greek laterite ores is in many cases practically impossible because the mineral phase in laterites is a colloidal like mixture so the separation is hard. Moreover, the beneficiation of laterites in other countries shows poor results.

<u>Hydrometallurgical technologies.</u> The main industrial scale hydrometallurgical technologies to extract nickel from lateritic ores are high-pressure acid leaching (HPAL), atmospheric pressure tank leaching (AL) and heap leaching (HL), however none of them was applied for the treatment of greek nickeliferous laterites (**Stanković et al. 2020; Stanković et al. 2022**) since there is a lack of capital investment far necessary for the transformation of the existing pyrometallurgical technology to a hydrometallurgical.

Pyrometallurgy

A pyrometallurgical process, applied since the 1960s, is used for the production of Fe-Ni from Greek laterites but does not allow the recovery at the same time of the Co content, which is lost in the metallurgical slag. The methodology is highly energy consuming as the laterite is pre-reduced/roasted at 850-1000 °C followed by reductive smelting for FeNi production at temperatures >1400 °C (**Zevgolis**,





E., Daskalakis, K. 2022). The Greek nickeliferous laterites requires a complete modification of the currently pyrometallurgical process to a hydrometallurgical one aiming to the increase of Ni recovery and the possibility of Co co-extraction. However, the capital investment is considered high. Larco has currently interrupted its activities and a privatization process is taking place. The joint venture of GEK Terna and AD Holdings which is the preferred investor for mining and metallurgical operations has been committed for a 250-million-euro business plan aimed at gradually increasing the production of ferro-nickel and nickel sulfate to 20,000 tons per year. Nickel sulfate is a highly demanded raw material used in the production of batteries for the automotive industry and its price is as high as pure nickel and significantly higher than ferro-nickel. The new joint venture aims to gradually convert the pyrometallurgical process into a hydrometallurgical processing permitting the production of lithium sulfate (ekathimerini.com, 2023). The hydrometallurgical processing of Greek laterites through heap leaching using sulfuric acid has been tested at pilot scale indicating nickel and cobalt recoveries around 60% and 36%, respectively (Agatzini-Leonardou et al. 2021; hydrometallurgy.metal.ntua.gr). Similar nickel and cobalt extraction values (60% and 59%) have by achieved by the column leaching of lowgrade greek limonitic ores (Komnitsas et al. 2018). The achievement of nickel and cobalt extraction degrees over 90% from limonitic laterites has been experimentally proved at intense pressure leaching conditions (temperature between 240-270 °C and pressure between 33-55 atm) (Georgiou and Papangelakis, 1998). The application of the most profitable technique in case of Larco Company should be examined under various techno economical (yield degree of Ni-Co extraction, capital and operating costs etc.) and environmental parameters.

1.4 GRAPHITE

There are three types of natural graphite (NG) for commercial use, classified by purity and particle size: flake graphite, amorphous graphite and vein graphite. China hosts half of the world's graphite reserves, estimated at 110,000 kt. Significant reserves are also located in Mozambique and Tanzania, each with a 15% share of world's total. Concerning the EU, the largest natural graphite deposits are situated in Sweden, Czech Republic and Finland. China is the largest global supplier of natural graphite with 69% of production, followed by India (12%) and Brazil (8%) (CRM, 2020). Small quantities of natural graphite are currently produced in Germany and Austria. Globally, natural graphite has been used for electrodes, refractories, lubricants, foundries, batteries, graphite shapes, recarburizing and others. Moreover, natural graphite ores are mined from either surface or underground mines depending on the proximity of the ore body to the surface. Most staple graphite deposits are tapped using open extraction methods, which have a high environmental impact. A significant amount of natural and synthetic graphite globally produced is used in green energy technologies such as various kinds of batteries and electrodes for industrial purposes (i.e. extractive metallurgy industry) (CRM, 2020) (Figure 6).







Figure 6. The most significant applications of natural and synthetic graphite (ECGA, 2022).

Grade, shape, flake size, and purity of the NG are the most important factors for each application, however the grade of purity is the most determinant factor. Various purification techniques or a combination of them are industrially applied. Hydrometallurgy, pyrometallurgy and physical metallurgy (including comminution, froth flotation, reverse flotation, air elutriation, gravity separation, magnetic separation and electrostatic separation) consist the technological options (Jara et al. 2019).

Hydrometallurgical treatment is the most efficient purification process presenting also a small-scale infrastructure investment, easy implementation. On the other hand, it has a significant environmental footprint as various strong acids, such as hydrofluoric and sulfuric acid, are used. Recently, the industry attempts to the replacing of leaching process by less environmentally hazardous techniques. For example, Renascor Resources Company in Australia applies a combination of flotation separation followed by chemical treatment involving a low-temperature caustic roasting to achieve a high purity grade for the construction of batteries anodes (**invest.sa.gov.au**, **2023**).

Synthetic graphite is generally produced from petroleum cokes. Synthetic graphite can be primary and secondary. Primary synthetic graphite is produced in a very energy-intensive way, known for its associated greenhouse gas emissions. However, its high quality and consistency among products make it desirable for energy transition technologies. Secondary synthetic graphite is produced from the residue of the primary graphite.

The main producers of Synthetic Graphite (SG) are China, India, Europe and USA. The advantage of the SG with respect NG are regular grade, or controllable grade, with absence of impurities whereas the disadvantages can be costly energy intensive (processed with heat treatment in the range of 2500–3000 °C). The heat treatment process can be extremely effective in purifying a graphite material. The high cost of synthetic graphite production acts as a key economic impetus to the development of new natural graphite sources for energy conversion and storage devices, in particular regarding secondary source and recovering from end-of-life devices. Synthetic graphite produced in China and used in EVs that are sold in Europe is not a sustainable material. Increasingly strict environmental, social, and governance (ESG) norms across the EU, together with the development of the carbon border tax and the EU Taxonomy for sustainable finance, will cause issues for European EV producers (Jara et al. 2019; Ritoe at al. 2022).





2. SECONDARY RESOURCES: TECHNOLOGY GAPS AND INNOVATION POTENTIALS

2.1 LITHIUM

Li-ion batteries are the only secondary resource from which Li can be recovered as it is contained at notable concentrations (**Table 3**). Recently, much attention has been given to the recycling processing of electric vehicle batteries (EVB). The following salt materials: $LiCoO_2$, $LiNi_xMn_yCo_zO_2$, $LiNi_0.8Co_{0.15}Al_{0.05}O_2$, $LiMnO_4$, $LiFePO_4$ are used for the construction of Li-ion EVBs. Li is also contained as conducting salt component (most commonly as $LiPF_6$) in the electrolyte of the battery (**Brückner et al. 2020**).

Table 3. Types of Li-containing batteries for: electric vehicles (BEVs), plugin hybrid electric vehicles (PHEVs), electric bikes (pedelecs), and mobile phones and their content in lithium (**Brückner et al. 2020**).

Characteristic	BEV	PHEV	Pedelec	Mobile Phone
Voltage U [V]	355-800	351	22.2-36	3.7
Capacity C [Ah]	60-117	26-34	8-10	0.7-1.2
Energy E [Wh]	21,000-93,000	9000-12,000	189-288	2.4-4.1
Mass m [kg]	235-680	80-135	1.3-4	0.021-0.038

The recycling of Li-ion batteries in EU at an industrial level is limited due to the low availability of endof-life batteries. The recycling legislation (Batteries Directive 2006/66/EG) set by the European Commission obliges a minimum recycling efficiency at least 50% aiming to the sustainable production of metallic values by end-of-life batteries and the decreasing, at the same time, of the environmental footprint (**European Commission, 2020**).

Two main routes: pyrometallurgical and hydrometallurgical followed for the recovery of metallic values from end-of-life batteries (**Figure 7**) (**Brückner et al. 2020**). In case of a pyrometallurgical treatment, a Co-, Cu-, and Ni-containing alloy (metallic phase) or matte (sulfidic phase), an Al-, Mn- and Li-containing slag (oxidic phase), and a fly ash are produced. A further hydrometallurgical step is necessary for the separation of metals contained in the alloy. In case of the hydrometallurgical approach, pre-processing steps (such as mechanical treatment and pyrolysis) are necessary. A metallic fraction containing the electrode materials, which is called black mass, is obtained. The metallic values are separated via precipitation or solvent extraction. The recovery of lithium is challenging in case of the pyrometallurgical route as it is diluted into the slag mass.







Figure 7. The two main routes: pyrometallurgical and hydrometallurgical that applied for the recycling of Li-ion batteries (**Brückner et al. 2020**).

Various techniques have been industrially applied for the recycling of EoL batteries mainly focused on the recovery of Co, Li and Mn, however only a limited number of European recyclers has the possibility to recover lithium. Accurec GmbH Company in Germany has developed a recycling methodology comprising: (a) the pyrolysis of the scrap aiming to the evaporation of the electrolyte and (b) the mechanical/thermal treatment of the remaining. Four different fractions are obtained which are processed individually for the recovery of respective metallic values. Lithium remains in the slag and flue dust after the recovery of Al, Cu, Co, Mn and Ni. Slag and dust are treated through leaching and precipitation and lithium carbonate is obtained as end-product (**Figure 8**) (**Vezzini, 2014**).



Figure 8. Recycling process developed by Accurec GmbH Company (Vezzini, 2014)

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Recupyl company in France applies a pure hydrometallurgical process for the simultaneous extraction of Cu, Co, Co(OH)₂ and Li₂CO₃/Li₃PO₄. The methodology comprises various leaching and precipitation steps after the isolation of the electrode materials via mechanical pre-treatment and electrolysis for the recovery of metallic cobalt (**Figure 9**) (**Vezzini, 2014**).



Figure 9. Recycling process developed by Recupyl Company (Vezzini, 2014)

The main techno-economical barriers that impede the recovery of Li from EoL batteries and the respective innovations that could be investigated are the following:

- The early recycling stage of batteries dismantling is usually preformed manually rendering the up-scale of the process problematic. Further investigations are thus required in order to develop automatic or semi-automatic pretreatment processes (Forte et al. 2021).
- Hybrid recycling technologies should be developed in terms of the simultaneous processing of both lithium-iron-phosphate and nickel-cobalt-manganese batteries aiming to the reducing of both capital and operating cost (Wang et al. 2022).
- The recycling concept is necessary to become more cost-effective through the valorizations of the whole waste including the anode (graphite) and the electrolyte (Larouche et al. 2020).
- The operating cost of the hydrometallurical processes should be reduced focusing on the minimization of amounts of the acid means, while, at the same time, the purity of the lithium products should be improved in order to increase profit margins considering the relatively low value of the cathode material (Wang et al. 2022).
- The environmental footprint of the recycling processes is necessary to be reduced via: (a) the used of more gentle acid in the leaching steps and (b) the better management of the released organic fluorides (Wang et al. 2022).





2.2 COBALT

The recycling rate of cobalt in EU is 35%. Despite there is great interest in Europe in recovering cobalt from secondary resources little information exists on potential cobalt availability from these resources. Secondary resources of cobalt from urban mining are a fast-growing and an important source of cobalt for the supply chain. The Cobalt Institute estimates world cobalt recovery from secondary sources at 10.6 kt for 2020 representing only the 5% of total cobalt supply (Rachidi et al. 2021; Cobalt Market Report 2022). In 2022, secondary production of cobalt is about the same level, 9.3 kt, compared to about the 178 kt of cobalt from primary production. However, this ration is going change by 2040 as the battery scrap material is expected to increase by 60 times, so that the secondary production could reach the 41% of total cobalt supply. The total volume of recyclable material, in terms of energy, in 2022 was approximately 48 GWh and by 2040 will be 2,948 GWh. Produced Co amount by batteries in 2022 represented the 65% of the total production by secondary resources (Cobalt Market Report 2022). End-of-life tungsten cobalt carbides, superalloys and catalysts for carbonylation and hydrosilylation processes are further major secondary resources. In addition to urban mining, secondary resources of cobalt can be derived from metallurgical wastes like copper smelter slags and nickel smelter slags. The solid wastes produced by the metallurgical processing of primary ores however are very limited (Scrreen, 2019). Table 4 summarizes the most significant secondary resources for Co recovery.

SECONDARY RESOURCES, 9.3 kt in 2022				
METALLURGICAL WASTES	Production	Commercial applications		
Copper smelter slags	Very limited	No, only laboratory		
Nickel smelter slags	Very limited	-		
URBAN MINED	The vast of see	condary resources		
Battery recycling	65% of urban mined	Yes, many		
Cermets, tungsten carbide-cobalt	24% of urban mined	Yes		
Alloy scraps & catalysts	11% of urban mined	Yes		
Metal oxide varistors	-	No, only laboratory scale		

Table 4. the most significant secondary resources for Co recovery (Scrreen, 2019).

Mining and metallurgical wastes

Solid wastes produced by the metallurgical processing of primary ores are very limited and there are not data about the metallurgical wastes for commercial recovery of cobalt. Nickel slags and Cu-Ni-Co containing sulfide mine tailings consist the vastest generated Co secondary resources of the mining/metallurgy sectors (**Mäkinen et al. 2020; Lim et al. 2023**). The extraction of valuable metals, among them Co, from the simultaneous exploitation of primary resources, slags and tailings has been proposed and called "Technospheric" mining (**Figure 10**). However, Technospheric mining of mine This project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement No 958211





wastes seems far from the commercialisation stage yet as mine tailings usually contain various metal impurities (such as heavy metals) rendering their simultaneous treatment with scarp challenging (Lim et al. 2021).



Figure 10. Proposed approach for the simultaneous exploitation of primary and secondary (mining/metallurgical wastes) resources (Lim et al. 2021).

Lithium-ion batteries

A global amount of 500 kt of Li-ion batteries was generated in 2019 with a 15% wt.% cobalt content (60 kt). Various recyclers of end-of-life Li-ion batteries worldwide and Europe are targeting on the recovery of cobalt (**Table 5**).

Table 5. Important end-of-life Li-ion batteries recyclers aiming to the extraction of cobalt in Europe and worldwide (**Baum et al. 2022**).

	Worldwide	Europe
Worldwide (tonnes/year, methodology)	Brunp Recycling Technologies, CN, 100,000, Pyro/hydro combo GEM, CN, 30,000, Hydro Quzhou Huayou, CN, 40,000, Pyro	Valdi, FR, 20,000, Pyro Umicore Valeas, BE, 7,000, Pyro/hydro combo Accurec, DE, 4,000, Pyro/hydro combo Redux, DE, 50,000, Pyro Akkuser, FI, 4,000, Pyro/hydro combo

The metallurgical processes applied for the recycling of end-of-life Li-ion batteries, as well as their technological barriers, were extensively described in the previous 2.1 section. **Table 6** quotes the most basic advantages and disadvantages of the pyro and hydro metallurgical processes for the recovery of cobalt by end-of-life Li-ion batteries (**Baum et al. 2022**).

Table 6. Basic advantages and disadvantages of the pyro and hydro metallurgical processes for the recovery of cobalt by end-of-life Li-ion batteries (**Baum et al. 2022**).





PYROMETALLURGY		HYDROMETALLURGY		
Advantages	Disadvantages	Advantages	Disadvantages	
Require simpler pre- treatment methods				
Suitable to recycle LIB of differing compositions,	Energy consumption is very high	Smaller facility cost to implement	High environmental footprint	
shapes, and sizes (i.e. Umicore method is used for both lithium-ion and nickel	Large amount of material loss	High metal recovery High product purity	Use of hazardous strong acids	
batteries) Fixed investment in existing	Large amounts of produced slag	Low energy consumption	Complex processes involving in most of cases an initial thermal	
facilities Mature technology, dominant today thanks to his simplicity, flexibility and rapidity	Li is lost in the slag	Low gas emissions	treatment process	

Cermets, tungsten carbide-cobalt

A limited number of Companies worlwide are activating on the recycling of Co-containing tungsten carbides (WC). Sumitomo Electric Group began recycling cemented carbide in the 1980s using the zinc treatment process where molten zinc is applied to remove the cobalt binder by forming a ZnCo alloy. Zinc is distilled, leaving fragile tungsten carbide and cobalt precipitated at the surface of the particles. The zinc treatment process is favorable because it consumes low quantities of chemicals and energy, requires low capital investment, and is feasible at a small industrial-scale. However, the recycled material quality is degraded because of the scrap coating components also being recycled simultaneously. In 2011, Sumitomo began using a new process based on pyro-hydrometallurgy named oxidation-wet chemical treatment process. Unfortunately, the cobalt in the scrap is lost in the residue and further recovery is not yet performed (**Chandra et al. 2021**).

Co-containing cermet scrap (mainly tools) are commercially recycled by Ceratizit controlled companies as Stadler Metalle in Turkheim, Germany, and Tikomet Oy in Jyvaskyla, Finland. Stadler Metalle specializes in the trade of secondary raw materials, while Tikomet involved in the recycling of hard metal scrap into tungsten carbide-cobalt powder. CERATIZIT, applies two different methods (pyrometallurgy and chemical processing) for the processing of the carbide end-product to the production of a powder which contains over 99% tungsten carbide and Co as a by-product. (ceratizit.com).

Vacuumschmelze and CRONIMET Holding GmbH activating in Germany can be also mentioned as important recyclers of WC containing scrap material (vacuumschmelze.com; cronimet.de).

The identification of specific technological barriers through the industrial processing of tungsten carbide is challenging as very limited information have been published.





Alloy-superalloys scraps & Catalysts

Recycling of superalloys, due to their high melting point and complex chemistry, is very difficult. It is reported that recycling by a single pyrometallurgical process causes a loss of the 20 % of alloying elements, therefore the application of a pyro-hydrometallurgical process seems more suitable. Recovery of cobalt from superalloys is limited at commercial level. MOXBA METREX, a company with headquarter in The Netherlands and with recycling facilities located in the Netherlands (Almelo, Heerlen) and Brazil, processes about 3 kt/y of recycled catalyst materials, after an initial mechanical pre-treatment, a thermal treatment is followed (up to 1200 °C) and next either a hydrometallurgical leaching or pyrometallurgical melting (**Windisch-Kern, 2022**).

2.3 NICKEL

About the 69% of globally produced nickel is used for the construction of stainless steel, while a supplementary 10% is consumed for the construction of non-ferrous alloys and other alloy steels (**nickelinstitute.org**). Nickel in various steels and alloy products is highly recycled. Recently, the scientific interest has been focused on the optimization processes for Ni recovery from end-of-life batteries. Currently, about 11% of world nickel production is consumed in the batteries industry, mainly for rechargeable nickel metal hydride (NiMH or Ni–MH) batteries. However, nickel demand for Li-ion batteries is expected to double from 400 to 800 kt during the next two years (**Goldman Sachs**, **2022b**).

The current recycling practice of Ni-containing batteries (i.e. Umicore company), comprises the simultaneous processing of both NiMH and Li-ion batteries via a complex pyro and hydro metallurgical methodology without mechanical pretreatment of battery cells. The process is designed to co-recover nickel, cobalt and copper as an alloy, which is further processed by hydrometallurgy. The alloy is dissoluted and Cu, Ni/Co are separated via solvent extraction. After separation and raffination, cobalt and nickel can be converted into precursor chemicals for new cathode materials (**Figure 11**). The capacity of the facilities in Hoboken (Belgium) is about 7000 t of scrap per year (**Elwert et al. 2015**). The most significant weakness of the process is that Li contained in Li-ion batteries is lost in slag phase (**Porvali et al. 2020**). Furthermore, there are no available published data concerning: (a) the recovery rate of Ni, Co and Cu, (b) the electric energy consumption amount at the pyrometallurgical step, (c) the kind of acid means that are used at the leaching step.

The increase of the available batteries scrap amount in short term is possible to lead to the preconditions for the separate processing of NiMH and Li-ion batteries. The specialization of the recycling processing in respect of battery type in combination with the performance of a dismantling/enrichment step will reduce the energy consumption and the environmental impact (limitation of produced slag amount, limitation of strong acids use) and increase the recovery yields. An holistic approach for the co-recovery of various metallic values, among them Ni, was already described in the section 2.1.

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2.4 GRAPHITE

Natural graphite

Refractory brick and linings, alumina-graphite refractories for continuous metal castings, magnesiagraphite refractory brick for basic oxygen and electric arc furnaces, and insulation brick led the way in the recycling of graphite products. Recycling of refractory graphite material is increasing, with material being recycled into products such as brake linings and thermal insulation. Recovering high-quality flake graphite from steelmaking kish is technically feasible, but currently not practiced due to the high cost and complexity of methodologies. Information on the quantity and value of recycled graphite is not available (**USGS, 2022**).

Synthetic graphite

There are several sources of graphite for recycling. To date, the main source is still LIBs anodes, which produce the largest volume. A commercial LIB anode has a graphite content of more than 90%, which is obtained from both natural and synthetic source. As it was previously described, Ni and Co are currently commercially recovered by EoL Li-ion batteries, however little attention has been paid to recover graphite via industrial processes by the black mass material formed by the pyrolysis of the initial scrap. It is reported that graphite is recovered at a limited level from EoL batteries through flotation separation, nevertheless the product is not pure enough for those applications requiring graphite of high purity grade (**Abdollahifar et al. 2023**). The general process for graphite recovered by pyrolyzed black mass comprises the steps of floatation, purification and re-graphitization at temperatures over 2600 °C (**Salces et al. 2022; Abdollahifar et al. 2023**). Aiming to the examination of graphite recycling sustainability, a life cycle assessment has been performed in case of 100 kg graphite recovery by EoL Li-ion batteries. The results revealed that enrichment and thermal treatment are the





most energy-intense and environmental hazardous steps, while purification presents a high differentiation in terms of CO_2 equivalent, ranged between 0.53 to 9.76 kg, in relation to the applied technique (**Figure 12**) (**Rey et al. 2021**).

Global Warming Potential (kg CO, equiv)				
0	10 2	0 3	0 40	50
			Fenton +	flotation 48.4
2.5	Leaching +	filtration		
2.9	H₂SO₄ curir	ng-leaching		
	6.8 Oxygen-fr	ee roasting		
2.5	Air he	eating	(in the star
0.5	Pyrolysis	+ flotation		
1.1	Calcination	+ leaching	L L	ARR I
3.8		Calcinatio	n for graphene	6
	9.8	Microwav	e for graphene	

Figure 12. Global warming potential of graphite recycling processes from spent lithium-ion batteries: GWP values in kg·CO₂ equiv. emissions for 1 kg of recycled graphite from spent LIBs (**Rey et al. 2021**).

Among the whole graphite recycling process, re-graphitization step is well investigated and commercialized. The optimization of enrichment (flotation) and purification steps consists the main technological barrier. This material is contaminated with Ni, Co, Li, Cu and Al impurities, therefore a leaching-curing step is necessary (Salces et al. 2022). Leaching/purification with H₂SO₄ consists the most probable and efficient technique, however it presents a high environmental footprint. Acetic acid has been proposed as an efficient and environmentally friendly purification means. The restored graphite exhibits superior electrical performance equivalent to that of commercial graphite (Perumal et al. 2022). An LCA study is necessary to be performed in order the value of the re-generated graphite be correlated with the cost of acid reagents. A new approach that is proposed in case of graphite recovery by spent batteries is based on the classification of the spent carbon material in relation to its damage degree and the implementation of respective specific treatment methodologies in each case. High damaged material should be submitted to structural repair through thermal reduction and modification, while minor damaged material is submitted mainly to leaching purification. Following this concept, various graphite products, targeting different applications, can be obtained (Figure 13) (Liu et al. 2022). End-of-life graphite electrodes that used as conductors of electricity in electric arc furnaces are industrially recycled at some extend. There are no detailed available data on the processes that are followed however, the general recycling concept includes the steps of: (a) collection and cleaning, (b) shredding/thermal/chemical processing and (c) manufacturing of new electrodes through high temperature sintering (orientcarbongraphite.com; coidan.com).







Figure 13. Classification and treatment/reuse methods of graphite with different degrees of damage (Liu et al. 2022).

3. CONCLUSIONS

The progressively increasing demand for critical raw materials involved in the green energy transition of EU requires the exploitation of currently not exploited primary and secondary resources and simultaneously the optimization of the existing industrial production routes. **Table 7** briefly presents the main technological barriers and the respective innovative technologies in case of lithium, cobalt, nickel and graphite supply chains. These specific CRM are significant materials for the green energy transition.

Table 7. Generalized conclusions of technological barriers during the exploitation of primary and secondary resources of Li, Co, Ni and graphite and respective proposed innovative solutions.

	Primary production		Secondary	production
Matariala	Technological	Proposed	Technological	Proposed
Materials	barriers	innovations	barriers	innovations
Lithium	Intensive energy consumption for the calcination of a- spodumene and high consumption of sulfuric acid for the leaching of b-	Replacement of the a- spodumene calcination and b- spodumene leaching steps by a single roasting process using various additives such	In most of industrial recycling practices of LiBs, Li is lost in slag phase	The implementation of a "holistic" recycling process (based on the combination of pyro and hydro metallurgy) aiming to the

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	spodumene and Li- bearing micas Relatively low (in some cases <90%) Li recovery degrees	as Na ₂ CO ₃ , Na ₂ SO ₄ etc. aiming to an easily soluble (even using water) calcine		simultaneously recovery of Co, Ni, Li, Cu and graphite. The performance of LCA studies aiming to the investigation of the sustainability of the "holistic" approach taking into account the increase of batteries scarp availability in short term
Cobalt	In case of Ni-Co-Cu sulfide ores, a significant Co amount is lost during the flotation and the Ni matte formation steps. The exploitation of laterites via a pyrometallurgical process does not permit the extraction of cobalt	Optimization of the flotation process and hydrometallurgical processing of the secondary matte (in case of sulfides) The application of hydrometallurgy for the co-extraction of Ni- Co (and REEs) from nickeliferous laterites	Cobalt is already recovered by Li-ions batteries scrap The recovery of Co from slags and tailings is challenging due to its low concentration in these wastes and the complex processing required for their exploitation	The detailed examination, from a techno economical point of view, of the simultaneous processing of primary and secondary (slags and tailings) Co resources
Nickel	The recovery of Nickel by Ni-Co-Cu sulfide ores is well established	The identification of the optimum hydrometallurgical technique for the maximization of Ni from laterites via hydrometallurgy (taking into account the low yields in case of heap leaching and the intense physicochemical conditions required in case of pressure leaching)	Nickel is already recovered from various types of batteries, however specific barriers (i.e. recovery yield) have not been specified due to lack of published data The recycling of stainless steel is well established	
Graphite	The purification of natural graphite to obtain high purity grade material for green energy technologies is challenging (complex	The optimization of physical separation/beneficiati on technologies for purification	Graphite in spent Li- ion batteries is currently not recovered	The regeneration of the graphite contained in spent Li- ion batteries through a generalized concept of Co, Ni, Li and graphite co-recovery





methodology, use of environmentally hazardous acids) Low-grade graphite deposits can not be exploited

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