Trends and Status on the Current Battery Recycling Technologies – an Overview

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Firstly, we introduce the recycling challenges followed by the summary of recycling technologies with focus on hydrometallurgy, pyrometallurgy and bio-metallurgy. Several examples of combined processes and industrial recycling processes are presented in detail. Furthermore, the advantages, disadvantages and efficiencies of each process and as well the industrial recycling processes are discussed. Finally, the conclusions about the recycling process are summarized.

Trend und Status der Technologien zum Batterierecycling – ein Überblick

Einführend werden die Anforderungen an das Recycling beschrieben und die möglichen Technologien mit Schwerpunkt auf Hydro-, Pyro- und Bio-Metallurgie zusammengefasst. Verschiedene Beispiele für kombinierte Prozesse und industrielles Recycling werden detailliert dargelegt. Des Weiteren werden die Vor- und Nachteile sowie die Effizienz jedes einzelnen Prozesses einschließlich der industriellen Technologien diskutiert. Den Abschluss bildet eine Zusammenfassung der Recyclingtechnologien.

1 Introduction

In view of the worldwide increasing energy and raw material demand with continually shortening of the fossil resources, an increasing request towards environmental friendly recycling technologies is observed. The market for Lithium-ion batteries (LiBs) increases rapidly due to their uses as electrochemical power sources. They are widely used in mobile phones, laptops, videocameras and more recently in automotive applications [1, 2].

From the worldwide LiB consumption, 3400 metric tons of waste is estimated for the year 2020 just from electrical vehicles (EV) application, which is about four times the collection volume of LiBs waste from consumer electronics in 2012 [3]; while between 2015 and 2040 a 4 million metric tons of lithium-ion cells is predicted to be generated [4]. The use of cobalt in LIBs has grown from 700 tons to 1200 tons per year during the years 1995–2005, which is 25 % of the global cobalt demand [5]. London Metal Exchange [6] reports that the price of cobalt is then two times higher than of nickel and about 4 times more expensive than copper [6].

According to Dewulf et al. [5], if only recycled cobalt and nickel are used in the production of LiBs cathode active materials, 51.3 % savings can be achieved in natural resources, while 45.3 % in fossil fuel resources and 57.2 % in nuclear energy demand. Thus, a research increase is expected based on the optimization composition of new cathode materials that give higher energy and power density.

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2 Recycling challenges

An important issue in recycling of spent batteries represents the recovery of valuable components available in such wastes. As a result of a continuous market expansion and technological replacement, the generation of electrical and electronic waste becomes a challenge. Therefore, an increasing demand in developing new recycling technologies is required. The battery recycling market is mainly price driven and represents the key differentiating factor, which in a competitive market reduces profitability for battery recycling companies [7].

The main challenge which is hindering the industry in a long-term financial investment are the specialized processes and recycling plants required by market participants to develop specialized waste disposal services. Moreover, long-term recycling would complete the ecological benefits and the environmental laws, which will fulfill the handling requirements of the variety and volume of materials expected. As the market is still unexplored, the specific impacts and overall profitability of these investments are unknown and thereby create ambiguity and uncertainty about making such commitments [7].

Furthermore, the future battery chemistries which are under research and development will be employed for long-term recycling mainly for ecological benefits and for adherence to environmental laws. Phosphate or manganese based chemistries are still not standardized for recycling technologies, making the value of recycling challenging. However, the recycling of LiBs is expected to be one of the main sources of lithium supply, while more batteries are becoming available for recycling.

3 Battery recycling technologies

Over the last decade a research increase on efficient recovery methods has been observed. However, the expansion of the LiB recycling market will depend on the potential economic costs or revenues associated with material recovery from the continually increasing waste [8, 9]. Although there are a lot of research achievements developed on the recycling technologies of LIBs, most of them are based on hydro- and pyro-metallurgical chemistry [10–12].

Various battery recycling plants already exist on the market operate by e.g. Umicore, Sony/Sumitomo and Toxco Inc. Moreover, recycling represents a new concept and needs a good management of the collection and separation of the batteries. Some recycling processes combine pyro- and hydrometallurgical steps and often have integrated pre-treatment steps like pyrolysis or mechanical processing, i.e. crushing and material separation. Process examples for each category are given as follows.

3.1 Hydrometallurgical processes

The hydrometallurgical methods involve acid or base dissolution, solvent extraction, chemical precipitation, electrochemical methods and/or combination of these processes. Hydrometallurgical methods are used on the basis of their simplicity, environmentally friendliness (due to waste water and air emission minimization), adequate recovery of valuable metals with high purity and low energy requirements [13].

An environmentally friendly leaching process for the recovery of cobalt and lithium from the cathode active materials of spent lithium-ion batteries was proposed [13, 14]. A great interest was given to the recovery of lithium and cobalt from waste lithium-ion batteries of mobile phones. A systematic scientific study for developing an environmentally friendly recycling process for recovery of cobalt and lithium is proposed by Jha et al. [15] and Santos et al. [16].

In order to get basic information about the crushing and separation treatment of crushed products from spent LiBs a detailed chemical and mineralogical characterization was carried out [17, 18]. A mixture of organic DL-malic acid with H_2O_2 and citric acid with H_2O_2 were used to recover Co and Li from spent LiBs [13] while Kang et al. [19] dissolved the cobalt containing powder with H_2SO_4 and H_2O_2 to recover cobalt sulfate which with addition of oxalic acid was calcined to produce crystalline Co_3O_4 [20].

The feasibility of bioleaching for the solubilisation of metals from solid waste streams and by-products was evaluated [21]. Solubilisation of metals was achieved via sulphuric acid by sulphur oxidising bacteria. In order to achieve better solubilisation efficiency, the effects of inoculum, pH, supplemental ferrous iron, sulphur, and sodium chloride were investigated. They concluded that addition of ferrous iron and chloride ions does not enhance the metal solubilisation. In order to advance the spent LiBs recycling technologies, combined solvents were used due to their safety and simplicity. Further studies concerning nickel and cobalt electrowinning using a combination of hydrometallurgy with solvent extraction [22, 23] were reported, while a combined method of acid leaching and chemical precipitation aimed to recover Co and Li from spent LiBs [24].

A hydrometallurgical process is developed to recover valuable metals from the $LiNi_{0.8}Co_{0.15}Al_{0.05}O_2$ cathodes of spent lithium-ion batteries [25]. The effect of type of acid (H_2SO_4, HNO_3 and HCl), acid concentration, leaching time and leaching temperature were investigated to determine the most efficient conditions of dissolution. The preliminary results indicate that HCl provides higher leaching efficiency, while Co was selectively oxidized and recovered under the form of Co_2O_3 with 100 % recovery efficiency [25].

A lithium-ion battery recycling process was developed by Contestabile et al. [11] with the aim to characterize processes capable for treating the used batteries which will be further suitable for disposal. A combination of the electrochemical and hydrothermal methods were used to recover both Co and Ni from $LiCoO_2$ and $LiCo_xNi_{(1-x)}O_2$ in the lithium-ion and lithium-polymer batteries by Lupi et al. [26]. A good Co deposit was produced with a current efficiency of about 96 % and an energy consumption of 2.8 kWh/kg.

Freitas and Garcia discuss the electrochemical recycling of cobalt from cathodes of spent lithium-ion batteries [27, 28]. They used the electrochemical quartz crystal microbalance technique to study the electrodeposition of cobalt from spent LiB cathodes. A good electrochemical performance was obtained by re-using of a $Li_{1.2}Co_{0.13}Ni_{0.13}Mn_{0.54}O_2$ cathode material from spent LiBs [29]. A discharge capacity of 258.8 $mAhg^{-1}$ at the first cycle was obtained, while after 50 cycles the discharge capacities reached 225.1 $mAhg^{-1}$.

The works provided by Bernardes et al. [30], Xu et al. [31] and Bankole et al. [32] offer good reviews on the current recycling processes and technologies. According to Xu et al. [31] the recycling technologies must achieve the reduction in the volume of the scraps or cases and selective separation of the valuable components, with emphasis on the physical and chemical processes. These are considered the two main categories of processes employed on the lab- and industry scale to recycle various kinds of batteries.

Several companies in Germany like: Accurec in Mülheim an der Ruhr, Muldenhütten Recycling und Umwelttechnik GmbH in Freiberg, DK Recycling und Roheisen GmbH in Duisburg, Hoppecke Batterien GmbH & Co. KG in Brilon, Nirec Nickel Recycling and Redux GmbH in Dietzenbach, Nordische Quecksilber Rückgewinnung GmbH (NQR) in Lübeck and Varta in Hannover are dealing with the metallurgical recycling of batteries.

Furthermore, the company Batrec Industrie AG (Switzerland) is dealing mainly with the mechanical processing of LiBs. The batteries are crushed in CO_2 gas atmosphere, resulting in a subsequent material separation from where different material fractions are obtained and which represent feedstock materials for other processes.

One of the leading hydrometallurgical companies is Toxco Inc. (Canada) which was developed to recover valuable lithium products and other materials from all types and sizes of lithium batteries. This facility is also unique in its capability to recycle alkaline batteries to produce zinc and other metals and innovative due to its patented cryogenic process (-200 °C) for shredding and separation of the materials. Batteries like NiCd, NiFe, NiMH, Li-ion and Zn-Mn are recycled through the Inmetco process [33, 34]. A process of reducing iron ore in an ore/coal mixture on the hearth of a furnace is proposed. The rotary hearth furnace technology combines low material costs with simplicity and flexibility of operation. The unprocessed coals resulting from the Inmetco process, contains large amounts of volatiles, which consumes the energy needed for the reduction of the iron oxides. Nevertheless, the reduction of iron ore in an ore/coal mixture can be significantly improved by optimizing the experimental parameters.

Commercially available battery types (except Pb and Hg) are recycled at Accurec GmbH (Germany). Their aim is to develop innovative and cost efficient recycling technologies for Hybrid- and EV-batteries. Vacuum Thermal Recycling is used as the core technology and represents a self-developed technology. It is assumed that is an emission-free and safe technology, which enables the separation of the toxic metal gases hermetically from the environment. Due to its unique technology developed and applied, Accurec achieves a high recycling efficiency of 79 % for NiCd-batteries.

As part of the Berzelius Group, the Muldenhütten Recycling und Umwelttechnik GmbH in Freiberg (MRU) is bringing a significant contribution to battery recycling. With an average annual production of 55.000 tons of lead and lead alloys is the MRU not only one of the most modern, but also one of the productive secondary lead smelters in the country [35]. Furthermore, the MRU is placed on the second place with its own special waste incineration system and uses the waste heat from the combustion process for the crystallization of sodium sulfate as a heat carrier in the heating network operation, while electricity is generated through a turbine.

From the above, it is obvious that several recycling processes are available on the market and many are still under development. The available recycling plants demonstrate the compatibility of the hydrometallurgical methods with the environment, and make the process feasible for future development. Key factors, like separation, dissolution and extraction are under development and remain open and challenging for the new battery chemistries.

3.2 Pyrometallurgical processes

A specially recycling process dedicated to portable LiBs was developed by combining mechanical pretreatment with hydro- and pyrometallurgical process steps. Characterization and evaluation of the obtained material fractions were performed, while the focus of the project was the development of a selective pyrometallurgical treatment step in an electric arc furnace. The proof of feasibility of this facility aimed at the production of cobalt alloys and helps the melting of the fine fractions containing cobalt and lithium generated from the spent LiBs [36].

A combined pyro- and hydrometallurgical process for recycling of lithium-ion and NiMH batteries is developed by Umicore (Belgium and Sweden). The battery wastes are subjected to melting without any pretreatment, while nickel, cobalt and other metals are recovered. However, the Umicore process does not aim at the recycling of lithium and other organic materials and carbon are lost through the recycling process. With the future growing market of LiBs, increases the demand for lithium, while a potential increase application of LiBs in hybrid and/or electric vehicles is observed. In order to overcome these issues, some alternative competing recycling technologies need to be developed. Nevertheless, Umicore is also a producer of cathode materials for LiBs, thus their closed-loop recycling process aims at a recovery of cobalt and nickel in the form of $LiCoO_2$ and $Ni(OH)_2$.

In Feurs (France) Valdi sets for recovery of alkali-manganese and Zinc-carbon batteries as the central unit using the electric arc furnace treatment. In addition to the investment in Feurs operates Valdi in Le Palais another furnace submerged arc with a melting capacity of around 3 t/h. In the screening stage, the sorting of the materials is carried out in a partner company. Different fractions from the battery mixture are separated and collected and then forwarded to appropriate recycling companies [37].

Another research project designed to recycle metals from portable batteries uses a patented fine grinding technology to reduce the *black mass* solid inner core of alkaline batteries into a powder form. The aim of the project is to develop an economically viable treatment for extracting and reusing valuable metal concentrates from spent batteries. According to their ideas, after reducing the black mass to a powder, the material is suitable for treatment by different chemical and biological processes to extract the various metallic ions such as zinc, carbon and manganese [38].

The effect of impurities caused by a recycling process on the electrochemical performance of $Li[Ni_{0.33}Co_{0.33}Mn_{0.33}]O_2$ was investigated by Krüger et. al [39]. Fresh cathode material was synthesized from the recycled solutions using an industrial process. The recycled materials and thus the prepared solutions exhibited different aluminum impurities (stemming from the current collector) which had an impact on the formation of secondary particles during synthesis. They concluded that, the electrochemical performance of the recycled materials is influenced by the impurities.

Furthermore, as part of the CoLaBats initiative, Tecnalia, a spanish company operates pilot projects at battery recycling plants. Their aim is to provide new industrial processes for the recycling of the critical metals like cobalt and lanthanides. In addition to the critical metals, it is also hoped to improve the recycling of key economic metals nickel and lithium from waste batteries by improving the recycling efficiencies and metal purity from the existing recovery processes. The lithium-ion and NiMH batteries will represent the target of this initiative using specific ionic liquids for selectively extraction of the metals [40].

Sun et al. [41] used vacuum pyrolysis in combination with hydrometallurgy for recycling spent LiBs. Vacuum pyrolysis was considered as an alternative pretreatment method for recycling spent LIBs.

Recently, an acidic organic solvent was employed to separate $LiNi_{1/3}Co_{1/3}Mn_{1/3}O_2$ cathode material and aluminium foil from spent LiBs. It was found that nickel, cobalt and manganese mostly remained in the recovered cathodic active material and that the impurities (aluminium, iron, copper) were less than 2 wt%. After removing the impurities the cathode material was re-synthesized by a high temperature solid state reaction method [42].

At the current stage, since the recycling efficiency can only be achieved if the desired components are recovered, several improvements are still required. Furthermore it has to be mentioned that the economic efficiency of the process shows that the cost effectiveness is strongly dependent on the cobalt price, which is known to be very volatile. Finally, the pyrometallurgical methods have been associated with high energy consumption, high air emission of dioxins, chloride compounds and mercury, and therefore require strict standards for air filtration systems to avoid pollution. In order to be able to recycle new types of LiBs the pyrometallurgical process needs to be improved and/or adjusted to the chemical system.

3.3 Bio-Metallurgical Process

Compared with the aforementioned pyro- and hydrometallurgical processes the bio process is considered as having higher efficiency, low cost and being environmentally compatible [30, 45]. In the last years, the bio-metallurgical processes have been gradually replacing the hydrometallurgical processes because of their higher efficiency, lower cost and fewer industrial requirements [43–45].

However, the treatment period of the bio processes is longer and different types of bacteria are required. Among the different types of bacteria and inorganic chemical solutions, the acidithiobacillus ferrooxidans are extensively investigated for the battery waste treatment. Elemental sulfur and acidithiobacillus ferrooxidans ferrous ions were used to produce metabolites, where the H_2SO_4 and ferric ion were responsible to recover Li and Co from $LiCoO_2$ of LiBs. The results show that, the metabolites enhanced the dissolution of metals and that, the bio-dissolution of Co was faster than the one of Li [43, 45].

The bioleaching behaviour of the spent LiBs at pulp densities ranging from 1 % to 4 % was investigated with exploration of the process controls. A bioleaching efficiency decrease from 52 % to 10 % was observed for Co, while a decrease from 80 % to 37 % occurred to Li recovery. This decrease was attributed to a pulp density increase from 1 % to 4 %, with an extraction efficiency of 72 % for Co and 89 % for Li at 2 % pulp density [46].

In comparison with the chemical leaching and based on thermodynamic analyses, the bioleaching process has a good potential to recover metals from spent LiBs. A product layer diffusion model showing the best behaviour for Co and Li bio-recovery is presented [46]. Analysis of reasons for the decline of bioleaching efficiency of spent Zn-Mn batteries at high pulp densities was investigated [47]. The reason for the bioleaching efficiency decrease was attributed to the reduction of the bacteria activity when an increase pulp density was applied. This was confirmed by the extraction efficiency of Zn which dropped from 100 % at 1 % of pulp density to 29.9 % at 8 % of pulp density and to Mn from 94 % to only 2.5 %. Optimization of the leaching media brought a maximum extraction efficiency of approx. 100 % for Zn and 89 % for Mn at 4 % of pulp density.

The bioleaching of heavy metals from spent Ni-Cd and NiMH batteries using acidithiobacillus ferrooxidans was investigated [48]. In order to investigate the effects of initial pH, powder size and initial Fe^{3+} concentration on the percentage of metals recovered, a Box-Behnken design was used. Under optimization of the experimental conditions an 87 %, 67 %, and 93.7 % for Ni, Cd and Co, respectively recovery was obtained. It was confirmed that, acidithiobacillus ferrooxidans is an effective toxin resistant microorganism for bio-recovery of heavy metals.

Other groups [49] discuss the influence of silver ions on bioleaching of cobalt from spent LiBs. Using the same bacteria type (A. ferrooxidans) and a concentration of 0.02 g/L Ag^+ a 98.4 % Co recovery was obtained, while in the absence of Ag^+ a 43.1 % Co being observed. The proposed mechanism is based on catalytic interactions: firstly Ag^+ reacts with Co to form $AgCoO_2$, while an enhancement of the Co recovery using A. ferrooxidans and Ag^+ is explained [49].

Xia et al. [50] dealt with the recycling of spent Zn-Mn batteries via bio-metallurgy. Under optimum conditions a 96 % of Zn extraction was achieved within 24 h, while 60 % of Mn extraction was obtained using a biological mechanism with incubation for more than 7 days. For a better understanding of the mechanism of the leaching reaction a modified shrinking core model was proposed [51]. The recovery of Cr and Ni using bioleaching of dewatered metal-plating sludge was subjected, showing that pH = 1, pulp density of 9 g/l and initial Fe^{3+} concentration of 1 g/l are the optimum values and represent an recovery efficiency of 55.6 % for Cr and 58.2 % for Ni.

However, even if the bioleaching process shows good results with respect to the recovery efficiencies, the procedure needs to be more effective by improving the methods and reducing cost for culturing the bacteria. Nevertheless the bacteria need to be exchanged at a specific time intervals being unable to remain active in a concentrated waste solution. In order to bring the process to a commercial operation, the biometallurgy process remains challenging.

4 Conclusions

Effective and feasible recycling technologies with a complete life cycle analysis are under development and demonstrate the immediate need to develop comprehensive recycling solutions. More and more lithium based batteries are used in development of hybrid and/or electric vehicles. With the hybrid technology under continuous development, an increase of return flow of spent batteries is expected.

However, the batteries commercially available are not yet at a high enough technological level to meet the power requirements of hybrid and/or electric vehicles. Challenges remain with the development of novel concepts that may lead to advance batteries. Many innovative materials based on new chemistries, such as $LiFePO_4$, LiNiCoMnO, LiNiCoAlO, LiMnO, LiNiMnO, LiTiO, LiSi, LiSn, are investigated intensively with the aim of upgrading battery performance to automotive levels.

On the other hand, the economic and political implications of many countries worldwide are considered important in budgeting the funding, in order to stimulate the research and development of LiB technology. The development of innovative, cost effective, simple, flexible and environmental friendly solutions will challenge tomorrow's recycling processes.

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