

Technology Developments for Recycling of Lithium Battery Wastes

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Abstract

As new functional electronics are being developed fast, the commercialization rate of advanced battery as a power source proceeds rapidly. Lithium battery is satisfying the needs of high-energy source for its lightness and good electrochemical property. Especially lithium ion battery, adopted as a new power source for portable electronic equipments around the globe, has been mass-produced. Under the circumstance, the generation of lithium battery wastes is becoming a new environmental problem. In this paper, we are going to inspect technology developments for recycling of lithium battery wastes and scraps in domestic and foreign area, and to suggest how to treat domestic lithium battery wastes and scraps better.

Key words : battery recycling, lithium, cobalt, lithium ion battery

1. Introduction

Battery, used as a power source for electronic equipment, is a device transforming chemical energy into electric one and can be classified into two groups, primary and secondary. Primary batteries are those impossible to reuse once it is discharged such as manganese, alkaline, silver oxide, mercury oxide, and primary lithium battery. Secondary batteries can be reused with a numerous recharging like lead-acid, Ni-Cd, nickel metal hydride(Ni-MH) and rechargeable lithium ion battery[1].

Among these batteries, primary lithium battery is light and uses lithium for anode that helps obtain high voltage and energy density. For these merits, primary lithium battery has been widely used as a power source for memory back-up and LCD digital watches with a long cycle life, in various sizes and forms. Furthermore, as the use of portable electronic equipments such as notebook, cellular phone, and camcorder increases rapidly, demand of high performance rechargeable battery increases accordingly for their power source. Since portable electronic equipment requires light weight and long cycle life the most, most of secondary batteries in 1990's are now replaced by lithium ion

secondary battery (LIB) that exhibits a high energy density and light weight.

In case of Korea, the scale of LIB market is more than 40 million cells in 2000, and it is anticipated that the demand of the year 2003 would exceed 60 million cells as a 20% growth is expected each year. On the other hand, in case of Japan, the production of lithium ion batteries in 2000 is nearly 350 million cells, which dominates 85% of LIB worldwide market. The scale of world market for LIB is 680 million cells in 2001 and it is estimated to grow up to 1 billion cells till 2005 under current increasing rate[2].

Likewise, the increase in the consumption of high performance secondary batteries after 1990's resulted in the rapid growth of accumulated battery wastes and scraps. As for LIB with a relatively long cycle life, it is just an expendable supply lasts for about two years, equivalent to the charge-discharge of 1000 times. So the amount of battery wastes and scraps generated since mid 1990's has been gradually increased. Especially in 1998, the sales volume of mobile communication terminals was 250 million pieces and more than 40 million pieces of spent lithium batteries are estimated to be generated accordingly [3]. With this trend of rapidly increasing consumption rate for LIB which will occupy most of the high performance secondary battery in the near future, it is estimated spent LIBs were generated more than 500 tons in

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Korea in 2000, and also anticipated to increase in a large scale every year.

Though there are some differences in chemical compositions according to each type, LIB is composed of 15% organic solution and 7% plastic, and contains some valuable components like 5-20% Co and 5-7% Li. So the recycling of LIB wastes through recovering valuable metals like over 30 tons of Co and 3 tons of Li annually is necessary for securing available resources and keeping environmental treatment.

Technology developments for the recycling of LIB wastes are under trials in some lab-scale projects in Korea, but fairly great deal of time and work are demanded to raise the research scale for the practical use in the future [4]. The recycling process includes physical or mechanical treatments as well as chemical treatments for the separation and recovery of valuable metals.

Even the advanced country like Japan recycling battery wastes and scraps produced in bulk, commercialized recycling skills are still insignificant due to the short period of using lithium batteries, less than 20 years.

In this time, the necessity of recycling technology is gathering strength for the lithium battery wastes and scraps which are to be generated in a large amount in the near future, the object of this paper is to inspect current situations around lithium battery wastes and scraps focusing on the recycling technology development.

2. Technical development trend for recycling lithium battery wastes

2.1. Recycling trend of primary lithium battery wastes

Primary lithium battery is widely being used as a power source for memory backup, military communication equipment, camera and etc. Though the shapes and components are different to each manufacturer and type, generally a battery is composed of electrode materials such as Li, Mn, V, C and S, electrolytes such as polycarbonate, 1,2dimethoxyethane, thionyl chloride(SOCl_2), and salts such as LiClO_4 , LiCF_3SO_3 , LiAlCl_4 , LiPF_6 and LiBF_4 . Real commercialized ones among primary lithium batteries at this moment are MASUSHIDA, SANYO, HIDACHI and TOSHIBA in Japan, RAYOVAC in

America and fluoro-graphite($(\text{CF})_n$)-lithium battery and manganese oxide(MnO_2)-lithium battery developed by the SAFT in France.

Collecting these battery wastes and scraps and recycling them have a lot of difficulties due to their variety of use, structure and composition. Especially the danger of explosion exists in disassembling process because of the metallic lithium used for anode material, and sometimes it contains abundant metals environmentally harmful. So the recycling technology of primary lithium battery wastes is being studied mainly to the direction of pursuing the safe recovery of explosive lithium in disassembling process, and the recovery of valuable metals.

Followings are the process and technologies for the safe recovery of metallic lithium when disassembling primary lithium battery wastes, introduced from in/outside of the country.

① Korea

Up to now, there have been some recycling process for spent manganese and alkali-manganese battery among other primary batteries, recovering valuable metals like manganese, iron and zinc in the form of ferro-metal, as well as other studies in progress like utilizing Induction [1] or Plasma [5] at a high temperature of 1200~1400°C for the separation and recovery of mercury from a mercury battery. But these researches are still very insignificant compare to the amount of battery wastes and scraps growing rapidly every day.

② Japan

As for a recycling technology being carried out in Japan for primary lithium batteries, a separation process of valuable metals like iron and copper from MnO_2 -Li battery is known. Which is; after calcination of battery wastes at 500-750°C, crush the calcined material[6] and apply self-assortment to separate iron and copper. Also K. Soichiro[7], in the US Patent 5,491,037, introduced a recovery process; separating electrolyte through washing with organic solvent and distilling the disassembled battery scraps, and recovering lithium hydroxide from electrode material.

Furthermore, M Schumit[8] introduced a recovery process of metallic lithium from lithium battery wastes, so it can be reused for an anode material. That is; from the battery wastes composed of anode unit, electrolyte and cathode unit, first, electrode packages

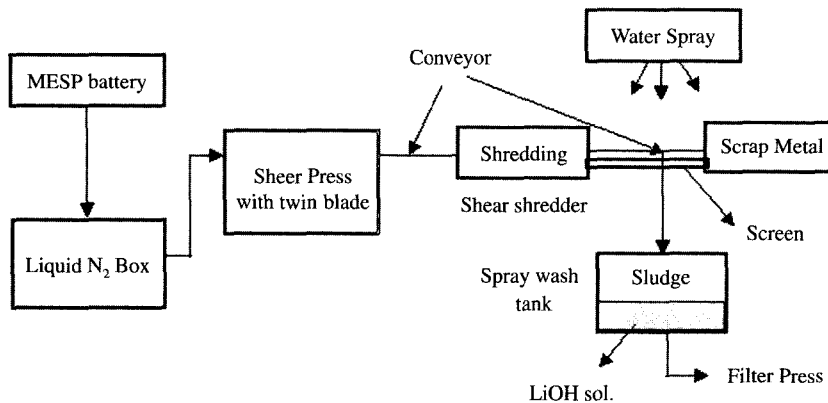


Fig. 1. Recycling process of primary lithium battery wastes [9].

are extracted directly with an organic agent. Secondly, anode unit has to be separated mechanically from electrode packages, and the residual electrode packages are crushed. Then the resulting materials obtained by sieving are treated in a high temperature of 300~700°C. Then the electrode material reusable for anode material is recovered finally through a series of process above.

③ Europe and America

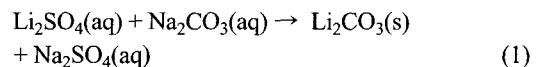
In the past, battery wastes have been disused by filling-up, incineration, denotation and neutralization. But all these methods are not useful for a long-term handling and also very dangerous. It can even cause secondary environmental problems. So, to recover valuable components from lithium battery wastes safely, the TOXCO in Canada has started a recycling process since 1993[9].

So to speak, explosive and inflammable battery wastes were crushed after freezing process with liquid nitrogen of -195°C. Highly concentrated lithium carbonate (Li_2CO_3) was recovered from it. And the separation technology of valuable metals like iron and copper completed the recycling process.

Meanwhile in New Zealand, the PLL(Pacific Lithium Limited, founded in 1994) developed a recycling technology by applying HPS(Hybrid Purification System) and made the direct recovery of high purity Li_2CO_3 reusable as a raw material for lithium batteries [10]. Moreover in 1996, recognizing each company's process newly, TOXCO and PLL have started a mutual cooperation, developing a unique blend technology.

The TOXCO provided highly concentrated lithium salt and the PLL converted it into pure Li_2CO_3 . Some of the recovered lithium salt was used as a raw material for producing metallic lithium, and some as an electrolyte of battery. Like this, TOXCO sent the compound of lithium salt collected from lithium battery wastes to PLL, where Li_2CO_3 was successfully recovered from it.

PLL is currently running a factory in New Zealand capable of generating 250 tons of technical graded- Li_2CO_3 for recycling lithium battery wastes. Lithium salt is provided in 30-40 % dehydrated condition from TOXCO, and the pH of its solution when dissolved in water is alkali, about 11. The recovery process operated in PLL is controlled by the condition of alkali. It is noticeable PLL used Na_2CO_3 , the source of CO_3^{2-} , instead of CO_2 when making Li_2CO_3 from lithium salt and produced 98.5 % pure Li_2CO_3 using the following reaction.



Also the TOXCO developed a recycling process in 1998, applying various chemical technologies to the large capacity lithium batteries (LiSOCH_2) wasted from munitions. Through this, 570 lbs of lithium battery wastes (MESP) and 2,600,000 lbs of LiSOCH_2 were recycled and especially 70% of the battery wastes were successfully recovered in the form of Li, Al, Ni and stainless (steel). As stated above, liquid nitrogen was adopted for disassembling battery wastes and

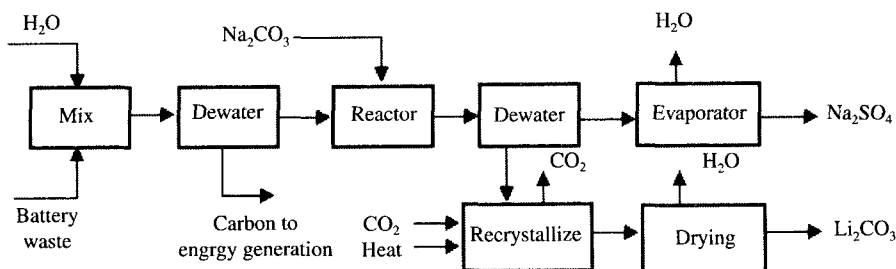


Fig. 2. Recovery process of Li_2CO_3 from primary lithium battery wastes [10].

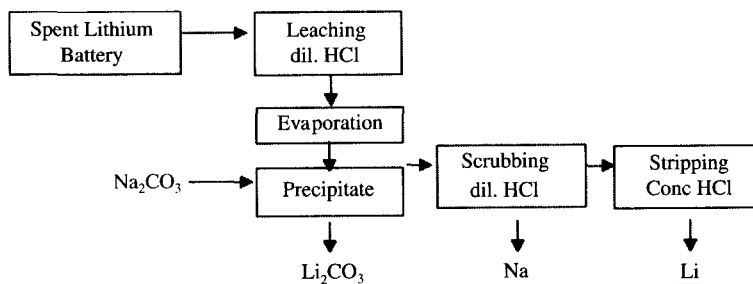


Fig. 3. Selective recovery process of lithium from lithium battery wastes using precipitation-solvent extraction [10].

scraps to prohibit rapid oxidation of lithium.

Since the lithium ion in anode exists in the form of $\text{Mn}^{3+}\text{O}_2(\text{Li})$ combined with MnO_2 from cathode, it can form insoluble Li_2MnO_3 by calcination. Thus dry process is not suitable for the selective recovery of lithium from lithium battery wastes. Therefore, considering lithium is an alkali metal, precipitation and solvent extraction using a weak acid is advisable for the selective recovery of lithium. Fig. 3 shows this process using leaching agent (HCl).

In Europe, a recovery process was suggested by B. Didlier[11] for the recycling of primary lithium battery wastes. First, lithium alkoxide is recovered by washing the separated lithium components with alcohol (1-Pentanol) after disassembling. Secondly, LiMnO_2 is transformed from lithium alkoxide by the reaction with manganese cathodic materials. As well, other materials are separated in solid and liquid phase by filtering.

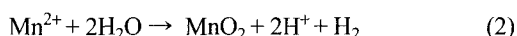
Meanwhile, a recycling process reported by M. Contestabilein[12] from Italy in 1999, could be roughly divided into several steps such as; crushing and riddling, hydrolysis for lithium components, precipitation of lithium salt, separation of organic agent and recovery of electrode materials. To introduce

this process briefly, first in the crushing and riddling process, battery wastes are crushed with a shredder or hammer mill and cut with a cutter. The battery wastes need to be frozen and before crushing with liquid nitrogen to reduce the risk of explosion caused by metallic lithium. There are steel casing, cathode assembly (MnO_2/C on nickel-plated steel grids) made of heavy metal components, and light residual active materials (metallic lithium, PP separator) in the crushed products.

Among these, steel casing is processed alone for recovery, and metal components and residual active materials are separated by a mechanical separation. A separated lithium component is recovered in aqueous LiOH by the hydrolysis process using bi-phase system. As injecting CO_2 gas into LiOH solution, through the first precipitation forming insoluble Li_2CO_3 , and the second reaction between the lithium ion remained in the solution and carbonate ion(CO_3^{2-}), lithium can be recovered in a high purity Li_2CO_3 .

In the recovery process of lithium, an organic agent, mainly ether derivatives, is dissolved in the alcohol phase and remains behind when hydrolyzing. A distillation separating organic solvent with a low boiling point is

applied to reuse it in a pure state. In the mean time, after having manganese cathodic material reduced to Mn^{2+} by dissolving in sulfuric solution, it can be recovered as MnO_2 by an electrochemical method like EMD (electrolytic manganese dioxide). At this point, nickel component contained in active material should not be dissolved in the sulfuric solution, and the ordinary electrolytic condition for the EMD recovery is $MnSO_4$ 80~180 g/L, H_2SO_4 50~100 g/L. The reaction of MnO_2 recovery from reduced Mn^{2+} by EMD process is described below.



MnO_2 recovered by this reaction is high purity so it can be used as an electrode material in battery production without any extra refinement process.

Furthermore in France, a recycling process was contrived for the large capacity lithium battery wastes used for munitions, with BDT (Battery Disposal Technology) incineration system. There are several steps for this process to recover active materials like metallic lithium and thionyl chloride ($SOCl_2$), a catholyte. First, the disassembled metallic lithium by hammer milling is hydrolyzed in a room with a ventilation capacity of 5,000 ft³/min. Then scrubbing the exhaust gas with NaOH solution is applied to eliminate acid gas and mist. Also there is a chemical treatment tank for inactivating toxic material like $SOCl_2$ in this system. So far, the BDT system has been evaluated for the most suitable process in handling active materials like metallic lithium, lithium alloy and anode scrap, generated as by-products in the manufacture or recycling of lithium battery wastes [13].

And in 1995, the Pira GmbH in Germany developed the hydrometallurgical process (BATENUS process) grafting electrochemistry and membrane technology, and used it to regenerate valuable metals like Li and Mn from primary lithium battery wastes [14].

2.2. Current developments of recycling technology for secondary lithium battery wastes

Secondary lithium batteries are widely being used as the power source for mobile phones, computers, video cameras and digital cameras. There is lithium ion polymer battery adopted $LiCoO_2$ for cathodic active material, currently in use.

The shape of a secondary lithium ion battery depends on its manufacturer and usage. Observing its composing elements, cathode unit stays with $LiCoO_2$ powder, electron-conducting carbon like acetylene black and organic binder like PVDF over the thin Al foil, and anode unit is with graphite(C), electron-conducting carbon and organic binder over the thin Cu foil. For an electrolyte, ethylenecarbonate(EC) or diethylcarbonate(DEC) is used alone or compounded, in which salts like $LiPF_6$ and $LiBF_4$ are dissolved.

As portable electronic equipments get smaller and lighter recently with technology development, rechargeable lithium batteries are also going through a big change in structure and shape, focusing on the high energy density and light weight. Especially lithium cell has changed a lot in its external shape, from cylinder type to rectangular.

For it hasn't been long since LIB came into the market, the amount of collected LIB wastes is not so much yet compare to the amount currently being used. But due to the great deal of recent supply, the generation of battery wastes is anticipated to grow greatly. Therefore these days, unlike past years, more researches are being commenced slowly on handling or recycling secondary lithium battery wastes growing inside and outside of the country. Table 1 shows the typical composition of LIB as the result of analysis on the collected spent LIB in Korea.

In case of Japan, NIKKO METAL, TOSHIBA and SUMITOMO METAL are testing technologies in pilot plant scale, separating harmful metals and recovering

Table 1. Typical chemical composition of secondary lithium ion battery.

LiCoO ₂ (cathode)	carbon (anode/electron- conducting)	EC/DEC (electrolyte)	LiPF ₆ (electrolyte salt)	Plastics (Outer case)	Ni/steel (Inner case, Tap, Cap)	Al (Vent)	PP/PE (Separator)
20~25	15~20	10~15	1	10~15	10~20	0.5	2.0

On the basis of 1996's Sanyo cell.

valuable metals by mechanical or hydrometallurgical steps, and they plan to commercialize them in 2002 [15].

The recycling process undergoing in Japan mostly adapted past technologies characterized by the separation of electrode materials with crushing, leaching, refinement and recovery process. This system not only costs a lot due to the excessive crushing of battery wastes, but also requires an extra refinement for Co recovery with a chemical treatment to get rid of some metal impurities like Fe, Ni and Cu in the electrode materials.

Recovering Co from cathode material is the most critical factor of economical efficiency in the process, as well as the cost of handling plastics or recovering other valuable materials like Li, Ni, Cu and Fe are taken into consideration. Generally, recovery process of valuable metals is composed of separation, concentration and recovery technology, which are varying from the targeted recovery metal. Separation process is a step of sorting out, disassembling, cutting and crushing

battery wastes, concentration is of separating metals from nonmetallic crushed scraps and concentrating them, and purification is of recovering each metals.

Following introductions are a few cases of technical developments for the recovery or separation of valuable metals from LIB wastes, mostly on the basis of the technologies described above.

① Korea

Currently in Korea, only the recycling research team in KIGAM has been promoting researches on the commercialization of recycling process for LIB wastes. Their studies can be divided into two major parts. One is the physical process separating electrode materials selectively, and the other is the chemical process maximizing the recovery rate of valuable metal, cobalt. Physical process focuses on the separation of LiCoO_2 from the LIB wastes. And chemical process consists of leaching and ion separation for Co recovery.

The mechanical/chemical process in the Fig. 4

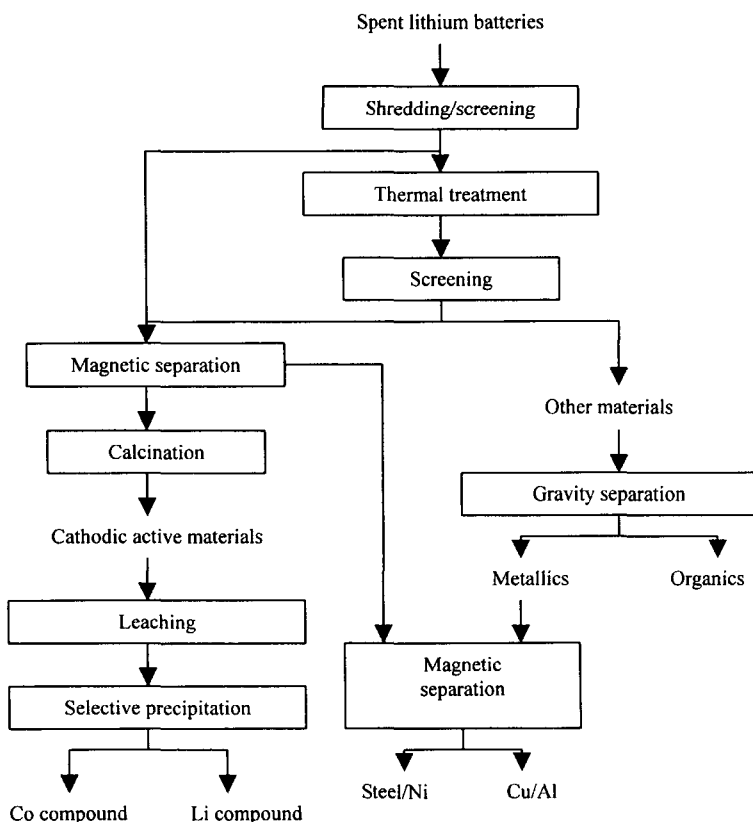


Fig. 4. Recycling lithium ion battery wastes by mechanical/chemical process

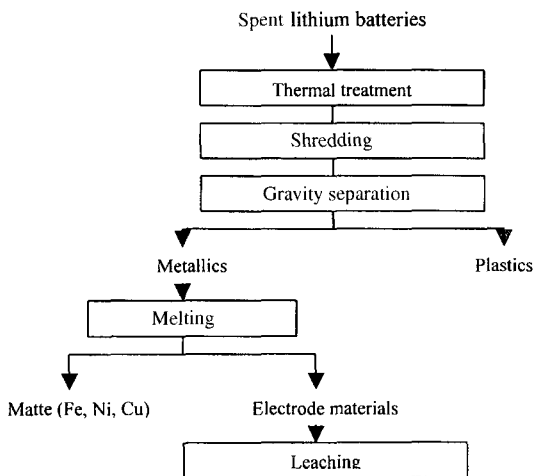


Fig. 5. Recycling of lithium ion battery wastes with high temperature/chemical process

roughly explains the process concentrating cobalt and separating cathodic material from LIB wastes. It is completed with a series of thermal treatment, shredding, gravity separation, and magnetic separation [16].

Cobalt can be recovered from the electrode materials separately concentrated, by leaching and selective precipitation. Generally there are neutralization, precipitation, solvent extraction, chemical precipitation, cementation, ion exchange, electrolysis extraction and etc. in refinement technologies that lead to the recovery of valuable metals in the solution as metal (direct reduction) or in the form of compound (insoluble precipitation).

The process in the Fig. 5 can separate electrode materials from other metals with a high temperature without any complicated physical separation. Because the cathodic material concentrated of valuable metals remains in the form of an oxide like LiCoO_2 , it doesn't melt at high temperature while as other metal components burn or melt under such condition. Thus cobalt can be concentrated in the form of an oxide, slag [17]. According to the temperature and atmosphere controlled, phase changes arise such as LiCoO_2 into the slag form, and metals like steel, copper into the matte form. Valuable metals can be separated by the phase separation in molten-state, because of the gravity difference between matte(5.5-7) and slag(1.5-3).

Co can be recovered with a chemical process

mentioned above. The leaching efficiency of Co is very low as 40% with a general acidic leaching, where Co exists in the form of trivalent. Hence the leaching efficiency can be improved by adding a reducing agent together with leachant, and especially the combination of $\text{H}_2\text{SO}_4\text{--H}_2\text{O}_2$ is known as the best industrially.

② Japan

Though various experimental researches have been tried out on recovering valuable metals from LIB wastes so far, commercial technologies are still in trial step and establishing practical process is desperately needed.

But, the JAPAN CHEMICAL INDUSTRY, Ltd. has tried a process for the recovery of valuable metals like cobalt and lithium from LIB wastes. First, a leachate containing lithium and cobalt is separated from other materials, using a compound has the phosphorus in it like bis (1,1,3,3-tetramethylbutyl) phosphoric acid derivatives as a leachant. Then this leachate is taken under a reverse extraction process with strong acid and finally Li and Co are recovered in the form of salts.

Also this company earned a patent with a recovery process of valuable transitional metals like Co and Ni from LIB wastes. First, a leachate is separated by a aqueous acid or H_2O_2 only or a mixed solution, and this separated solution gets contacted with an organic agent. Then, through the reaction between extracted organic agent and mineral products, Co and Ni are recovered selectively [18].

Apart from this, JAPAN ENERGY, Ltd. [19] invented a recovery/recycling process. First, cathode scraps of lithium ion cell are dissolved in acetic acid and LiOH is added into the residual solution containing cobalt, nickel or manganese acetate and lithium acetate. Then valuable materials like cobalt, nickel, manganese, and lithium are recovered in the form of hydroxide. TOSHIBA also presented a separation process of Cu and Al current collector and electrode materials by gravity separation, which is accomplished by the dissolution of organic binder after cathode scraps soaked into the acid solution or by heating under reductive circumstance.

JAPAN MAGNETIC SEPARATION, Ltd. [20] earned a patent for a process recovering valuable materials like Co, Ni and Mn in precipitates from the metal oxide containing alkali metals. For this, valuable

metals were induced by the reducing agents like FeSi, Si, CaSi and SiC in molten state.

And the ASAKARIKEN INDUSTRY, Ltd. [21] carried out a project to solve the problem occurred in the previous system of recovering cobalt from LIB wastes, that the installation cost increases sharply because of a lot of HF gas generated in calcining organic binder. In other words, after dissolving the separated active material with an inorganic acid, useless parts were eliminated by filtering. And noble metals with the standard potential of $-2.4 \sim -0.6$ V were added into the electrolyte to recover the reduced cobalt. Also the noble metals added during the recovery process were washed off with alkali. By repeating these elimination processes, cobalt could be recovered with high purity and a relatively large specific surface area. Recently, a highly effective and economical way of recovering cathode material was suggested, enabling the metal compound in battery wastes to be reused directly for the raw electrode materials. This method recovering cathode material from battery wastes directly is achieved by the continuation of following two steps, which is very peculiar. First, electrode materials are separated by heating battery wastes in the gas containing oxygen at $300 \sim 500^\circ\text{C}$, and recovered in powder. Secondly, they are heated again in the same gas at $500 \sim 600^\circ\text{C}$ to incinerate other volatile parts like organic binder and electrolyte. This method of recovering anode materials was developed by the TAMA chemical industry [22].

On the other hand, K. Mitsuhiro [23] chose to disassemble batteries first and got electrode materials deposited in a solution before heating, because it is dangerous to burn up charged battery directly with high heat. As for another case, the recent technology released by the TOHOKU NATIONAL INDUSTRIAL INSTITUTE in Japan [24], had 95~97 % Li and Co deposited by adding inorganic acid like sulfurous acid (H_2SO_3), hydroxylamine hydrochloride ($\text{NH}_2\text{OH}\cdot\text{HCl}$) and HCl into LiCoO_2 , the cathode material. From this deposit, Co is recovered by solvent extraction using a leachant based on organic phosphoric acid like dissolving D2EHPA and PC-88A into kerosene. In this step, pH 6~7 maximizes the extraction efficiency of Co. Since the distribution coefficient among metals is a critical variable in such solvent extraction, balancing

up the extractor type, phase ratio and pH of solution is important.

③ Europe and America

Europe had a very short period of using secondary lithium battery and studies are not in progress actively yet. But as for an experimental achievement, P. Zhang [24] in America and M. Contestabile [25] in Italy published their result of a recycling research for cylinder type LIB wastes like following.

Crush lithium ion battery wastes and divide them into steel casing and electrode materials. Extract this with N-methylpyrrolidone at 100°C , and filter off the extracted solution to remove Al, Cu scraps. There are LiCoO_2 and electron-conducting carbon contained in the filter cake. This mixture is immersed in 4.0 M HCl solution at 80°C for 1h to extract and then separate Co^{2+} from electron-conducting carbon. The concentration of Li and Co were verified with chemical analysis at 6.5 g/L, 36.2 g/L each. And Li/Co ratio appeared as 5.56, smaller than theoretical number of 8.53. It means Co precipitation rate is higher than Li's when using HCl. The cobalt ion is finally recovered as $\text{Co}(\text{OH})_2$ by the neutralization with NaOH injected. The whole process of this technology is shown in the Fig. 6.

Differently from above, recovery of Co in acid type without neutralization was developed by M.L. Lain at the AEA Technology Battery, Co. in England [26]. First, battery wastes are crushed mechanically under

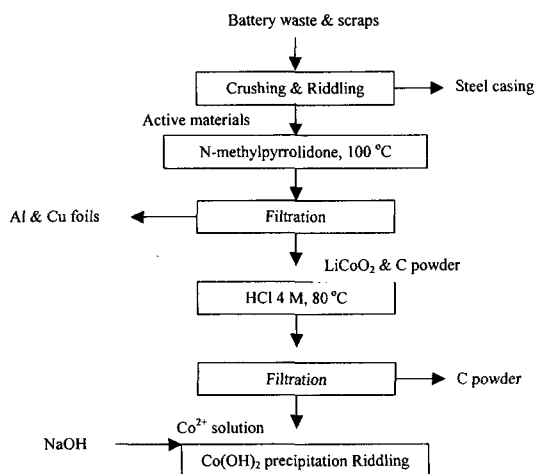


Fig. 6. Recovery process of Co from cylinder type lithium ion battery wastes.

inert dry air. Secondly, the electrolyte is deposited with an organic agent, and the rest of electrode materials can be recovered in powder containing LiCoO_2 and carbon conducting material. From this powder, Co can be recovered as Co^{2+} by electrochemical process, and finally as CoO again, by reacting with O. And Li can also be recovered as LiOH under the same procedure.

3. Conclusion

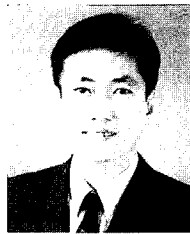
Recycling of lithium battery wastes and scraps is a newly arising social problem. Some of the technical developments are being carried out partially, as well as the government and industrials are paying attention to it more and more. Though a perfect recycling process has not been found yet, many scientists are working on establishing new technologies. So it is anticipated that a new effective and practical method of recycling lithium battery wastes will emerge soon in the near future. But it is considered that more attentions are desperately needed from government in supporting policies and research developments, and also from related industrials in putting continuous interests and investment.

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種 類	內 容
論 說	提案, 意見, 批判, 時評
展望, 解説	現況과 將來의 견해, 研究 技術의 綜合解説, Review
技 術 報 告	實際의 試驗, 調査의 報告
技術, 行政情報	價値있는 技術, 行政情報를 간결히 解説하고, comment를 붙인다.
見 聞 記	國際會義의 報告, 國內外的 研究 幾關의 見學記 등
書 評	
談 話 室	會員相互의 情報交換, 會員 自由스러운 말, 隨霜 등
Group 紹介	企業, 研究幾關, 大學 등의 紹介
研究論文	Original 研究論文으로 本 學會의 會誌에 掲載하는 것이 適當하다고 보여지는 것

수시로 원고를 접수하오니 많은 투고를 바랍니다.