

Battery Recycling Technologies: Recycling Waste Lithium Ion Batteries with the Impact on the Environment In-View

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Abstract

This survey is to review the advancement recorded so far in the lithium-ion battery recycling technologies in compliance with environmental laws. Amongst many of the technologies used to date, the best is the use of simple but scale-up dissolution technology involving non-toxic suitable organic solvents that can effectively dissolve toxic binder, PVdF in battery to avoid much pollution. Pollution through the hydrolysis of LiPF₆ from the lithium-ion battery can also be minimized through conversion to useful compounds instead of using



virgin materials for the synthesis. More environmentally friendly recycling technologies are still needed to meet the demands for materials, for scale-up processes and in compliance with environmental laws.

Keywords: Environmental pollution, Waste lithium-ion battery, Recycling, Technologies



1. Introduction

The environmental pollution caused by the valuable chemical components such as cobalt. copper, lithium, mixture of organic electrolyte and salts of either low quality or spent lithium-ion batteries (LiBs) deposited into the environments necessitates responsive recovery technologies. Since Sony made the first commercial lithium-ion cell in 1991, it has been accorded more attention being superior to other types of batteries in terms of energy density, which is a critical parameter for portable electronics as well as hybrid and electric vehicles. Lithium ion batteries are the systems preferred as electrochemical power sources in portable batteries segment such as mobile telephones, personal computers, video-cameras and other modern-life appliances as well as in vehicles with electric drive due to its favorable characteristics (Contestabile et al., 2001; Gaines, 2011; Nan et al., 2005; Wang et al., 2011). As LiBs progressively dominate, the amounts of valuable chemical components that will be deposited will be proportional to the number of LiBs used after their life-span has expired. Therefore, recycling that constitutes the most generally acceptable environmentally friendly method of managing these wastes must be taken serious, to minimize environmental toxicity, for economic gains and reduction in dependence on foreign resources or on virgin materials for productions in the industry as well as for sustainability of the natural resources (Contestabile et al., 1999; Dewulf, et al., 2010; Graham-Rowe, 2010; Hitachi, 2011; Kumar, 2011; Wang et al., 2011). The methods could be on the laboratory scale, industrial or commercial scale level. These as-recovered metals or their respective compounds (cobalt, lithium, manganese, and nickel) are not only valuable metals but are alternative precursors for new batteries formulations. Thus, several attempts have been made to review the old processes considered green and non-green chemistries to either improve on the existing ones or propose new recovery processes that are considered simple and of industrial-scale (Kondás et al, 2006; Nan et al., 2005). However, the cells used in cell phones and laptops are not fully recycled and consequently causing unsustainable open loop in the industrial cycle (Wang et al., 2011).

Although according to the U.S. government, spent LiBs have been classified as non-environmentally hazardous wastes or rather call "green batteries" and thus safe for disposal in the normal municipal waste stream unlike other battery chemistries that contain Cd, Pb or Hg, the presence of flammable and toxic elements or compounds may make their safe disposal to become a serious problem. For instance, the mixture of dimethyl carbonate (DMC) and ethylene carbonate (EC) used as solvent is flammable, while the polyvinylidene fluoride (PVDF) used as binder irrespective of its percentage in the battery formulation is toxic when burns consequent to the release of gaseous HF. Besides, the NMP commonly used as a solvent for the electrode active materials (cathode and anode) fabrication during slurry preparation has been reported as toxic and therefore environmentally incompatible (Alfonso et al., 2004; Castillo et al., 2002; Mitchell, 2006; Robert, 2000; Roth and Orendorff, 2012; Wang et al., 2011). As there is a general saying and belief that "health is wealth", similarly, "healthy environment is a wealthy environment". Therefore, recycling is of great importance to save our immediate environment and for waste management sustainability.



2. Structural Composition of Lithium-Ion Battery

All batteries consist of cathode, anode, electrolyte mixture and separator. The cathode has the aluminium foils coated with a mixture of the active material, $LiCoO_2$ or $LiNi_{1/3}Mn_{1/3}Co_{1/3}O_2$ depending on the type, PVdF or PTFE, carbon graphite, while the anode is a copper foil coated with blended slurry of carbon graphite and PVdF or PTFE.

The electrolyte mixture consists of the water-proned electrolyte salt, LiPF₆ and organic solvents dissolved in varying ratios such as 1:1:1 (v/v) for 1M LiPF₆, dimethyl carbonate (DMC) and ethylene carbonate (EC) respectively. In addition, other lithium salts used for lithium-ion battery are LiAsF₆, LiClO₄, and LiBF₄, while the organic solvents among others are propylenecarbonate with dimethoxyethane (PC–DME), γ –butyrolactone with tetrahydrofuran (BL–THF) and dioxolane (1, 3-D) according to Contestabile et al (1999).

The separator is a non-conductor that separates the two electrodes from each other. The structure of a cylindrical lithium-ion battery according to Nishi (2001) is represented in Figure 1.



Figure 1. The structure of a cylindrical lithium-ion battery

2.1 The Chemical Reaction of a Typical Lithium-Ion Battery



Lithium ions move from the negative electrode to the positive electrode during the discharge process through the nonaqueous electrolyte and separator diaphragm and then undergo reversible reaction when charging (Figure 2). The ionic chemical reactions are shown in equations 1-3.

The cathodic half reaction:

Discharge

$$LiMO_2 \longrightarrow Li_{1-x}MO_2 + xLi^+ + xe^-$$
 (1)
Charge
The anodic half reaction:
 $Discharge$
 $xLi^+ + xe^- + 6C \longrightarrow Li_xC_6$ (2)
The everall reaction:

The overall reaction:

Discharge

$$LiMO_2 + 6C \longrightarrow Li_{1-x}MO_2 + Li_xC_6$$
 (3)
Charge

Where M represents Mn, Ni or Co depending on the cathode active material.







3. Processes for Recovery of Lithium Ion Batteries

According to Xu et al (2008), recycling technologies, irrespective of the processes must amongst others achieve the reduction in the volume of the scraps or cases, selective separation of the valuable components. The physical and chemical processes are generally the two categories of processes employed in the laboratory and industry to recycle all kinds of batteries.

3.1 Physical Processes

The physical processes are generally dissolution, manual or mechanical separation and pyrolysis. For instance, Contestabile et al (1999) and Bankole and Lei (2013) extracted the electrolyte solution into organic solvents such as ethanol or iso-butylalcohol/water after manually or mechanical dismantling LiBs and this enhanced reduction in the environmental pollution caused by the hydrolysis of electrolyte salt, LiPF₆ and also the toxic electrolyte mixture. Interestingly, innovative conversion of LiPF₆ to useful compound such as Li_2SiF_6 was achieved for the first time (Bankole and Lei, 2013).

3.1.1 Hydrometallurgical Process

In hydrometallurgical method, mechanical separation was employed as pretreatment by subjecting LiBs to skinning, crushing removing of crust, sieving and separation of both anode and cathode material for easy recovery of the valuable components of the batteries (Xu et al., 2008; Zhou et al., 2010). However, safety precautions are required due to flammability of the electrolyte mixture (Roth and Orendorff, 2012). Although the stress in manual separation will

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be reduced, the components of the batteries may not be fully separated from one another due to the structural arrangement of the LiBs (Xu et al., 2008).

3.1.2 Dissolution Process

This process recently dominates and enhances effectiveness with maximum recovery of valuable components from batteries. The adhesive force from the PVdF holding the electrode active materials (anode and cathode) unto the current collectors is weakened. Therefore, the choice of suitable organic solvents capable of dissolving the binder, PVdF or PTFE becomes very important during recovery processes. Among these suitable solvents that have already been tested and found effective are N, N-dimethylformamide (DMF), N, N-dimethyl acetamide (DMAC), N-methylpyrrolidone (NMP) and dimethylsulfoxide (DMSO) with their order of effectiveness in dissolving the adhesive investigated. For instance, LiCoO₂ was recovered from LiBs with the solubility of PVdF in the first three solvents recorded as DMAC > DMF > NMP (Zhou et al., 2010). N-methylpyrrolidone separated both LiMn_{1/3}Ni_{1/3}Co_{1/3}O₂ and LiCoO₂, from LiBs at 40 °C for 15 minutes and at 100 °C for 1 h, respectively (Contestabile et al., 2001; Li et al., 2007). Although the powders were effectively recovered, the cost of buying 1L of NMP which is about \$ 207.90 makes its application not cost-effective and suitable for a large scale recovery operation (Sigma-Aldrich 2011; Xu et al., 2008). Among all these solvents, DMSO used at 60 °C for 85 minutes could be the most suitable for its cheapness (\$ 144.54/ L), non-toxicity and environmental safety (MTI Corporation, 2009; Sigma-Aldrich 2011). Moreover, the clean and shiny current collectors (Aluminium foils) obtained after the separation could be used for other applications in the laboratory and industries. The flow-sheet for the recycling of LiBs by dissolution method is shown in Figure 3.



Figure 3. Flow sheet for the recovery of valuable components from LiBs by dissolution process



3.1.3 Pyrolysis or Pyrometallurgical Process

The name comes from the two words "pyro" and "lysis" meaning "fire" and "decomposition", respectively. Therefore, this process decomposes the components of the LIBs by heating to high temperatures under heat and pressure. Pyrometallurgical process has been associated with high air emission of dioxins, chloride compounds and mercury, and therefore requires strict standard for air filtration systems to avoid pollution. It was used as pre-treatment for waste batteries before leaching process, especially to remove Hg, papers and plastics under a controlled atmosphere (Bernardes et al., 2004; Johnson and Derrick, 2010; Pietrelli et al., 2005)

3.2 Chemical Processes

The chemical processes are mainly hydrometallurgical methods involving acid or base leaching, solvent extraction, chemical precipitation, bioprocess and electrochemical process or combination of the processes. The multiple-steps will consume more chemicals.

3.2.1 Hydrometallurgical Processes

The scraps of the spent LiBs were put in either acid or alkaline solution to dissolve the metallic fraction of the batteries to recover valuable components (Bernardes et al., 2004). Hydrometallurgical was used on the basis of its simplicity, environmentally friendly due to waste water and air emission minimization, adequate recovery of valuable metals with high purity and low energy requirements (Li et al., 2010a, 2010b; Pietrelli et al., 2005). For instance, cobalt-containing slag was treated through hydrometallurgical process by Lain (2002) and Espinosa et al (2004).

This process also used the mixture of H_2SO_4 and H_2O_2 to recover Li and Co from LiBs and achieved full recovery of the metals within 10 min at 75 °C with an agitation of 300 rpm. However, the thermal pretreatment of LiCoO₂ particles to remove carbon and organic binder before chemical leaching significantly reduced the leaching efficiency. Also, LiPF₆ decomposed into lithium fluoride and phosphorus pentafluoride during crushing process (Shin et al., 2005).

Also, with an enhanced leaching efficiency, mixture of an easily degradable organic acid DL-malic acid and H_2O_2 was used to recover Co and Li from LiBs (Li et al, 2010a). Instead of DL-malic acid with H_2O_2 , both Co and Li were effectively recovered using citric acid and H_2O_2 (Li et al., 2010b). Kang et al (2010a) leached cobalt-containing powder from LiBs with H_2SO_4 and H_2O_2 to recover cobalt sulfate, while addition of oxalic acid to the filtrate from another powder produced crystalline cobalt oxalate, which was then heated to produce Co_3O_4 (Kang et al., 2010b). Zhang et al (1998) recovered Co and Li using HCl solution. The Co in the leached liquor was selectively extracted with PC-88A in kerosene and then as cobalt sulfate with high purity, while Li was obtained as LiCO₃.

A combination of ultrasonic washing, acid leaching and precipitation was proposed by Li et al (2009a, 2009b) to recover Co from spent LIBs. The ultrasonic washing improved the recovery efficiency of Co, reduced energy consumption as well as environmental pollution.



This process was considered feasible for recycling spent LIBs for scale-up operation (Li et al., 2009a). A recycling process that combined hydrometallurgical and sol-gel steps in addition to other general steps was also used to recover Co and Li from LiBs. The acid media (hydrogen peroxide in HNO₃) used enhanced the leaching efficiency. A gelatinous precursor was prepared by adding citric acid to the leaching liquor to obtain amorphous citrate precursor process (ACP), followed by pyrolysis to obtain pure crystalline LiCoO₂ (Lee and Rhee, 2002).

3.2.2 Combined Acid-Alkaline with Organic Solvents Process

As a means of advancing the process of recycling spent lithium-ion batteries, combined acids-alkaline and organic solvents was used for safety, simplicity and other benefits observed in other methods. Lithium, Ni, Mn and Co were leached from LiMnNiCoO₂ using HNO₃ and then precipitant, NaOH by Castillo et al (2002), while Consestabile et al (2001) also leached LiCoO₂ with HCl and then precipitated the cations with NaOH solution. In similar steps, the batteries inner rolls were treated with NaOH to dissolve the aluminium foil to separate the cathode material powders from other components. The powder obtained was burnt to remove PVdF, followed by dissolution to produce sulfate solution. Cobalt in the solution was deposited as oxalate, while Acorga M5640 and Cyanex272 (di-(2,4,4 trimethyl pentyl) phosphoric acid) were used to selectively extract small quantities of Cu²⁺, Co²⁺ (Nan et al, 2005) and Ni²⁺ ions (Nan et al., 2006) in the solution. Wang et al (2009) selectively used KMnO₄ to recover Mn as MnO₂ and manganese hydroxide from the leaching liquor, while dimethylglyoxime was used to recover Ni. Cobalt was precipitated as cobalt hydroxide, while addition of a saturated Na₂CO₃ solution to the liquor precipitated Li as Li₂CO₃. The process can be represented by the flow-sheet in Figure 4.





Figure 4. Flow sheet for general acid-alkaline with selective recovery and recycling of LiBs.

3.2.3 Bio-Metallurgical Process

Compared with the aforementioned pyrometallurgical, hydrometallurgical processes, bioprocess was considered as having higher efficiency, low cost and environmentally compatible (Bernardes et al., 2004; Xin et al., 2009). The process used bacteria and inorganic chemical solutions. For example, acidithiobacillus ferrooxidans utilized elemental sulfur and ferrous ion to produce metabolites, H_2SO_4 and ferric ion in the leaching medium to recover Li and Co from LiCoO₂ of LiBs. The metabolites enhanced the dissolution of metals from the batteries. Comparatively, bio-dissolution of Co was faster than Li (Mishra et al., 2008). Xin et al (2009) also recovered Co and Li from the spent LiBs through the same processes. However effective the procedure may be, the cost of culturing the enzymes or bacteria may somehow hinder its commercial operation.

3.2.4 Electrochemical Process

Electrochemical methods have been used to recover metals from the leached liquor of the cathode active materials of LiBs. Meanwhile, it was impossible to recover Ni directly by the method from the liquor obtained. Therefore, Ni was first separated from Co by solvent extraction, followed by its recovery through galvanostatic and potentiostatic electrowinning (Lupi and Pasquali, 2003). Also, Freitas and Garcia (2007) electrochemically recovered Co, while combination of the electrochemical and hydrothermal methods were used to recover

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both Co and Ni from $LiCoO_2$ and $(LiCo_xNi(_{1-x})O_2$ in the Li ion and Li polymer batteries, respectively (Lupi et al, 2005). The ionic equations for the electrochemical reactions of a divalent cation during electrolytic recycling process could follow:

Anode: $M^{2+}+2H_2O \longrightarrow MO_2 + 4H^+ + 2e^-$ Cathode: $2H^+ + 2e^- \longrightarrow H_2$ Overall: $M^{2+}+2H_2O \longrightarrow MO_2 + 2H^+ + H_2$

3.2.5 Pyrometallurgical Process

The process chemically recovered valuable components of the waste materials or concentrates at elevated temperatures (Espinosa et al, 2004). Pauline et al (2008) fused the mixture of active mass (cathode and anode) and electrolyte with KHSO₄ in a furnace. Although precaution was taking to avoid reduction of sulfate to SO₂ of sulfide, industrial dumps like CaF₂, Ca₃(PO₄)₂ and other byproducts were generated along with the desired products.

4. Conclusion

From the review so far, several attempts have been made to clean the environment and achieve the general objectives of waste sustainability and management. The chemical processes have been improved upon with great success recorded. However, the amount of chemicals involved in most of the multiple-steps used could render them economically unsuitable beside the effect of byproducts or other wastes generated that must be properly treated in conformity with the acceptable limit for the environments.

The use of suitable non-toxic organic solvents capable of dissolving the PVdF in the batteries to maximize the recovery values and enhance the reduction in the amount of HF gas released by pyrolysis before leaching has proved more environmentally compatible. We suggest that the inner materials of the LiBs be washed with suitable organic solvents to extract the toxic electrolyte mixture to avoid pollution caused by hydrolysis of LiPF₆. Also, conversion of the electrolyte salt to useful products is achievable to minimize wastes generation.

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