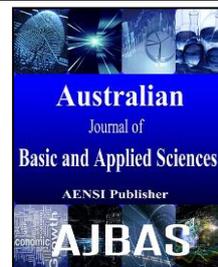




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### Characterization and recycling procedure of spent Lithium Ion Batteries from mobile phones.

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#### ABSTRACT

Even with the existing environmental and economic incentives for the recycling process of spent lithium batteries, many challenges are in need to be resolved in order to have an effective recycling process. Due to vast amount of types and brands of discarded batteries, the initial obstacle to overcome is to have a proper characterization of the discarded batteries available in the market. With the aim to contribute with the development of this recycling process, this study performed an initial chemical characterization of discarded mobile phone batteries in Bogotá (Colombia). A dismantling and characterization procedure was proposed and tested. A full characterization in terms of weight percentage of the main battery components, elemental chemical composition of electrodes, electrolytic materials and encasing was performed using atomic absorption spectroscopy, both cathode and anode are the batteries component with higher weight percentage (15.1 % and 9.8 %, respectively). Special attention was given to the characterization of the electrodes polymeric encasing element (coating), these thin films contain the active electrode material, some binders and graphite; cobalt and graphite were the most abundant material in the cathode; meanwhile graphite and aluminum were the most abundant material in the anode. Both anode and cathode present secondary composition of Lithium. The graphite collected after the filtration has a flat laminar shape and may be an interesting material for the synthesis of carbon base materials, as grapheme of high surface delaminated graphite. The proposed procedure proved to be a simple, safe and reliable methodology to dismantle and characterize spent lithium ion batteries from mobile phone devices.

#### INTRODUCTION

Due to the development of new technologies in the field of mobile electronic devices and the multiple technical capabilities and applications that this gadgets offer nowadays, their energy consumption has also increase the power density require for their proper operation and have challenged the current energy accumulators (mainly batteries) that are used on them (Rhenita *et al.* 2014; Kesavan and Rajeswari 2015). At the moment, the battery system most widely used is the lithium ion type (LIBs, lithium ion batteries), because of its high energy density and light weight. With the important number of mobile electronic devices sold every year, the quantity of dispose batteries has also increased and the proper disposal of these element after their cycle life has ended is also of paramount importance, especially when it is estimate that for world LBS reached the 4.6 billion of unit in 2010 (Scrosati *et al.* 2011). It has become very relevant to study the different alternatives to

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safely and responsibly discard this type of batteries at the end of their service life, reducing or eliminating any possible environmental and sanitary risks (Paulino *et al.* 2008; Yeong *et al.* 2015). Recycling chemical compounds from batteries could become an important source of raw material for the production of new batteries, reducing the need of using new natural resources in their fabrication and assuring the provision, in some cases, of the rare or expensive materials (Raa and Han 2006; Tong *et al.* 2006; Dorella and Mansur 2007; Paulino, Busnardo *et al.* 2008). Currently several research work is focused on the evaluation and selection of the most promising technology for the recovery of those materials; most of the current processes yield Li, Co and metallic related compounds that serve as raw materials for the fabrication of new active cathodic materials (Nayaka *et al.* 2016). The main components of this type of batteries are cobalt, copper, lithium and aluminum. Li and Co are element of big demand nowadays, because they are extensively used in the electronic industry. In order to propose a plausible recovering technology it is important to have a proper assessment of the average composition of the commercial available batteries. Today, the most common type of active cathodic material of the lithium ion batteries is lithium cobaltate. Due to their high energy density, high cell voltage, long storage life, low self-discharge rate and large temperature range lithium batteries are often preferred to conventional systems with aqueous electrolytes. Because of high reactivity of lithium in air or moisture, lithium-metal batteries are industrially processed only in a few places (Canada and USA) by Toxco Inc. and BDC Inc. using low-temperature process recycling plants. Lithium-ion batteries are reprocessed in France (SNAM) or in UK (AEA technology batteries) (Xu *et al.* 2008) mainly with the aim to recover electrolyte and valuable metals from the positive electrode. Most of the proposed recycling processes typically used hydrometallurgical techniques combined chemical leaching (Contestabile *et al.* 2001; Lupi and Pasquali 2003). Hydrometallurgical processes that included smelting have the shortcoming of high energy consumption and could be potentially linked with air pollution. Moreover, it does not recover lithium and aluminum (Dorella and Mansur 2007). On the other hand, chemical leaching brings metals into solution; some authors propose the use of organic oxalates and organic acids as lixiviating solvent (Li *et al.* 2010; Sun and Qiu 2012). Although some environmental concerns, recycling processes that use inorganic acids are still being studied (hydrochloric and sulphuric acid) (Wang *et al.* 2009; Zhu *et al.* 2012).

To have a clear understanding of the challenges that the process of recycling of LIBs presents it is very important to know chemical characteristics of the electronic waste to be processed. The following list of components will be used as a guideline to understand the proper way to handle this type of batteries and to recognize the kind of risks that must be confronted or avoided during the recycling process.

- **Electrolytes:**

- Lithium hexafluorophosphate [LiPF<sub>6</sub>]: decomposes to lithium fluoride and phosphorus fluoride, when the battery is opened. It is highly hygroscopic and must be keep out of contact with moist air or water, strong oxidizing agents and strong acids.

- Lithium perchlorate [LiClO<sub>4</sub>]: it is a strong oxidizer. Contact with other material may cause fire. Decomposition starts at 400 °C and accelerates at 430 °C yielding lithium chloride and oxygen.

- Lithium tetrafluoroborate [LiBF<sub>4</sub>]: Corrosive material. Conditions to avoid include exposure to moist air or water. Incompatibilities with Other Materials: Strong acids Hazardous decomposition products could be carbon monoxide, carbon dioxide, hydrogen fluoride.

- Propylene carbonate [C<sub>4</sub>H<sub>6</sub>O<sub>3</sub>]: Stable under ordinary conditions of use and storage. Hazardous Decomposition Products: Carbon monoxide, carbon dioxide. Incompatibilities: Water, strong oxidizing agents.

- Diethyl carbonate [C<sub>5</sub>H<sub>10</sub>O<sub>3</sub>]: Flammable liquid and vapor. May form explosive peroxides. Moisture sensitive. Stable under normal temperatures and pressures. Conditions to Avoid: High temperatures, mechanical shock, incompatible materials, ignition sources, exposure to air, strong oxidants, exposure to moist air or water. Forms explosive mixtures in air.

- **Polymer insulator:**

- Polyvinylidene fluoride [C<sub>2</sub>H<sub>2</sub>F<sub>2</sub>]: Exposure to thermal decomposition products can cause serious hydrogen fluoride burns and corrosion. Thermal decomposition may produce an irritating mixture of smoke and highly toxic gases. Fluoride emissions may be significant. Stable at room temperature.

- External plastic case and brand coverage.
- Electric circuit which has the contacts.
- Metallic shell.
- Cathode

- Aluminum [Al]: Flammable solid. Dust may form flammable or explosive mixture with air. Stable under ordinary conditions of use and storage. Reacts with some acids and caustic solutions to produce hydrogen. Conditions to Avoid: Moisture, heat, flames, ignition sources and incompatibles.

- Lithium cobaltate [ $\text{LiCoO}_2$ ]: Stable under recommended storage conditions. Avoid moisture. Materials to avoid: Strong oxidizing agents Hazardous decomposition products: Hazardous decomposition products formed under fire conditions.

- Lithium oxides, Cobalt/cobalt oxides

- **Anode:**

- Cooper [Cu]: Stable under ordinary conditions of use and storage. Copper becomes dull when exposed to air; on exposure to moist air it gradually converts to the carbonate. On long standing, a white, highly explosive peroxide deposit may form. Incompatibilities: Copper is incompatible with oxidizers, alkalis, acetylene, chlorine plus oxygen difluoride, phosphorus, nitric acid, among others. A potentially explosive reaction occurs with acetylenic compounds. Copper ignites on contact with chlorine, fluorine (above  $121\text{ }^\circ\text{C}$ ), chlorine trifluoride, and hydrazinum nitrate (above  $70\text{ }^\circ\text{C}$ ). An incandescent reaction occurs with potassium dioxide.

- Graphite [C]: Conditions of Instability: Excess heat, incompatible materials. Incompatibility with various substances: Highly reactive with oxidizing agents. Special Remarks on Reactivity: Reacts vigorously with liquid potassium, and potassium peroxide.

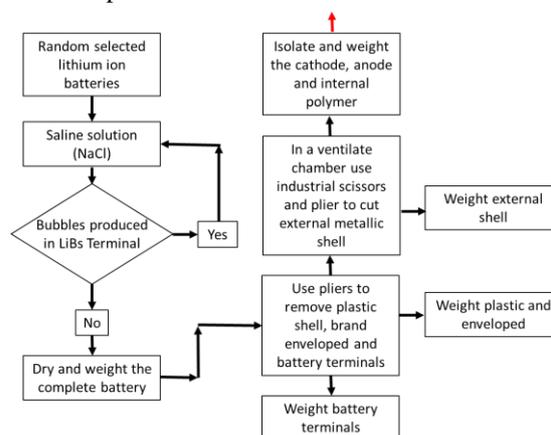
- Lead [Pb]: The product is stable. Reactive with oxidizing agents. Can react vigorously with oxidizing materials. Incompatible with sodium carbide, chlorine trifluoride, among others.

The previous information emphasizes the importance of a proper recycling and recovery process for discarded batteries, unappropriated disposal of this materials could pose an important environmental and health risk, many of them produces dangerous compounds like hydrogen fluoride or other highly toxic gases when they get in contact with the atmosphere humidity. Other compounds like cobalt are capable to cause cancer to the living being that enter in contact with it.

The purpose of this study is to perform an initial characterization of the discarded mobile phone batteries in Bogotá (Colombia) employing a simplified and safe dismantling and recovery procedure. It is estimated that annually between 200 to 500 tons of lithium ion batteries are discarded (Lee and Rhee 2003; Tetsopgang and Kuepouo 2008) . The produced materials must fulfill some technical specifications so them can be used in the electronic industry, this is why is very important not only to recover them but also to guarantee that them have the appropriate characteristics, and this is achieved by doing analysis to the products of the mentioned processes (Paulino, Busnardo *et al.* 2008; Xu, Thomas *et al.* 2008; Nayaka, Pai *et al.* 2016; Ordóñez *et al.* 2016)

## RESULTS AND DISCUSSION

Due to the particular combination of mechanical strong structures (battery cases) and the multitude of chemical compound containing in LIBs, in order to recover the component material a combination of mechanical, chemical and hydrometallurgical procedures have proposed (Zhang *et al.* 1998; 1999; Contestabile, Panero *et al.* 2001; Lenormand *et al.* 2008; Li, Ge *et al.* 2010; Zhu, He *et al.* 2012). The current study proposes a simplified but efficient and safe procedure to dismantle and characterize discarded LIBs. To determine the composition of the studied batteries, a series of experiments were carried out. This, and the entire following test, was performed by randomly selecting some batteries from a group of 1000 LIBs that were collected at some mobile phones technical service centers in Bogotá, Colombia. The initial test performed had the goal to determine the average weight of the main parts of the lithium ion batteries. The procedure used for this determination is presented in Figure 1 and summons up the best characteristics of previously proposed procedures (Zhang, Yokoyama *et al.* 1998; Shin *et al.* 2005). The references of the three randomly selected batteries used were: Siemens (A&T Battery Corp. Japan), GH (Standard Battery BST-36 China), Motorola (Japan, SNN 5779A1), and the results obtained are presented in Table 1.



**Fig. 1:** Procedure for the determination of the weight of the main parts of the lithium ion batteries.

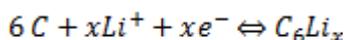
**Table 1:** Average weight of the main parts of the lithium ion batteries.

Component	Average [g]	Percentage [%]
Total weight	17,8	-
Plastic shell and brand envelope	1,8	5,2
Battery terminals	0,3	0,9
Metallic shell	4,3	12,2
Cathode	5,4	15,1
Anode	3,5	9,8
Internal polymer	0,6	1,8
Electrolytes	1,8	5,0
Total	17,8	100

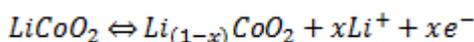
Before the opening procedure of the batteries to gain access to the internal parts, it is very important to submerge the discarded battery in a saline solution in order to remove any residual energy storage in the battery. The importance of this step lies in the fact of potential short circuits that could happen during the opening process, leading to a possible overheating and even an explosion. According to the results reported in Table 1, the battery part that weights the most is the cathode, meanwhile the battery terminals are the less significant. The battery terminals consist on a small circuit board and some metallic contacts, the board is not lixiviated by the digestion process, but the metallic terminals and connections are. Because the electrolytes evaporates and are difficult to isolate when opening the battery, the weight of this component was calculated using a mass balance. These vapors must be treated carefully because they produce some gases when entering in contact with moist air, which can be dangerous to human health. To avoid this risk, a gas mask and an extraction chamber were employed.

The basic parts of this type of batteries are a cathode, an anode, an organic electrolyte, and a separator. Delamination of the cathode, anode and separator are achieved by pressing the electric contacts. The anode is a copper plate coated with a mixture of carbon graphite, conductive material, PVDF binder, and additives. In the same way, the cathode is an aluminum plate coated with a mixture of active cathode material, electric conductor, PVDF binder, and additives. Lithium cobaltate is commonly used as an active cathodic material for almost all commercialized LIBs. The chemical reactions in the two electrodes can be simply expressed as follows:

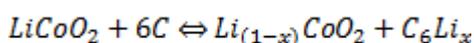
The cathodic reaction:



The anodic reaction:

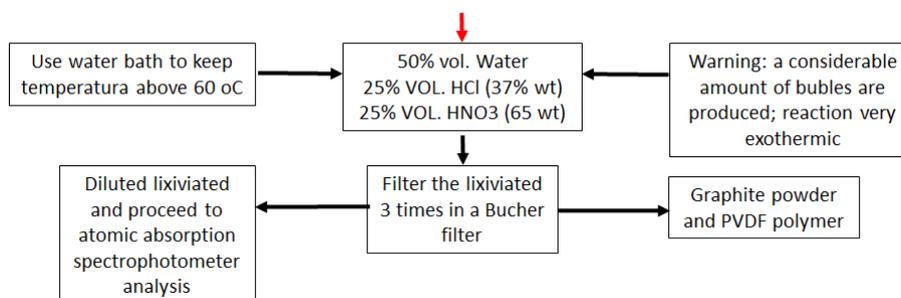


Taking into account that the forward direction of these reactions is carried out while charging and the reverse reaction is during the discharge. Basically, the energy in this kind of batteries is stored by the migration of lithium ions from the cathode to the anode, represented in the following reaction:



Reaction that generates the typical voltage of this kind of batteries: 3,6/3,7 V.

Metallic chemical elemental analysis of the battery component was performed using an atomic absorption spectrophotometer. The procedure used is described in Figure 2, where the red arrow represents the final stage of the procedure described in Figure 1. Atomic absorption spectrophotometry was carried out analyzing the main metals present in the batteries (Table 2):

**Fig. 2:** Procedure to determine the average metallic composition of the lithium ion batteries

**Table 2:** Average metallic composition of the lithium ion batteries

Component	Wt. %
Co	21,3
Fe	23,3
Ni	2,2
Mn	2,6
Pb	0,2
Cu	9,2
Li	3,2
Al	14,3
Not analyzed mass	17,4

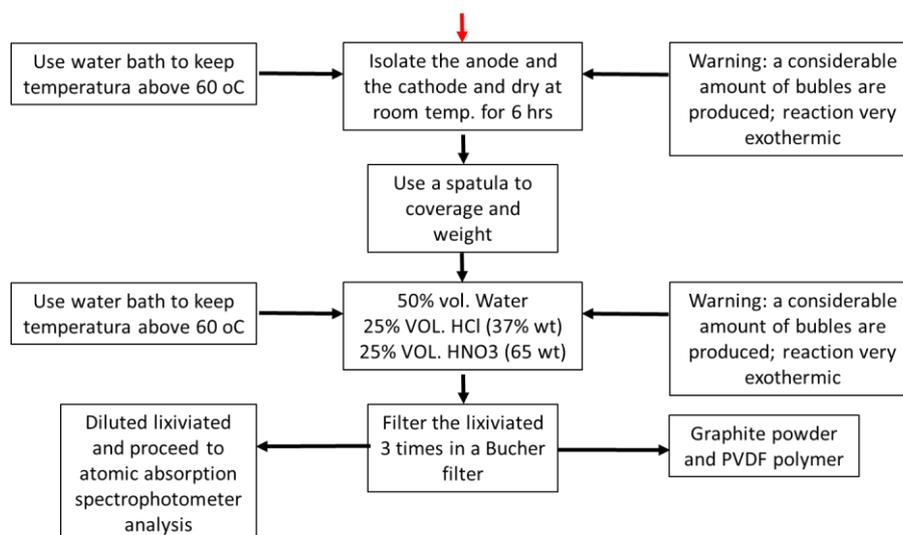
The not analyzed mass consist of those material not possible to measure by atomic absorption spectrophotometry, due to the fact that during the digestion process of the battery components necessary to dissolve the sample is carried out, many of the compounds like electrolytes and active cathodic materials produces gases, which are volatile and will not be quantified by the atomic absorption spectrophotometry, however the other parts were carefully weight and the corresponding not analyzed weight was calculated by a mass balanced, results that are comparable with the obtained by Li et. al. (Li, Ge *et al.* 2010), who performed leaching experiment with several type of organic acids.

As it is reported in Table 2, among the weight percentage of metals present in the lixiviated batteries, iron is the most abundant element and it is mainly present in the external metallic shell. The second metal in order of weight percentage is cobalt, which is the main active cathodic material and as is expected to be found in high proportion in a lithium ion battery. Aluminum is mainly detected in the cathode current collector as in the case of cooper in the anode current collector. Lithium is found in a small weigh proportion, but not because the amount of moles existing in a battery is low, but because of low its molecular weight. Manganese and the nickel are found in batteries because in some cases the lithium cobaltate is complemented or replaced by other lithium salts like lithium nickel dioxide [LiNiO<sub>2</sub>] or Lithium manganese oxide. The lead found in the batteries is mainly present in the junctions between the current collectors. Also, the main internal parts of the batteries were weighted to determine the percentage of mass that each one of them represented and are summarized in Table 3.

**Table 3:** Mass percentage of the main internal parts of the batteries.

Component	Wt. %
Total representative metals	55,6
Not analyzed mass	17,4
Graphite weight	16,1
Internal polymer weight	2,5
Plastic shell and brand envelope	7,7
Circuit weight	0,8

The information presented in Table 3 shows that most of the internal battery parts are suitable of being lixiviated by the mixture of strong acids employed in the test, and this is an important information to take into account due the fact that this metal ions can be then employed to synthesize other compounds, which is one the main goal of any recycling process. The amount of graphite found in the analysis is also high and it comes mainly from the active anionic material. The plastic shell and internal polymer encompasses an important percentage of the potential recyclable material, however due to the risk of being in contact with dangerous materials (as in the case of a leakage or a battery explosion) their recovery must be done following the same protocols of safety followed in the recycling of the battery internal parts. Special attention was given to the characterization of the electrodes polymeric encasing element (coating), these thin films contain the active electrode material (the one where the charges movement and storage take place), some binders and graphite. This part of the study is very important because it reveals the target metals, like lithium and cobalt, are located and this information can be used to design a more efficient and specific recycling process. The procedure to characterize the coating of the electrodes is the one presented in Figure 3, where the red arrow once again represents the final stage of the procedure described in Figure 1.



**Fig. 3:** Procedure to obtain the coating chemical composition of the electrodes (active electrode materials)

Independent experiments were performed for the anode and cathode coating, the results of the characterization of the cathode coating material is presented in Table 4.

**Table 4:** Cathode coating material composition.

Component	Wt. %
Non analyzed mass	42,2
Co	32,1
Graphite weight	19,0
Li	3,6
Al	3,1
Ni	0,0
Fe	0,0
Mn	0,0
Pb	0,0
Cu	0,0

Results from the characterization presented in Table 4 show that most of the weight percentage is related to cobalt and graphite. Also lithium and aluminum are present, in a small proportion because of their light molecular weight, but in terms of number of moles, it is obvious that there is more lithium than aluminum in the lixiviated cathode cover. Also, the amount of aluminum is caused by the pieces of the aluminum anode current collector to where is attached the active cationic material, comparable with result published in recent literature (Wang *et al.* 2009; Song *et al.* 2013). A small amount of Cu was detected in the polymeric electrode encasing element, but for the other metals listed in the Table 4, no detectable signal was observed. About this lixiviating procedure, it is important to state that the reaction between the cathodic coating and the acid mixture is exothermic and therefore it must be taken into account as a risk due to its tendency to get into the well-established run away condition of exothermic reactions when not proper control of temperature and heat transfer in the reaction chamber is done. The graphite collected after the filtration has a flat laminar shape and may be an interesting material for the synthesis of carbon base materials, as grapheme of high surface delaminated graphite. The results of the characterization of the anode coating material are presented in Table 5.

**Table 5:** Anode coating material composition.

Component	Wt. %
Graphite weight	78,9
Non analyzed mass	12,4
Al	6,2
Li	2,1
Cu	0,4
Fe	0,0
Ni	0,0
Co	0,0
Mn	0,0
Pb	0,0

The atomic absorption spectroscopy analysis performed on the anode coating reported in Table 5 mainly reports that it is compose of graphite as a main component. The amount of lithium and aluminum follow the

explanation made for the case of the cathode, which a common characteristic result of recovery process that follow similar recycling routes (Lee and Rhee 2002; Li *et al.* 2012). The amount of copper present is also due to some pieces of copper present in the current collector to where is attached the active anionic material.

### Conclusions:

End of cycle life LIBs pose an important health and environmental risk and if proper recycle could become an important source of materials (Li, Co, aluminum, copper) for new batteries and other applications. The first stage to have a successful recycling process is to have an adequate characterization of the discarded LIBs. In this study, an initial chemical characterization of the discarded mobile phone batteries in Bogotá (Colombia) was performed following simplified and safe dismantling procedure, the discarded batteries were characterized following a simple but effective procedure. The process includes mechanical separation of internal parts, saline blend, acid leaching and thermal treatment. The proposed procedure allowed determine that the battery component that weights the most is the cathode, meanwhile the battery terminals are the less significant in weight. Metallic chemical elemental analysis of the battery component was performed using an atomic absorption spectrophotometer; out of the metals detected in the leached batteries, iron is the most abundant element and it is mainly present in the external metallic shell. The second metal in order of weight percentage is cobalt, which is the main active cathodic material and as is expected to be found in high proportion in a lithium ion battery. Aluminum is mainly detected in the cathode current collector as in the case of copper in the anode current collector. Special attention was given to the characterization of the electrodes polymeric encasing element (coating), these thin films contain the active electrode material, some binders and graphite. The graphite collected after the filtration has a flat laminar shape and may be an interesting material for the synthesis of carbon base materials, as graphene or high surface delaminated graphite.

The remarkable results obtained in this bench scale experiments open a window for a future pilot plant test, where mass and heat transfer effect must be accounted, and detailed prospective economic analysis could be performed.

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