



Identification and quantification of gases emitted during abuse tests by overcharge of a commercial Li-ion battery



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HIGHLIGHTS

- The overcharge abuse test of a 35 cm³ commercial LFP cell is studied.
- A new analytical setup and a specific procedure have been established.
- Some species are produced after the first deconfinement of the cell.
- HF has a specific behavior: its formation is decorrelated from the other species.
- The gases released by the cell are mainly composed of flammable volatile solvents.

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ABSTRACT

As hazardous situations can occur during the life of a Li-ion battery, it is of great importance to understand its behavior under abusive conditions (mechanical, thermal or electrical). In particular, the study of overcharge, which consists of forcing a current through the cell, can be very helpful in improving battery safety. Very few studies in the literature have focused on the chemical reaction mechanism responsible for failure during overcharge. This is, however, of great interest because a Li-ion battery can produce reactions in a sealed container and is thus a highly reactive system. Here, experimental approaches are employed to understand the reaction mechanisms that occur during overcharge testing. Experiments consist of studying the overcharge kinetics of a commercial battery at an initial state of charge of 100%. The battery is maintained in a known volume and gaseous samples are withdrawn both at the end of the test and continuously during the test. The main gaseous species are then identified and quantified by gas phase chromatography coupled with mass spectrometry and FTIR spectroscopy. This experimental study is completed by a numerical investigation to determine the combustion parameters of the exhaust gases using a detailed reaction mechanism associated with a numerical code.

1. Introduction

Many devices involve Li-ion Batteries (cell phones, laptops, electric vehicles or airplanes) for their weight/power advantages. As hazardous situations can occur during the life of the battery, it is of great importance to understand its behavior under abusive conditions, whether mechanical (cross/deformation/penetration), thermal (external heating) or electrical (internal short circuit/external short circuit/overcharge/overdischarge). The literature concerning abuse tests on Li-ion batteries is extensive because these tests are aimed at improving battery safety. Moreover, there are a large number of possible tests as well as possible cell compositions and geometries.

In particular, the study of an abnormal electric situation such as

overcharge, which consists of forcing a current through the cell, can be very helpful in improving battery safety. In real life, overcharge can be caused when the cell voltage is abnormally increased by the charge control system, by a defective charger or when the wrong charger is used. The Li-ion battery can thus produce reactions in a sealed container and can be considered as a highly reactive system. This is an uncommon event, but when it occurs the consequences can be very severe [1] such as toxic and/or corrosive damage to the device and to persons, a risk of flammability or hot spot.

As Li-ion cells with carbonate-based electrolytes are widely marketed, the physical hazards are relatively well known. The chemical and health hazards are less well-known, however, and only a few studies have aimed at establishing correlations between cell response during an

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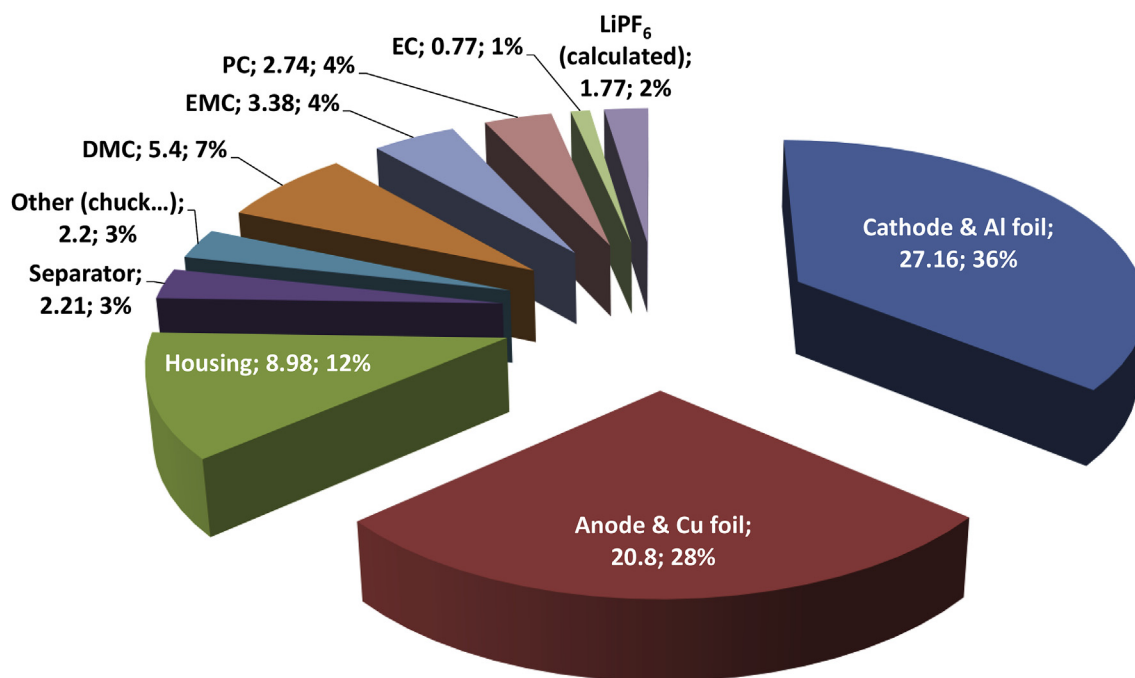


Fig. 1. Cell composition (elements; mass in g; mass ratio in %w). The mass of the complete cell before disassembly is 75.41g.

abuse event and gas exhaust. The review of the available literature (see next section) shows that, whereas the behavior of cells with carbonate-based electrolytes is relatively well documented in the case of thermal abuse, few studies deal with overcharge abuse tests. Hence there is a need to gain more insight into the reaction mechanisms that occur during overcharge abuse tests. Besides, from a flammability hazard point of view, the gases released by the cell can be combustible, depending on the environment. There are two main reasons for the flammability/explosive hazard: i) the gas generation is large and sudden (venting) [2]; ii) the gas and flammable electrolyte solvent vapor released is a flammable or explosive mixture that can be easily ignited [3,4]. This hazard has thus to be taken into account. Consequently, there is a need to investigate the chemical reaction mechanism responsible for cell opening during overcharge. This is the aim of the present study.

In this paper, the overcharge abuse test of a 35 cm³ commercial Li_xFePO₄ LFP cell was studied. A new analytical setup and a specific procedure were established, making this study original and innovative. Gachot et al. (2014) [5] demonstrated that it is possible to identify gaseous released by a Li-ion battery by coupling Gas chromatography (GC) with Fourier Transform InfraRed (FTIR) and/or Mass Spectrometry (MS). In the present paper, not only the identification but also the quantification of each species was performed using GC/MS and FTIR. The battery was maintained in a closed chamber and the gaseous sample was withdrawn not only at the end of the test as is usually done, but also continuously during the test, enabling the main gaseous species to be continuously identified and quantified by FTIR and GC/MS. This study thus provides important new elements in the understanding of the reaction mechanisms, which still remain unclear, that occur during an overcharge abuse test. Finally, to complete the study from a flammability hazard point of view, the typical combustion parameters (fundamental flame velocities, adiabatic flame temperatures and heat release rates) of the exhaust gases were determined using a detailed reaction mechanism associated with a numerical code.

2. Experimental

2.1. Cell characteristics

A commercial cell, based on an LFP cathode, was tested. It is a cylindrical 26650 cell with an inner diameter of 26 mm and a length of 65 mm. The weight of the cell is 75g. According to the manufacturer's data, its nominal capacity is 2.5Ah and its nominal voltage is 3.6V. Current-limiting or temperature trip safety devices were not used in the cell. In order to carry out quantitative analyses, it is important to know the mass split of the cell components. As this information is kept confidential by the manufacturers, we conducted this analysis ourselves in collaboration with the LITEN laboratory of Grenoble. To characterize the cell components, a cell was completely discharged with a 1C-rate and then dismantled in a glove box providing an inert environment (H₂O and O₂ < 10ppmv).

The cell components (the anode, the cathode, the current collector foils, the separator and the housing material) were rinsed with acetonitrile and dried in a dessicator until stabilization of the weight. As expected, the studied cell has a cathode that belongs to the family of active material Li_xFePO₄ (LFP) and the anode belongs to the carbon family. Consequently, the comparison with literature results obtained with such cathodes can be envisaged. These materials were identified, after being rinsed with acetonitrile, by Scanning Electron Microscopy (SEM) and X-Ray Diffraction (XRD).

The separator foil was examined in inert atmosphere with thermal gravimetric analysis with a heat ramp of 10K.min⁻¹. An endothermic peak was observed at 150 °C corresponding to the fusion temperature of polypropylene. The thickness of the separator was 20 μm (measured with a micrometer). A FTIR spectroscopy analysis confirmed the nature of the separator.

A different methodology was used to quantify the volatile solvents, as the time between removing the separator and its immersion caused the evaporation of most of the solvents. Another cell was therefore dismantled in a polyethylene glove bag, the bag was then hermetically sealed and introduced in the glove box. The glove bag was connected to an FTIR analyzer, which quantified the volatile solvents until their total volatilization. The IR spectra (not shown here) were integrated and the solvent mass ratios of the electrolyte were determined. It was observed

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