



BACHELOR'S THESIS

Study of cathode, anode, and ionic liquid-based electrolyte preparation for novel lithium-ion battery fabrication

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I confirm that the work is self-prepared and that references/source references to all sources used in the work are provided, cf. Regulation relating to academic studies and examinations at the Western Norway University of Applied Sciences (HVL), § 12-1.

Foreword

I would like to thank my internal supervisor Yansong Zhao for guiding me during this project and the Western Norway University of Applied Sciences for hosting me during the Spring semester.

Abbreviations

Abbreviation	Definition
LIBs	Lithium-Ion Batteries
ILs	Ionic Liquids
$[EtNH_3][NO_3]$	Ethylammonium nitrate
$LiPF_6$	Lithium hexafluorophosphate
HF	Fluoric acid
$Ca(NO_3)_2$	Calcium nitrate
H_2O	Water
RTILs	Room Temperature Ionic Liquids
TDS	Total Dissolved Solids
$LiFePO_4$	Lithium iron phosphate
SoC	State of Charge
DoC	Depth of Discharge
SoH	State of Health
BMS	Battery Management Systems
FCC	Full Charge Capacity
SOLI	State of Life Indicator
$LiSO_4$	Lithium sulfate
TiO_2	Titanium dioxide

CB	Carbon Black
CMC	Carboxymethyl Cellulose
EC	Ethylene Carbonate
DMC	Dimethyl Carbonate
(Fe)	Iron
(LiOH)	Lithium hydroxide
Cu	Copper
TGA	Thermogravimetric Analysis
<i>BmimPF₆</i>	1-Butyl-3-methyl-imidazolium-hexafluorophosphate
<i>BmimBF₄</i>	1-Butyl-3-methyl-imidazolium-tetrafluoroborate

Abstract

Nowadays, Lithium-ion batteries (LIBs) are probably the most utilized batteries in the world. They are the primary electrical source for several devices such as cars, phones, or laptops because of their excellent specific capacity (about $3860 \text{ mAh} \cdot \text{g}^{-1}$) and other properties such as energy density and voltage capacity. Nevertheless, it is known that some their compounds are very flammable, and this may affect to the battery's efficiency.

This led to many researchers to investigate other green solvents. One of the most important are the Ionic Liquids (ILs), which are not flammable and can be liquid a room temperature. Besides, most of their physical properties (e.g: density, conductivity, and viscosity) are tunable since they depend on what anion and cation are used.

In this bachelor thesis, I am going to prepare Li-ion batteries by using Ionic Liquids as the electrolyte to discuss whether they will improve batteries' security and their electrical properties.

Keywords: Ionic Liquids, green solvents, Lithium-ion batteries, renewable resources, energy technology.

Sammendrag

I dag er litium-ion-batterier (LIBer) sannsynligvis de mest brukte batteriene i verden. De er den primære elektriske kilden for flere enheter som biler, telefoner eller bærbare datamaskiner på grunn av deres utmerkede spesifikke kapasitet (om $3860 \text{ mAh} \cdot \text{g}^{-1}$) og andre egenskaper som energitetthet og spenningskapasitet. Likevel er det kjent at noen av forbindelsene er svært brannfarlige, og dette kan påvirke batteriets effektivitet.

Dette førte til at mange forskere undersøkte andre grønne løsningsmidler. En av de viktigste er ioniske væsker (ILs), som ikke er brannfarlige og kan være flytende en romtemperatur. Dessuten er de fleste av deres fysiske egenskaper (f.eks. tetthet, ledningsevne og viskositet) justerbare siden de avhenger av hvilken anion og kation som brukes.

I denne bacheloroppgaven skal jeg forberede Li-ion-batterier ved å bruke ioniske væsker som elektrolytt for å diskutere om de vil forbedre batterienes sikkerhet og deres elektriske egenskaper.

Nøkkelord: Ioniske væsker, grønne løsningsmidler, litium-ion-batterier, fornybare ressurser, energiteknologi.

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1. Introduction

1.1 Background

To reduce the carbon dioxide emissions that are producing our internal combustion engines, several alternatives to fossil fuels are being studied at this moment. According to this, it is expected that the electrification of transport will have an exponential growth soon.(1)

Lithium-ion batteries are one of the most utilized batteries in the world. These batteries are a very attractive alternative because of their good electrical properties, especially their specific capacity (about $3860 \text{ mAh} \cdot \text{g}^{-1}$) and its lower potential of reduction (-3.04 V).⁽²⁾ However, they have some issues related to their security.

The electrolytes that are used in LIBs are mainly based on aprotic organic solvents, which are usually highly flammable. They are quite often mixtures of various carbonates (e.g. propylene carbonate) and a dissolved salt (e.g. lithium hexafluorophosphate (LiPF_6)). The electrolyte starts to decompose during the event of thermal runaway into gases. Thus, significant overpressure is generated in the cell, which will eventually lead to venting and/or rupture. A major hazard is the presence of fluorinated compounds in the electrolyte, leading to the release of toxic and corrosive hydrogen fluoride (HF).⁽¹⁾

To try to solve those problems, many researchers are struggling to find a substitute for those compounds. One alternative is the use of ionic liquids (ILs) for the electrolyte because they have excellent properties such as non-flammability, they are liquids at room temperature or have vapor pressure near to zero. Besides, they have a good chemical/electrochemical stability, wide potential window (up to 5.5 V) and they are tunable, which means that their properties can be changed depending on the cation and anion that constitute the ionic liquid.⁽³⁾

1.2 Aim and problem formulation

The aim of this bachelor thesis is to develop novel Li-ion battery technology using novel Ionic Liquid based electrolyte and cathode. It is known that Li-ion batteries are one of the most batteries utilized in the world, mainly because of their electrical properties (high energy and power density, long cycle life, relatively high safety, and the continuously decreasing cost)(4). Therefore, that is the reason why researchers are struggling to find a new electrolyte that increases their safety and their electrochemical properties.

On the other hand, the objective of my work is to prove that ionic liquids will improve Li-ion batteries' security and their electrical properties. I will compare my experimental results with some literature to end up in a conclusion.

To achieve those objectives, several LIBs have been prepared. Therefore, it was necessary to learn how to prepare the cathode and the anode. The electrolyte solution, which uses the ionic liquid 'lithium hexafluorophosphate' ($LiPF_6$) as solvent, was already prepared by my internal supervisor. Once the batteries were prepared, they were tested on a battery test system as it is going to be explained with more details during the methods section. Finally, electrolyte's physical properties -such as density, viscosity, and conductivity- were measured.

2. Theory

2.1 What is an Ionic Liquid?

Nowadays, it is considered as an ionic liquid every compound that has a boiling point lower than 100°C and it is only composed by ions, which means that it is liquid a room temperature. However, there is no scientific justification for believing that a salt with a melting point of 90°C is totally different from one with 110°C.

The earliest discovering of these was carried out by Paul Walden in 1914 when he discovered that $[EtNH_3][NO_3]$ has a melting point of 12°C. However, ILs' potential was not recognized for a long time, until some researchers such as Colin Poole picked up his work (1982). He led one group of researchers to investigate the use of Ionic Liquids in analytical chemistry.(5)

In the following figures it is showed the most common cations and anions that are utilized for those compounds:

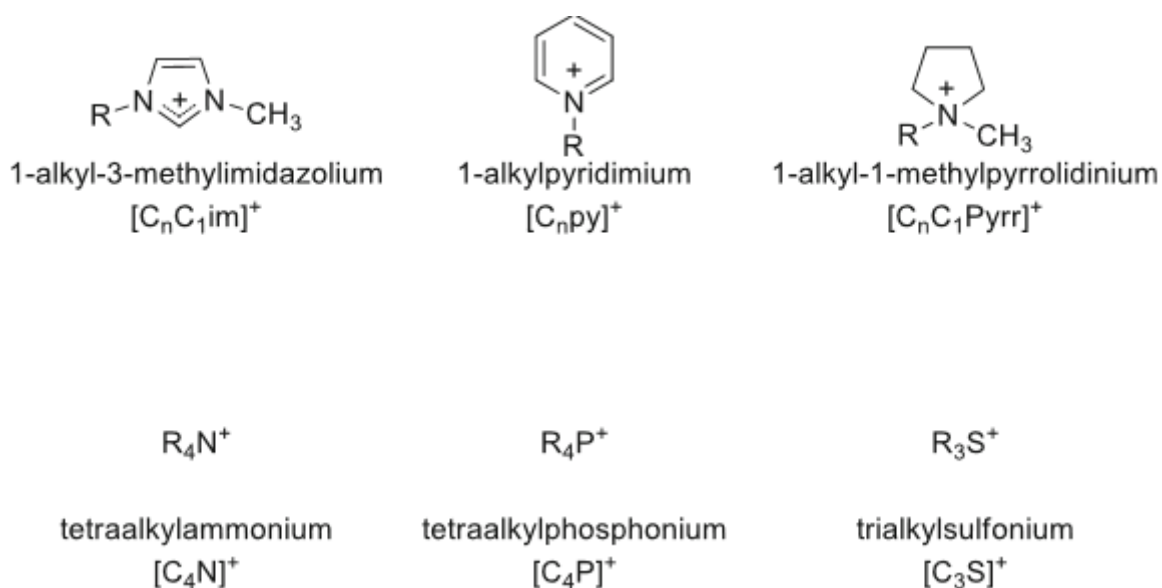


Figure 1. Cations that are commonly used on ionic liquids. (6)

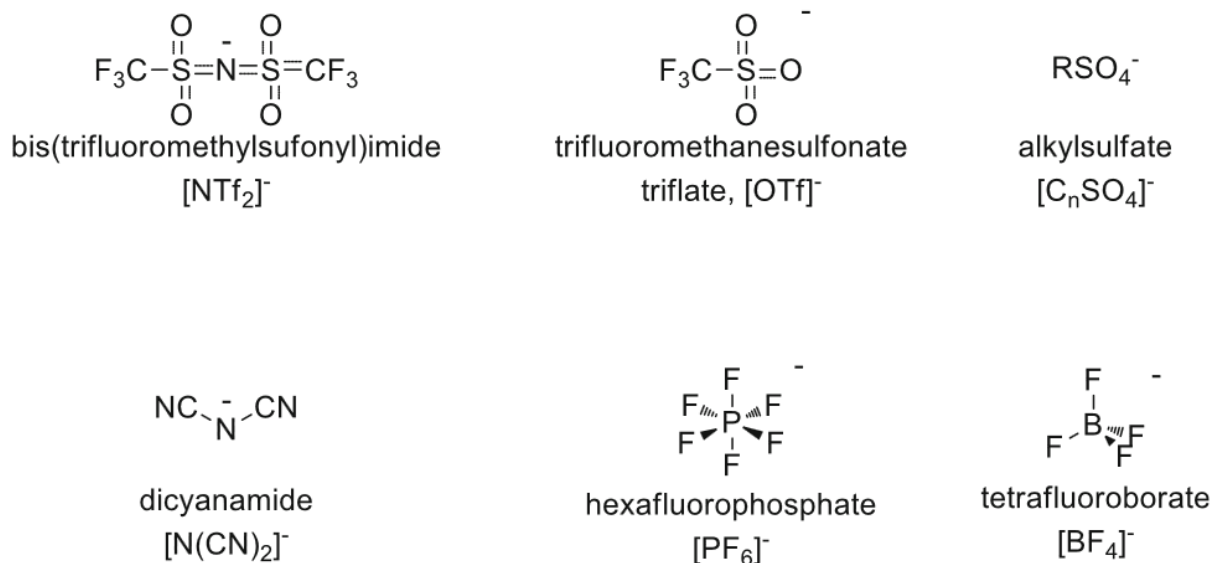


Figure 2. Anions that are commonly used on ionic liquids. (6)

There are three types of ionic liquids:

1. **Aprotic:** Most of them and all are organic molecular ions. Nowadays, it is quite utilized the cyclic (and non-cyclic) tetraalkyl ammonium salts and are usually charge-compensated by fluorinated anions. That type of anions is prominent because of the viscosity-lowering reduction of the van der Waals interactions (thanks to the tightly bound, hence unpolarizable, character of the fluorine electrons). One example of those anions is the hexafluorophosphate (PF_6), which is the one that I am going to use during my experimental work.
2. **Protic:** These are formed by the simple transfer of a proton from pure Brønsted acid to pure Brønsted base. Since this process is reversible – and because of its nature- it is possible to tune the proton transfer energy, which lends those ILs higher conductivities than the aprotic cases. These features were the key to obtain some of the most conductive liquids ever known.
3. **Inorganic:** You can obtain them in aprotic or protic forms.
4. **Solvate(chelate):** These forms are quite unknown now, but this class includes all the ILs that does not have a melting point lower than 100°C. One example could be salt hydrates like $\text{Ca}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$, whose mixtures with alkali metal salts were found to be almost ideal mixtures, most with liquidus temperatures well below ambient. (5)

Speaking in terms of Green Chemistry, ionic liquids are very interesting solvents because of the following properties:

- Very low vapor pressure.
- Low flammability.
- High ionic conductivity.
- Extensive liquid temperature.
- High thermal and chemical stability.
- High solvation ability for organic, inorganic, and organometallic compounds.
- Very selective.
- Easy recyclable through the separation from volatile compounds.

Nevertheless, there are not as green as we might think because several reasons. The main of them is that these compounds are produced from non-renewable sources and are poorly degradable in the environment. Besides, they are not enough studies that can guarantee that ILs are not toxic for humans and environment.(7)

2.2 Physical properties

Density

The formula for density is the following one:

$$\rho = \frac{M}{V} \quad (1)$$

Where ρ is density, M is mass and V is volume. This property is usually expressed in units of grams per centimetre. However, it is commonly expressing it in units of kilograms per cubic metre.(8)

It is an important property because it lets us know whether the substance will float or sink when it is placed in a liquid.(9)

Viscosity

This property indicates how much resistance a fluid to a change in shape has, or movement of

neighbouring portions relative to one another. It is a major factor in determining the forces that must be overcome when fluids are used in lubrication and transported in pipelines.

Dynamic viscosity is measured in newton-second per square meter or pascal-second in SI units. It decreases very quick when the temperature increases if the substance is in liquid phase and the opposite when it is in gas phase.(10)

Ionic liquids are more viscous than water. They are about 30-50 cP while water's viscosity is about 0.89 cP at room temperature (25°C). Even though the ILs are tunable, if one salt $[Li^+][X^-]$ is added to one ionic liquid $[A^+][X^-]$, the resulting compound will have higher viscosity.(11)

Therefore, this a big drawback when we are using them as solvents.

Viscosity is measured by a viscometer. This equipment records how much does it take for a given volume of fluid to flow through an opening.



Figure 3. Example of viscometer.(12)

Conductivity

During this thesis we will only refer to electrical conductivity. In those terms, we can define electrical conductivity as the current or the quantity of electricity passing per second through a similar slab when the potential gradient is unity, and it is the reciprocal of the resistivity. (13)

Room temperature conductivity of aprotic Room Temperature Ionic Liquids (RTILs) is within a broad range of $0.1\text{--}18\text{ mS} \cdot \text{cm}^{-1}$. Besides, those ionic liquids whose cation is Li^+ usually have conductivities around $14\text{ mS} \cdot \text{cm}^{-1}$. If we did the same example I used for viscosity, we would notice that the conductivity lowers and viscosity increases.(11)

It is a quite useful parameter to measure when undertaking environmental of process monitoring, especially when we want to determine how is the quality of a water sample.

Whether we know its value, we can obtain the Total Dissolved Solids (TDS) and salinity.(14)

2.3 Battery performance

Li-ion batteries have two compartments which are called cells. Each of those cells has, at least, the following elements: Positive electrode (connected to the battery's positive terminal), negative electrode (connected to the battery's negative terminal) and the electrolyte. The positive electrode is frequently made of lithium iron phosphate (LiFePO_4) and the negative one is made of graphite. Therefore, in this bachelor thesis those compounds were both utilized.

When the battery is charging up, the positive electrode drops some Li^+ ions. They move through the electrolyte to negative the negative one and remain there. Whether the battery is discharging, Li^+ ions travel to the positive electrolyte and generate the energy that powers the battery.

Both ion movements depend on each other, which means that if one of them stops, you will lose all the power. However, this thing only happens when the battery is completely discharged and it will stop at a high rate.(15)

The following figure shows a scheme of what has been explained on the previous paragraphs:

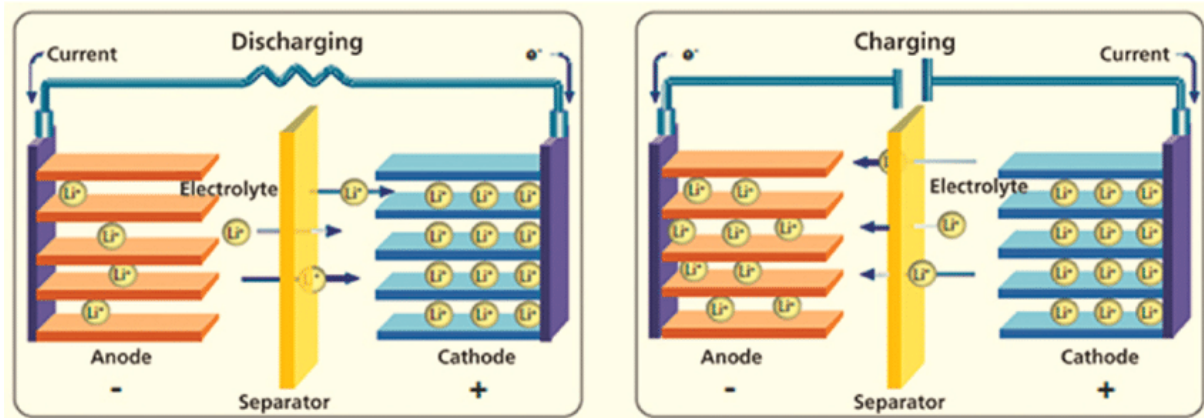


Figure 4. Scheme of Li-ion battery performance.(16)

2.4 Battery testing

First, it is necessary to define some concepts that are relative to this topic:

1. **Voltage:** It is usually defined as the ‘pressure’ that pushes electricity and it is measured in volt (V).(17)
2. **Current:** Rate at which electrons flow past a point in a complete electrical circuit. It is frequently measured in ampere (A), which means the quantity of electrons that flow on an specific point over a given time.(18)
3. **Battery Capacity:** Total amount of electricity generated by electrochemical reactions in the battery. Its units are Amp-hours (Ah), which shows how much current can a battery deliver in 1h until its voltage drops to a specific value for each cell.
4. **State of Charge (SoC):** Indicates the level of charge relative to the maximum capacity of the battery.
5. **Depth of Discharge (DoC):** Indicates the percentage of the battery that has been discharged.
6. **State of Health (SoH):** Takes into consideration the battery’s storage capacity when compared to its initial capacity, the internal resistance, and self-discharge due to stress-related conditions on the battery.(19)

In Figure 5 it is shown a scheme that makes easier to difference SoC, DoD and SoH:

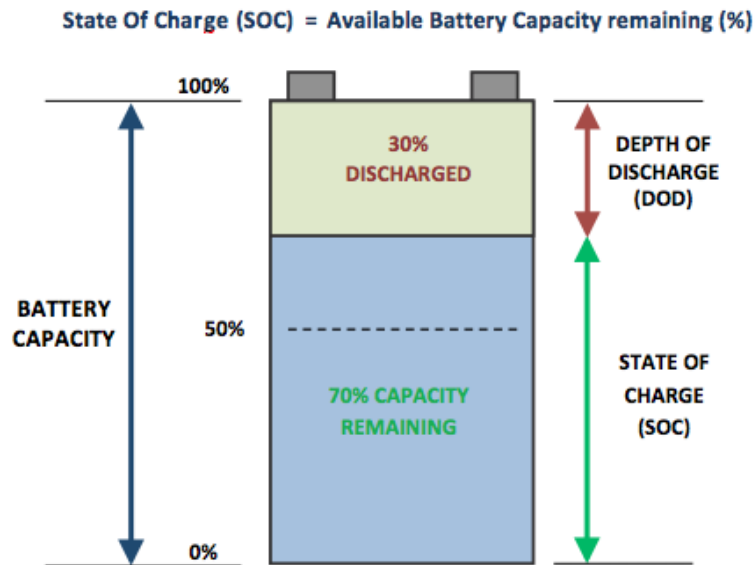


Figure 5. Battery capacity diagram.(20)

The most important battery test methods are:

- **Voltage:** It reflects the SoC in an open circuit condition when the battery is rested. Nevertheless, this method is not very useful to determine the SoH.
- **Ohmic test:** It is also known as an impedance test, which consists of measuring internal resistance. It is an interesting test to detect corrosion and mechanical effects.
- **Full cycle:** In this test, the battery is charged and discharged to evaluate its capacity.
- **Rapid test:** The battery is activated with pulses to observe ion-flow Li-ion, and frequency domain by scanning it with multiple frequencies.
- **Battery Management Systems (BMS):** For Li-ion batteries, it consists of monitoring voltage. Current and temperature.
- **Coulomb counting:** The Full Charge Capacity (FCC) of a smart battery provides coulomb count that relates to SoH. FCC readout is instant, but the data gets inaccurate with use and the battery requires calibration with a full cycle.
- **Read and Charge:** It reads SoC with a proprietary filtering algorithm and then counts the coulombs to fill the battery. For this test, it is necessary to do a onetime calibration for each battery model.
- **State of Life Indicator (SOLI):** It estimates battery life by counting the total coulombs a battery can deliver in its life.(21)

For this bachelor thesis, the method that has been used is the full cycle test as it will be

explained on the 'methods' section.

2.5 Thermogravimetric Analysis (TGA)

On this project, the method that has been used for measuring the electrolyte physical properties is called Thermogravimetric Analysis (TGA). It can be determined most of them, for example thermal stability, oxidative stability, effects of different atmospheres, moisture and volatile content.(22)

This technique consists of heating one small sample of the electrolyte (1 or 2 tiny drops) and permit us to determine the material's thermal stability and its fraction of volatile components by monitoring the weight change that occurs as a sample is heated at a constant rate.(23) However, it is important that the instrument can make a mass and temperature calibration. Most of them consist of working out the Curie point, which is the temperature when the material loses its magnetic properties. To determine it, a strong magnet must be placed below or on top of the furnace the cause an initial weight gain or loss at room temperature.(22) This performance can be done either horizontally or vertically as it shows the following schemes:

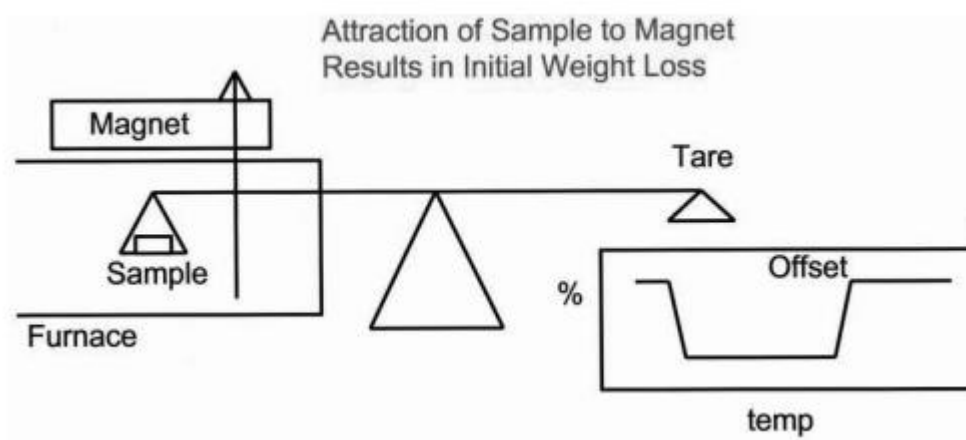


Figure 6. Horizontal temperature calibration configuration.(22)

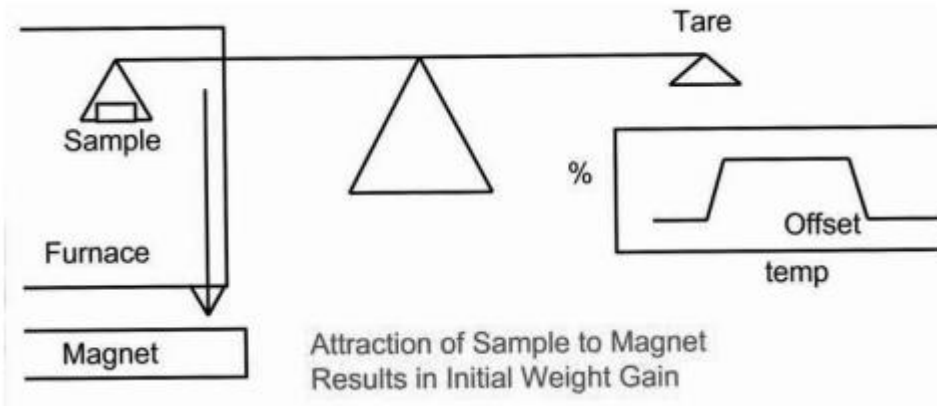


Figure 7. Vertical temperature calibration configuration.(22)

3. Materials and methods

3.1 Materials

Instruments:

- Battery test system
- TGA equipment

Equipment:

- Doctor blade
- Automatic pipettes
- Magnets
- Al foil
- Cu foil
- Scissors
- Crimping machine
- Cutting machine into round pieces
- Spoons
- Tweezers
- Laboratory spoons-spatulas
- Beakers
- Weight scale

Chemicals:

- Lithium sulfate ($LiSO_4$)
- Titanium dioxide (TiO_2)
- Carbon black (CB)
- Carboxymethyl Cellulose (CMC)
- $LiPF_6$
- Ethylene Carbonate (EC)
- Dimethyl Carbonate (DMC)
- Iron (Fe)
- Iron sulfate III ($FeSO_4$)
- Lithium Hydroxide (LiOH)

3.2 About the chemicals utilized in the electrolyte

The following chemicals were utilized to prepare the electrolyte that I used on my experiments:

1. Dimethyl Carbonate (DMC)

Formula: $C_3H_6O_3$

Molecular weight: 90.08 g/mol

Melting point: 2-4°C (24)

Density: 1.069 g/cm^3 at 25°C

Boiling point: 90°C

Flash Point: 16.7°C – closed cup

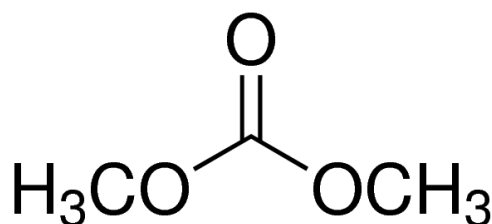


Figure 8. Molecular structure of the DMC.(25)

Dimethyl Carbonate has a liquid form at room temperature, and it is a colorless compound. Its principal characteristics are: nontoxic, flammable, it smells like methanol but does not have any mutagenic or irritating effects either you smell or inhale it. Besides, it has a versatile and tunable chemical reactivity depending on the reaction conditions, especially when it is in presence of a nucleophile.

Normally the reaction conditions are not green, since DMC's methylating ability requires high temperatures (about 160°C) and that means autogenic pressure (>3 bar).(26)

2. Ethylene Carbonate (EC)

Formula: $C_3H_4O_3$

Molecular weight: 90.08 g/mol

Melting point: 35°C

Density: 1.321 g/cm³ at 20°C

Initial boiling point and boiling range: 243-244°C at 987 hPa

Flash point: 143°C – closed cup

Vapor pressure: <1 hPa at 20°C

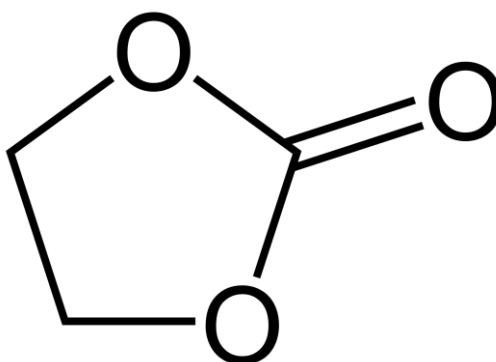


Figure 9. Molecular structure of the EC.(27)

This compound is white at room temperature but when its temperature is above the melting point it seems colorless. Ethylene carbonate is a highly polar solvent and dissolves large amount of electrolyte, it is mainly used in lithium batteries electrolyte solution. It can also readily dissolve polymers leading to use as a release agent and detergent.

EC contributes greatly to both surface parasitic reactions performance fading and self-heating before thermal runaway (TR). That lead many researchers to investigate more about 'EC-free' electrolytes, which are cheaper, less viscous, and more environmentally friendly. However, electrochemical performance of those electrolytes has to be more investigated.(28)

3. Lithium hexafluorophosphate

Formula: LiPF_6

Molecular weight: 151.91 g/mol

Melting point: 200°C

Density: 2.83 g/cm^3 at 25°C

Flash point: Not applicable

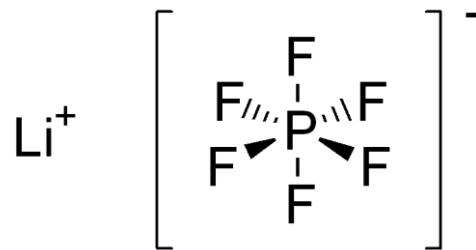


Figure 10. Chemical structure of lithium hexafluorophosphate.(29)

This compound is used frequently as the electrolytic solution in lithium-ion batteries. It has an excellent conductivity even though it does not stand out in terms of ionic mobility and dissociation constant when comparing it to other ionic liquids:

Average ion mobility: $\text{LiBF}_4 > \text{LiClO}_4 > \text{LiPF}_6 > \text{LiAsF}_6 > \text{LiTf}$

LiIm Dissociation constant: $\text{LiTf} < \text{LiBF}_4 < \text{LiCl}_4 < \text{LiPF}_6 > \text{LiAsF}_6 < \text{LiIm}$ (30)

3.3 Methods

3.3.1 Cathode preparation

The reactants that were used to carry out these experiments were: Carboxymethyl Cellulose (CMC), Carbon Black (CB) and a powder mixture which consisted of lithium hydroxide, iron, and iron (III) phosphate. However, at the beginning I tried to use $LiSO_4$ and TiO_2 but it did not success as it will be shown later.

To prepare the cathode, several mixtures with different ratios were prepared to determine which one is the best for the batteries' performance. The total amount of reactants was 3 g, regardless of the ratio used. The following ratios (CMC: Powder: CB) were prepared:

Table 1. Ratios for cathode preparation.

Date	Compounds	Ratio
07/02/22	CMC + CB + $LiSO_4$	1:1:8
10/02/22	CB + $LiSO_4$ + CMC + TiO_2	1:5:1:3
17/02/22	CMC + CB + Powder	1:1:8
31/03/22	CMC + CB + Powder	3:1:6
01/04/22	CMC + CB + Powder	2:2:6
04/04/22	CMC + CB + Powder	3:5:2

Firstly, CMC is added to the vial and 1 mL of water to get it dissolved. The sample starts to be stirred for, approximately, 12h. In the meanwhile, it is important to add more millilitres (about 2 or 3 mL) of water to get a slurry. After that, the powder and the CB are added to the mixture. After waiting for 1 day, the sample is spread on an aluminium (Al) foil by using a spoon and the doctor blade. Finally, the foil is laid inside of an oven at 80°C for 1 day.

CMC is usually utilized to prepare Li-ion batteries because it is more environmentally friendly, not too expensive, and also does not affect to the intercalation of Li^+ in the graphite

layer or the formation of solid electrolyte interface (SEI) when the battery is being charged and discharged.(31)

Carbon Black is quite used on Li-ion batteries because some studies have shown that it has a large irreversible capacity when cycled in the low-potential region. (32)

3.3.1.1 Powder mixture preparation

To prepare the powder, I used the following reactants: lithium hydroxide (LiOH), iron (Fe) and iron (III) phosphate ($FePO_4$). The sample was in total 5.5729 g and the reactants' amounts were:

Table 2. Reactants and quantities for the powder mixture preparation.

Compound	Quantity (g)
$FePO_4$	4.5239
Fe	0.3351
LiOH	0.7139

Iron phosphate (III) has some properties that make it a very attractive for Li-ion batteries:

- Good cycle life, which is higher than 500 cycles.
- Low cost.
- Low environmental impact.
- Stability.
- Temperature tolerance (-20°C to 70°C).

Nevertheless, it has some disadvantages comparing it with $LiCoO_2$:

- Low electronic and ionic conductivity ($10^{-10} \frac{S}{cm}$ and $10^{-8} \frac{cm^2}{s}$). (33)
- Relatively low capacity (170 mAh/g). (34)

Since the $FePO_4$ has a big particle size, it is necessary to smash the mixture by using a mortar. Finally, the powder is put inside of an oven at 250°C during 12 h since the boiling point of the LiOH is 462°C (35) and the value for $FePO_4$ is exactly 250°C (36).

3.3.2 Anode preparation

The method to prepare the anode is quite like the one that was used for the cathode. The same amount of sample is prepared (3g) but other ratios were used. The big differences are that the layer is made of Cu and graphite were utilized instead of Al foil and the powder mixture respectively.

Thus, the reactants that were used for this step were: Carboxymethyl Cellulose (CMC), Carbon Black (CB) and Graphite. The ratios that were utilized were the following ones:

Table 3. Ratios for the anode.

Date	Compounds	Ratio (%)
04/02/22	CMC + CB + Graphite	3:1:96
30/03/22	CMC + CB + Graphite	10:1:89

3.3.3 Battery preparation

The elements that are needed to build one Li-ion battery (apart from the anode and the cathode) are the following ones:



Figure 11. Slide.



Figure 12. Positive case.



Figure 13. Negative case.



Figure 14. Separator.



Figure 15. Ring.

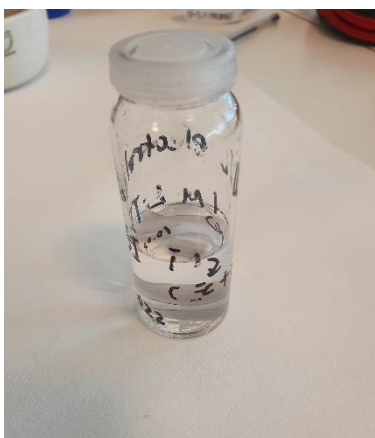


Figure 16. Electrolyte.

The electrolyte solution consisted of a 1 M solution of $LiPF_6$ in ethylene carbonate (EC) and dimethyl carbonate (DMC) instead of diethyl carbonate (DEC) as it is said on literature.(37) I did not prepare the solution. It was given to me by my supervisor.

Therefore, the order to assemble the coin cell -starting from the bottom- is:

1. Positive case
2. Slide
3. Cathode
4. Separator + electrolyte drops
5. Anode
6. Slide
7. Ring
8. Negative case

There are some aspects that are relevant when building the battery. The first one is that it is recommendable not to use too much electrolyte drops, since it could affect to their performance. Here below it is shown a scheme of how the battery is constructed, even though that the cathode is not made of $LiMn_2O_4$.

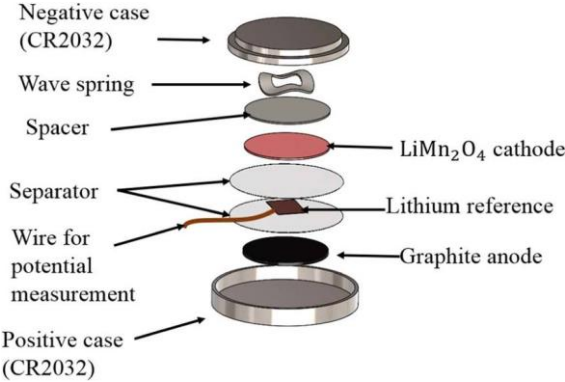


Figure 17. How to build a Li-ion battery.(38)

3.3.4 Battery test system

The batteries were tested on the following equipment:

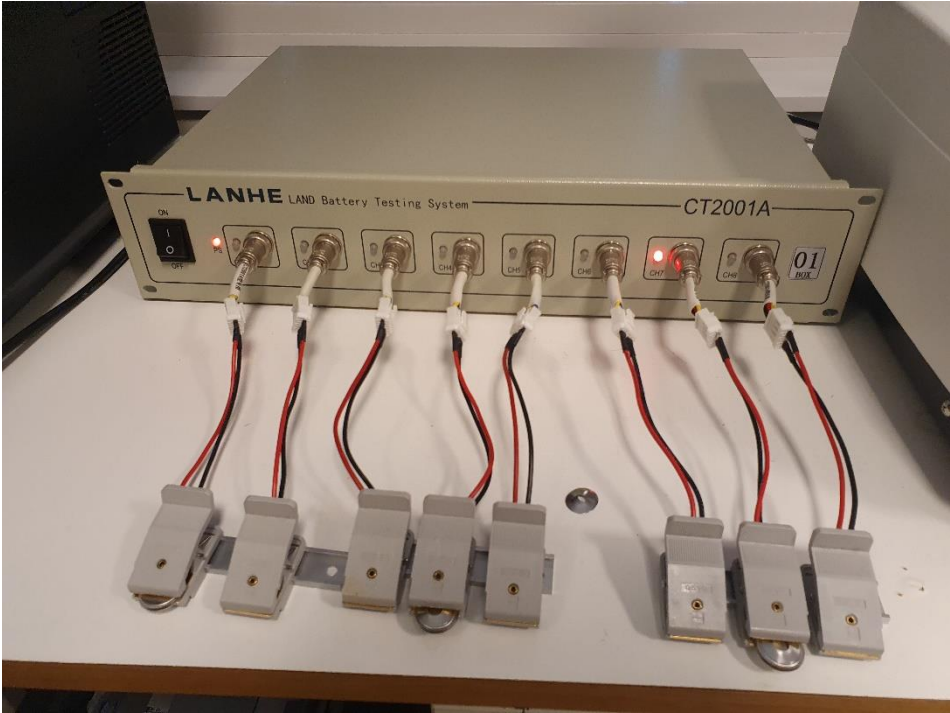


Figure 18. Battery test system.

As it is shown on Figure 18, it is possible to test 8 batteries at the same time. Every channel must have their operating conditions defined, which can be either the same for all the batteries or not.

The equipment is connected to one computer where folders can be created to store the results. Besides, it is necessary to define the operating conditions. They can be defined by using steps that are relative to the following parameters: voltage, current and number of cycles. During my experimental work, the values for testing the batteries were:

Table 4. Operating conditions.

Parameter	Value
Voltage range (V)	2.8-3.8
Current (mA)	1
Number of cycles	100

And the steps that were defined:

Table 5. Battery test system's steps.

Step	Condition	End condition	Action
1	Rest	-	Next step
2	Charge CC: 1 mA	Voltage \geq 3.8 V	Next step
3	Charge until 3.8 V	Current \leq 0.5 mA	Next step
4	Discharge CC: 1 mA	Voltage \leq 2.5 V	Next step
5	If	Number of cycles \geq 100 Times	Go to step 2
	Or	-	END

None of the parameters were changed during all the performances, especially the voltage

whose nominal value is 3V for this kind of batteries.(39)

3.3.5 Electrolyte properties

The TGA equipment that has been used on the lab is the following one:



Figure 19. Thermogravimetric equipment (TGA).

The cylinder is the place where the sample is introduced and then it starts to be heated on a given conditions. The software requires a setpoint – the maximum temperature that we want to achieve- and how fast the sample is going to be heated (for example, 20°C/min). Moreover, one gas flow must be used for the performance, which was oxygen for the heating process and nitrogen to cool it down.

4. Results and discussion

4.1 Cathode

During February, I have only prepared cathodes since the anodes were given to me by my supervisor.

As I said on Table 1, the first ratio that I tried was (1:1:8), which means CMC: CB: LiSO_4 . The following quantities were added:

Table 6. Quantities for the cathode (5th of February).

Compound	Theoretical quantity (g)	Real quantity (g)
CMC	0.3	0.2979
CB	0.3	0.3054
LiSO_4	2.4	2.4021
Water	4 mL	

The following slurry was obtained:



Figure 20. Cathode (5th February 2022).

As it is shown on Figure 21, the slurry was not attached to the Al surface. I thought that this

result was due to the excess of water and the lithium sulphate. Therefore, my supervisor suggested me that I would get better results if I added titanium dioxide. The quantities for this experiment were:

Table 7. Quantities for the cathode (10th of February).

Compound	Theoretical quantity (g)	Real quantity (g)
CMC	0.3	0.3017
CB	0.3	0.3066
LiSO ₄	1.5	1.5009
TiO ₂	1	1.006
Water	3 mL	

The reactants' order was CMC, TiO₂, CB and LiSO₄. Nevertheless, I got this slurry:



Figure 22. Cathode (14th February 2022).

A priori, it seemed that the slurry was good since it was completely attached to the Al foil. Thus, I decided to cut some slides to try my first battery. However, after a couple of hours the capacity was still 0 which meant that something was wrong.

Those results led me to the use of the powder mixture that I explained on the 'cathode preparation' section and to add the CB and the powder almost at the same time as CMC. All

the samples I prepared had this appearance:



Figure 23. Powder mixture (17th February 2022).

The following quantities were utilized to prepare the powder:

Table 8. Powder preparation (17th February 2022).

Compound	Quantity (g)
$FePO_4$	4.5239
Fe	0.3404
LiOH	0.714

And the slurry that I obtained looked so much better and worked better when I used it to prepare one battery:



Figure 24. Cathode slurry (22-02-22).

After that cathode, I kept preparing the same ratio during March without success. That led me to consider the possibility of changing it for the ones on Table 9.

The 31st of March, I prepared one cathode with the following quantities:

Table 9. Cathode quantities (31-03-22).

Compound	Theoretical quantity (g)	Real quantity (g)
CMC	0.9	0.9017
CB	0.3	0.3025
Powder	1.8	1.798
Water	2 mL	

Before I had added the powder and CB, I realized that amount of CMC was massive and thus I decided not to add them. The cellulose got dissolved quickly even though I did not utilize as much water as the previous experiments.

During the first weeks of April, I changed to some other ratios just to see how the CMC quantity influences the performance of the battery. None of those ratios gave me any

interesting results but I noticed that the stirring time – and which step was the water added- has something to do with the previous failures. Therefore, I decided that the next samples would be stirred only until the CMC is dissolved -which is about 30 min approximately- and some part of the water would be added after adding the powder and the CB just to make sure that everything gets perfectly dissolved.

So, during the first week of May, I prepared some batteries with a 2:6:2 ratio. The most relevant one was the following:



Figure 25. Cathode slurry (03-05-22).

And these was the amount of each reactant:

Table 10. Cathode quantities (02-05-22).

Compound	Theoretical quantity (g)	Real quantity (g)
CMC	0.6	0.598
CB	0.6	0.599
Powder	1.8	1.806
Water	2 mL + 1 mL (after CMC) + 1 mL (after adding CB)	

As it will be explained on the 4.3 section, the cathode is mainly responsible for the battery failures.

4.2 Anode

At the beginning of my experimental work, I was given the anodes that are shown in Figure 26.



Figure 26. Example of anode.

Therefore, all the investigation was focused on preparing cathodes from the 4th of February until the 21st of March. That day I prepared one anode whose quantities of reactants were:

Table 11. Anode quantities (21st March).

Compound	Theoretical quantity (g)	Real quantity (g)
CMC	0.09	0.0899
CB	0.03	0.0336
Graphite	2.88	2.8798
Water	3 mL	

The results were not good because the sample was not completely attached to the Cu foil as it is shown on the following images:



Figure 27. Anode (21st March).



Figure 28. Failed anode (21st March).

After that, another anode was prepared the 30th of March. This time I considered changing the ratios to: 89% graphite, 10% CMC and 1% CB. The following quantities were added, and the results were great:

Table 12. Anode quantities (30th March).

Compound	Theoretical quantity (g)	Real quantity (g)
CMC	0.09	0.0899
CB	0.03	0.0336
Graphite	2.88	2.8798
Water	1.75 mL	

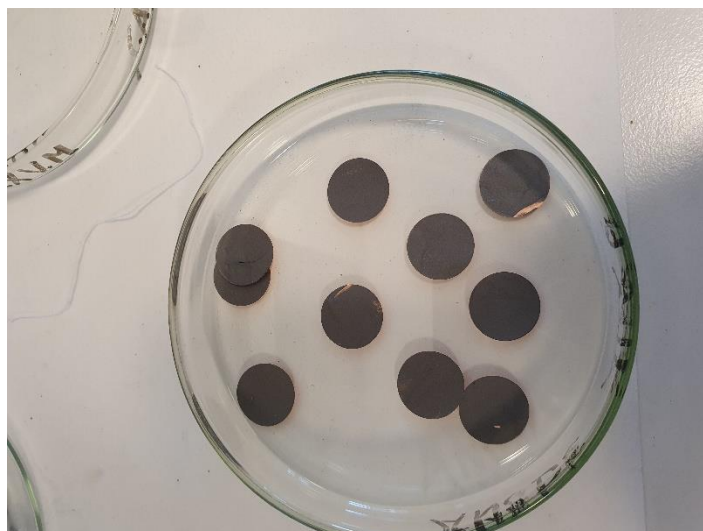


Figure 29. Anode (30th March).

That anode gave me better results since the graphite surface is perfectly attached to the copper foil. Therefore, the problems about the batteries' performance are more related to the cathode building procedure.

Finally, one anode sample was made on the 5th of May and the following quantities were utilized:

Table 13. Anode quantities (5th May).

Compound	Theoretical quantity (g)	Real quantity (g)
CMC	0.09	0.0899
CB	0.03	0.0336
Graphite	2.88	2.8798
Water	1 mL + 1.5 mL (after adding CB and graphite)	

For this anode, the reactants were added almost at the same time, but the sample was laid stirring for one day and then spread on the Cu foil with the doctor blade. After a couple of days and cutting the slurry into round pieces, this was how it looked like:

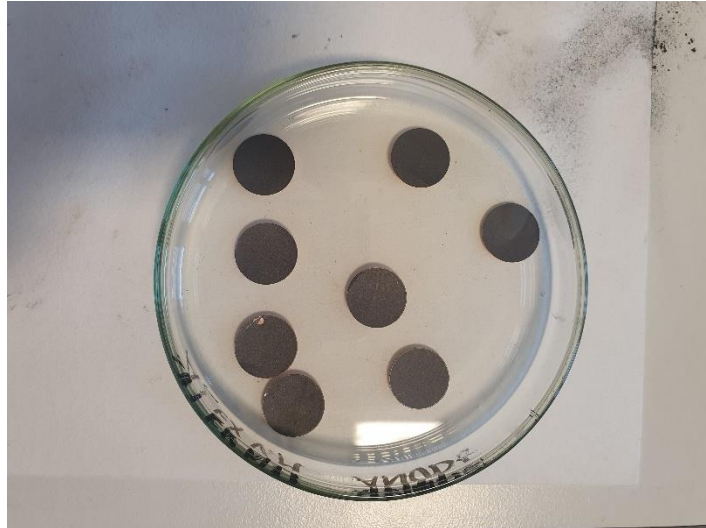


Figure 30. Anode (5th May).

Therefore, the results for the anode are quite good.

4.3 Battery performance

The first battery that gave me reasonable results -which means that its capacity was higher than 0- was crafted using the cathode from the 7th of February and one of the anodes that were given to me. Here below it is shown how the battery properties evolved during the testing process:

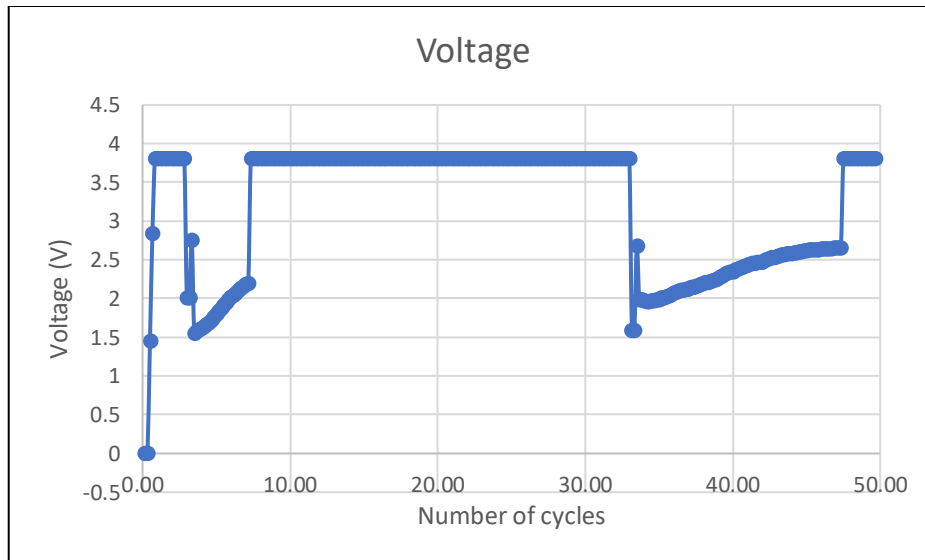


Figure 31. Evolution of battery's voltage (22-02-22).

In Figure 31, the voltage is usually around 3.8 V, which means that the battery is not able to discharge by itself. When the step is changed manually, the voltage goes down until the value of 1.5 V. After that, the value increases until the previous one. That means the battery has been discharged.

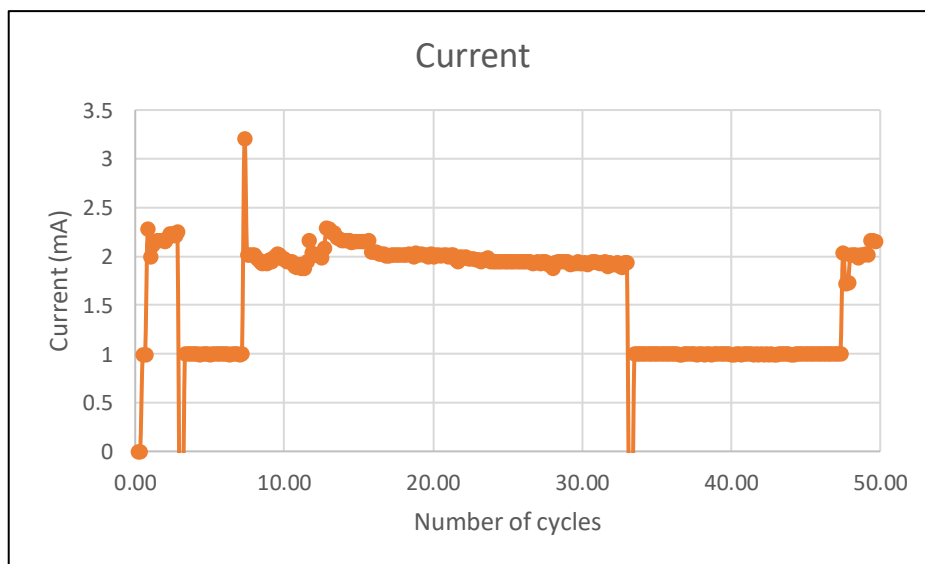


Figure 32. Evolution of battery's current (22-02-22).

In Figure 32, it is shown that the current trend is like the voltage. It increases until its ca. 2.3 mA when the battery is on step 3. However, most of the time the current is around that value and only decreases when the step is changed manually.

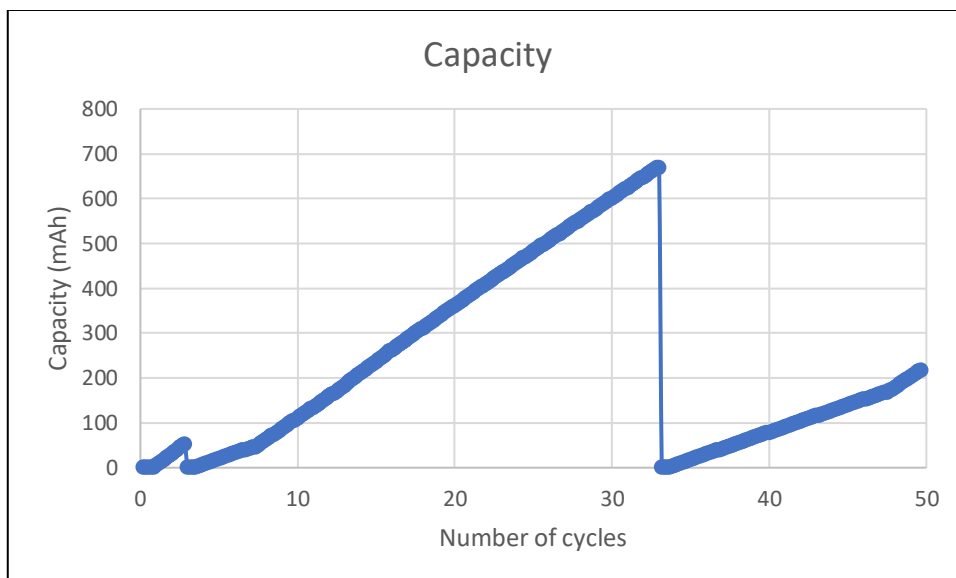


Figure 33. Evolution of battery's capacity (22-02-22).

About the capacity, Figure 33 shows that it is too high for a Li-ion battery coin cell. Some companies produce and sell batteries whose nominal capacity goes from 25 to 180 mAh (and also depends on their size).(40) Thus, it has no sense that the capacity reaches values up to 750 mAh.

Firstly, I thought that the particle size of the powder was huge as it is shown on Figure 24. To solve this, I tried to smash the powder during ca. 5 min and set the doctor blade to 40 μm . Besides, I decided to change the cathode and anode ratios for the ones on Table 10 and Table 13 respectively. The results were the following graphs, where the parameter 'index' is a synonym for 'number of cycles':

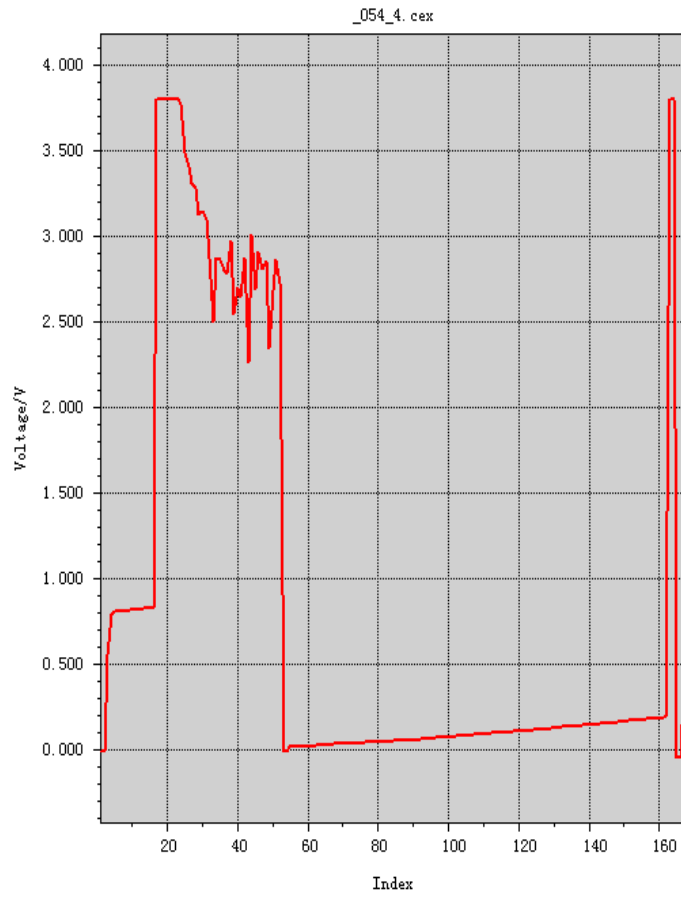


Figure 34. Evolution of battery's voltage (05-05-22).

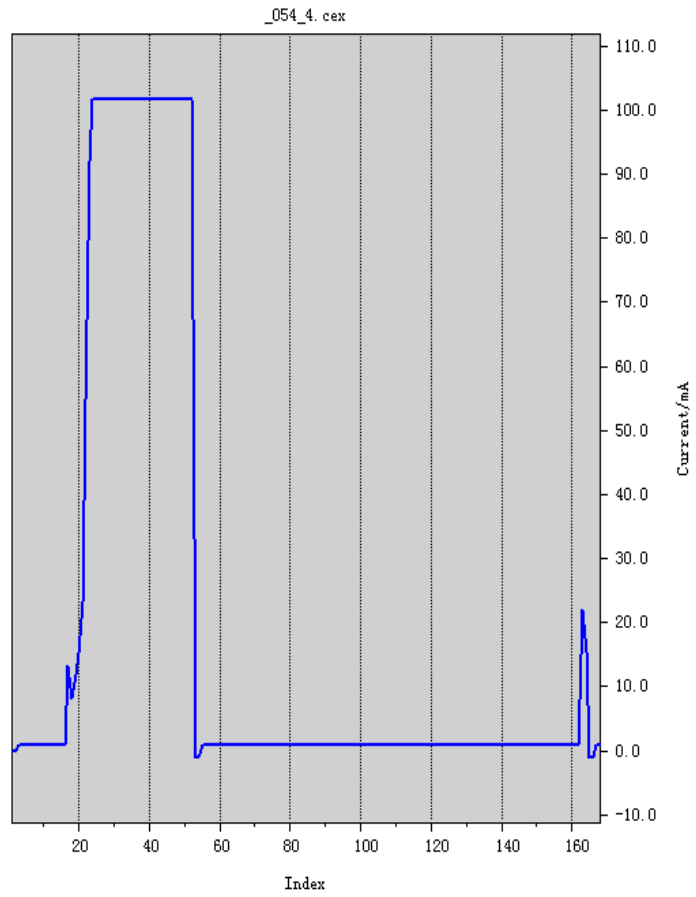


Figure 35. Evolution of battery's current (05-05-22).

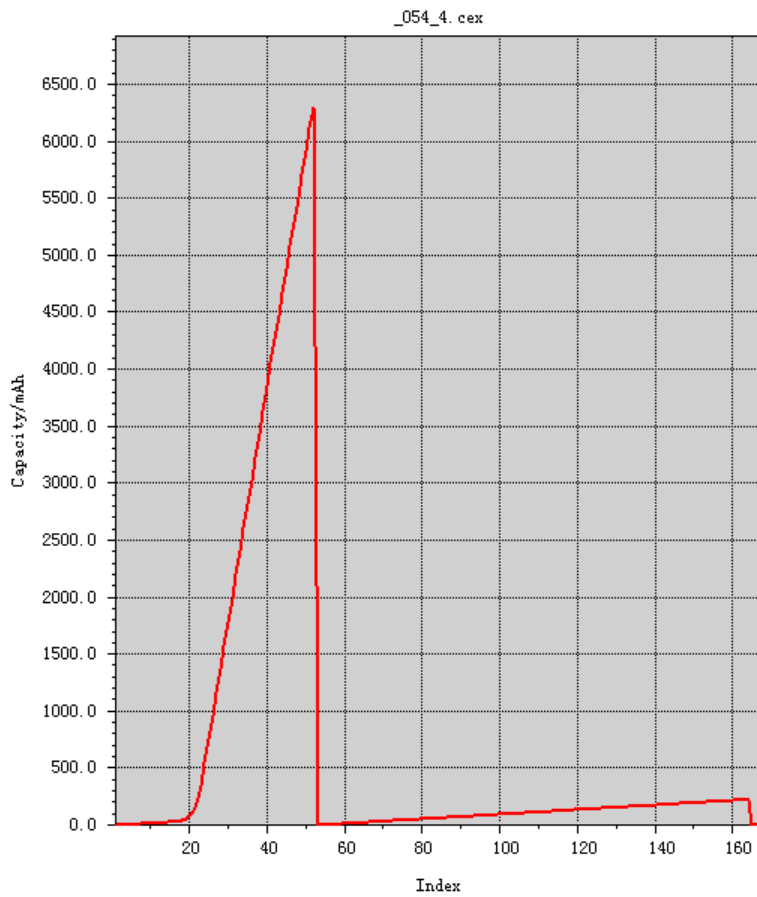


Figure 36. Evolution of battery's capacity (05-05-22)

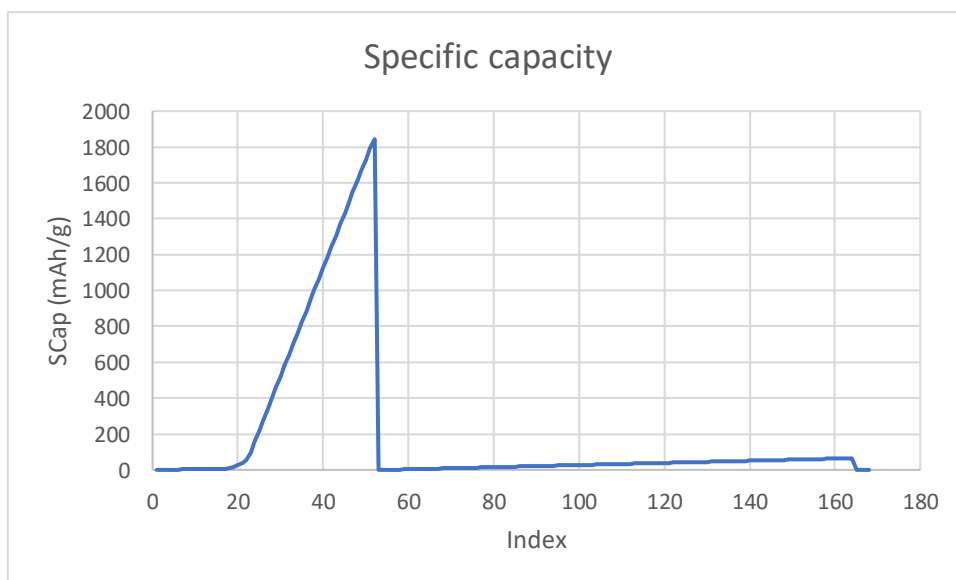


Figure 37. Evolution of specific capacity (05-05-22).

The previous graphs showed that this battery coin cell has the same problem as the other one,

but it has slightly improved. As shown on the Figure 34, the voltage does not decrease as fast as it was on the other battery. Thus, the battery can discharge in a non-drastric way. However, as it is shown on Figure 31, the change of its value from ca. 2.7 V to 0 V only happened because I made the equipment jump into the next step.

On the current graph (Figure 35) also justifies that something is not working properly. Its value remains approximately constant for several cycles, which means that the battery is most of the time on step 2.

Since the battery test system does not measure the specific capacity and most of the literature, it was crucial to find out how to calculate it. To do this, the coin cell was weighted on a weighing scale. Its value was 3.145 g and that made it possible to work out that parameter, which was calculated with the following equation:

$$SCap \left(\frac{mAh}{g} \right) = \frac{Capacity (mAh)}{mass \text{ of the coin cell } (g)} \quad (2)$$

Moreover, the trend of the specific capacity is not normal. Figure 37 shows that its value grows almost exponentially until 1800 mAh/g and suddenly decreases to near zero. According to other researchers, using the same electrolyte (1 M $LiPF_6$ in EC: DMC 1:1) and $LiFePO_4$ as the cathode material, its theoretical value is 170 mAh/g and 100-140 mAh/g when synthesized by direct solid state.(41) Thus, it is very unlikely that the battery is working well since the difference between those values is huge.

Changing ratios for the cathode preparation (Table 9) and Table 13 for the anode have improved the battery's behaviour. Nevertheless, it is quite probable the problems about the battery performance have something to do with the cathode. It is quite likely that the parameter that has the highest influence is the particle size of the powder. As I said on 3.3.1.1 section, it was my supervisor who told me about that method. However, I have not found any articles describing that method and thus I cannot end up in a conclusion. Apart from that, I realized that the best ratio -and thus the best CMC amount- is 0.6 g because the reaction does not take place if we add more than that quantity.

4.4 Electrolyte properties

It was not done any experiment related to this section because the Thermogravimetric Analysis equipment was broken. However, it is possible to compare the boiling point of EC and DMC – which are the main components of the ionic liquids that was used for the preparation of the batteries- with the decomposition temperature of some other commercial ionic liquids such as *BmimPF₆* and *BmimBF₄*. Here below it can be seen the molecular structure for those ILs:

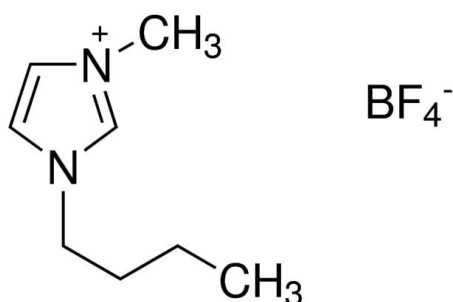


Figure X. BmimBF₄ structure.(42)

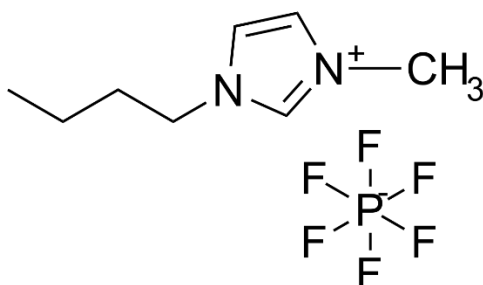


Figure X. BmimPF₆ structure.(43)

The decomposition temperatures for *BmimBF₄* and *BmimPF₆* – when they start to decompose- are 216°C and 248°C respectively.(44) On the other hand, the boiling temperature for EC and DMC are respectively 240°C and 90°C as it was mentioned on section 3.2.

This means that, as expected, ILs are more stable solvents than the conventional ones. Even though DMC and EC are also quite stable at room temperature, it would be better using ILs if

possible, especially *BmimPF₆* which was the highest decomposition temperature (and usually gets totally evaporated at 461 °C).(44)

5. Conclusion

In this thesis, I have prepared anode and cathode for Li-ion battery fabrication. In addition, some of the articles that were quoted on this thesis have shown that ionic liquids can increase the security and greenness of Li-ion batteries because of their excellent solvent properties. We tried also to utilize ionic liquid in Li-ion battery electrolyte preparation in this work. In the end, a full Li-ion battery was produced in our laboratory. The performance of the Li-ion battery was tested using battery testing system.

As it was explained on the 3.3.1 and 4.1 section, the reactants for the cathode preparation must be CMC, CB, and the powder. The ratio should be 2:6:2 – 3g in total-. However, it is important to smash it as much as possible so that it gets perfectly stuck to the Al surface. It has also been proved that this preparation can be done at room temperature instead of putting the foil inside of an oven, which is better in terms of Green Chemistry. Some other parameters such as stirring time might have influenced the quality of our cathodes.

About the anode, the reactants are CMC, CB, and graphite. It was shown on section 4.2 that the best ratio is 10:1:89 respectively. For this preparation, all the reactants were added almost at the same time – when CMC got dissolved- and the results were quite good.

On the other hand, there are a couple of things that are quite important for the Li-ion battery preparation, The first one is that it is important not to use more than 3-4 small electrolyte drops since some of our batteries did not success because of that. The other thing is that it is crucial adding all the compounds for the battery in the same way it was explained on 3.3 section. Some of our first batteries failed only because of that.

Finally, I did not find any difficulties with the battery test system. The conditions were suitable for the Li-ion batteries according to literature.

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7. Appendix

In this section can be found all the information relative to the chemicals that I used during my experimental work. Those who are not included in this section are not hazardous.

Product name: Iron (Fe)

Pictograms:



Signal word: Danger

Hazard statements:

H228: Flammable solid.

H251: Self-heating; may catch fire.

Precautionary statements:

P210: Keep away from heat, hot surfaces, sparks, open flames and other ignition sources. No

smoking.

P235: Keep cool.

Product name: Lithium hydroxide (*LiOH*)

Pictograms:



Signal word: Danger

Hazard statements:

H302: Harmful if swallowed.

H314: Causes severe skin burns and eye damage.

Precautionary statements:

P260: Do not breathe dusts or mists.

P280: Wear eye protection/face protection.

P303+P361+P353 IF ON SKIN (or hair): Take off immediately all contaminated clothing. Rinse skin with water or shower.

P305+P351+P338 IF IN EYES: Rinse cautiously with water for several minutes. Remove contact lenses, if present and easy to do. Continue rinsing.

P321: Specific treatment (see on this label)

P501: Dispose of contents/container to industrial combustion plant.

Product name: Lithium hexafluorophosphate (*LiPF₆*):

Pictograms:



Signal word: Danger

Hazard statement(s):

H301: Toxic if swallowed.

H314: Causes severe skin burns and eye damage.

H372: Causes damage to organs (Bone, Teeth) through prolonged or repeated exposure if inhaled.

Precautionary statement(s):

P260: Do not breathe dusts or mists.

P280: Wear protective gloves/ protective clothing/ eye protection/ face protection.

P301 + P310 + P330 IF SWALLOWED: Immediately call a POISON CENTER/ doctor.

Rinse mouth.

P301 + P330 + P331 IF SWALLOWED: Rinse mouth. Do NOT induce vomiting.

P303 + P361 + P353 IF ON SKIN (or hair): Take off immediately all contaminated clothing.

Rinse skin with water.

P305 + P351 + P338 IF IN EYES: Rinse cautiously with water for several minutes. Remove contact lenses, if present and easy to do. Continue rinsing.

Product name: Iron phosphate ($FePO_4$)

Pictogram:



Signal words: Warning

Hazard statements:

H315: Causes skin irritation.

H319: Causes serious eye irritation.

H335: May cause respiratory irritation.

Precautionary statements:

P261: Avoid breathing dust/fume/gas/mist/vapours/spray.

P280: Wear protective gloves/protective clothing/eye protection/face protection.

P305+P351+P338: IF IN EYES: Rinse cautiously with water for several minutes. Remove contact lenses, if present and easy to do. Continue rinsing.

P304+P340: IF INHALED: Remove person to fresh air and keep comfortable for breathing.

P405: Store locked up.

P501: Dispose of contents/container to hazardous or special waste collection point, in accordance with local, regional, national and/or international regulation.

Product name: Dimethyl carbonate (DMC)

Pictogram:



Signal word: Danger

Hazard statements:

H225: Highly flammable liquid and vapour.

Precautionary statements:

P210: Keep away from heat, hot surfaces, sparks, open flames, and other ignition sources. No smoking.

P403 + P235: Store in a well-ventilated place. Keep cool.

Product name: Ethylene carbonate (EC)

Pictogram:



Signal word: Warning

Hazard statements:

H302: Harmful if swallowed.

H319: Causes serious eye irritation.

H373: May cause damage to organs (Kidney) through prolonged or repeated exposure if swallowed.

Precautionary statements:

P301 + P312 + P330 IF SWALLOWED: Call a POISON CENTER/doctor if you feel unwell. Rinse mouth.

P305 + P351 + P338 IF IN EYES: Rinse cautiously with water for several minutes. Remove contact lenses, if present and easy to do. Continue rinsing.

P314: Get medical advice/ attention if you feel unwell.