

**SYNTHESIS AND CHARACTERIZATION OF
HIGH NICKEL CONTENT CATHODE
MATERIALS FOR HIGH PERFORMANCE AND
CAPACITY REACH IN LI-ION BATTERIES**

**A Thesis Submitted to
The Graduate School of Engineering and Science of
İzmir Institute of Technology
in Partial Fulfillment of the Requirements for the Degree of**

MASTER OF SCIENCE

in Chemistry

**by
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**July 2022
İZMİR**

ACKNOWLEDGMENTS

As I near the end of my master's thesis, I feel a sense of accomplishment and pride that has been building over the course of the last few years. To everyone who have supported and encouraged me along the way, I'd want to express my gratitude in this part.

First and foremost, I want to express my gratitude to Assoc. Dr. Engin Karabudak, my thesis adviser, for his wisdom, mentoring, and kind teacher-student connection.

The two members of my thesis committee, Associate Professors Ümit Hakan Yldz and Levent Pelit, deserve a special mention for their generosity in sharing their knowledge and expertise.

Yusuf Emre Göl and Ahmet Aytekin, members of the Karabudak research group, also supported me with my tests and study.

Last but not least, I want to convey my thanks and affection to my parents and siblings, who have never stopped supporting me and working hard to establish a brighter future for me throughout my educational career. And I would like to thank my friend Onur TEKİN from my undergraduate period for his morale-motivation and support.

ABSTRACT

SYNTHESIS AND CHARACTERIZATION OF HIGH NICKEL CONTENT CATHODE MATERIALS FOR HIGH PERFORMANCE AND CAPACITY REACH IN LI-ION BATTERIES

Due to their high energy density, low self-discharge properties, nearly negligible memory effect, high open-circuit voltage, and extended service life, lithium-ion batteries continue to gain interest as a promising energy storage technology. In the automotive industry, high-energy lithium-ion batteries have become the preferred power source for electric vehicles and hybrid electric vehicles in recent years.

With the development of lithium-ion battery technology, several materials have been used into the cathodes and anodes in order to improve performance. LiNiCoAlO_4 , LiMn_2O_4 , LiNiMnCoO_4 , $\text{Li}_4\text{Ti}_5\text{O}_{12}$ and LiFePO_4 are five lithium-ion batteries that are commonly utilized in commercial EVs today. NMC cathode material is one of the most effective lithium-ion battery materials for balancing specific qualities. The battery cathode of NMC is strengthened with a specific ratio of three synthetic components (Nickel, Manganese and Cobalt). Depending on the proportions of these three chemical constituents, battery performance can vary.

Synthesis, characterisation, and electrochemical studies of cathode materials with a high Nickel content were performed in this project in an effort to boost the specific capacity and durability of Li-ion batteries. In these preliminary studies, the synthesis and characterization of Ni(OH)_2 structures, which serve as a starting material for the synthesis of cathode materials with a high Nickel content, was also a goal. In the research, the spherical Ni(OH)_2 structure was effectively synthesized, and excellent electrochemical results were achieved. SEM and XRD analyses were performed on the resulting products.

ÖZET

Lİ-İYON BATARYALARDA YÜKSEK PERFORMANS VE KAPASİTE ULAŞIMI İÇİN YÜKSEK NİKEL İÇERİKLİ KATOT MALZEMELERİNİN SENTEZİ VE KARAKTERİZASYONU

Lityum-iyon piller, yüksek enerji yoğunluğu, düşük kendi kendine deşarj özellikleri, neredeyse sıfır bellek etkisi, yüksek açık devre voltajı ve uzun hizmet ömrü nedeniyle umut verici bir enerji depolama teknolojisi olarak dikkat çekmeye devam ediyor. Genel olarak, son yıllarda, yüksek enerjili lityum-iyon piller, otomotiv endüstrisinde elektrikli araçlar ve hibrit elektrikli araçlar için ideal güç kaynağı olarak kabul edilmiştir.

Lityum-iyon batarya teknolojisinin gelişmesiyle birlikte, daha iyi bir performans elde etmek için katotların ve anotların tasarımında farklı malzemeler kullanılmıştır. LiNiCoAlO_2 , LiMn_2O_4 , $\text{Li}_4\text{Ti}_5\text{O}_{12}$, LiNiMnCoO_2 , ve LiFePO_4 bugünlerde ticari EV'lerde kullanılan beş yaygın lityum-iyon bataryalarıdır. NMC katot malzemesi, belirli özelliklerini dengeleyen en başarılı lityum-iyon bataryalardan biridir. NMC katmanlı bir yapı içerir ve pil katodu belirli bir oranda üç sentetik bileşenle (Nikel, Manganez ve Kobalt) şiddetlenir. Bu üç kimyasal elementin farklı oranları değişken pil performanslarına yol açmaktadır.

Bu projede, Li-iyon pillerin hem spesifik kapasitesini hem de dayanıklılığını arttırmak için yüksek Nikel içerikli katot malzemelerinin sentezi, karakterizasyonu ve elektrokimyasal ölçümlerini gerçekleştirilmiştir. Bu amaçla yaptığımız ön çalışmalarda, yüksek Nikel içerikli katot malzemelerinin sentezi için bir başlangıç maddesi görevi gören Ni(OH)_2 yapılarının sentezi ve karakterizasyonu da amaçlanmıştır. Yapılan çalışmalarda, küresel Ni(OH)_2 yapısı başarıyla sentezlenmiş olup elektrokimyasal ölçümlerinde de tatmin edici sonuçlar elde edilmiştir. Elde edilen ürünlerin SEM ve XRD analizleri alınmıştır.

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CHAPTER 1

INTRODUCTION

1.1. The Reason For The Research

Nowadays, most of the studies focus on nickel rich cathode materials. NMC cathodes stand out from other cathode materials because of these materials show minimum danger to human health, they are easily available by an economic perspective. Moreover, they have excellent specific capacities in batteries. To the extent that, as a result of these qualities, its usage in many sectors of today's life has become fairly widespread, making it a contender for the top. Additionally, to the obvious aspects of NCA cathode materials, examples include the surface reactions than on compose during its usage, the degradation of the cathode material and the consequent fall in its lifespan. Our purpose for this study is to focus on increasing the lifespan and stability of the cathode materials.

1.2. Future of Energy Demand and Storage Technology

In today's world, there is a considerably greater need for electricity than there has ever been before. In addition, it must be utilized in the correct area and at the proper time. A more targeted and mobile approach to energy consumption management is required. In addition, it is necessary to improve the efficiency with which power is provided. Electrical energy may be converted into chemical energy with greater efficiency using electrochemical storage technology.

Capacitors and batteries can be used to store electrical energy. Since batteries have a poor molecular mobility during chemical processes, they take longer to charge and discharge than capacitors. Fuel cells may offer high energy with low power, whereas supercapacitors can provide greater power with less energy than a typical battery pack. The goal of high-battery systems is to maximize power capacity, particularly for automobile technology's electric motor systems.

The development of improved batteries and energy storage devices based on electrochemistry has received a lot of attention in recent decades. There are several reasons why this place is so popular. Internal combustion engines pollute the environment in a number of ways. Pollutant elements are released when fossil fuels are burned, which is well-known. When pollutants build up in nature, they act as both greenhouse gases and a source of harmful air chemicals that endanger human health.

The level of pollution is not uniform throughout the country. Cities with dense populations and active industrial zones tend to have higher levels. Emissions from the automobile sector have been regulated. Regulations such as this compelled the car industry to develop more environmentally friendly and efficient products. For vehicle propulsion systems, alternative fuels and energy storage medium should be developed. Using battery-powered electric cars as a transportation solution is an option. Hydrogen fuel cell can be the next step after that.

Enough power and energy must be fitted in the vehicle to offer enough acceleration and sufficient range on urban routes. As a result, a battery solution is required to provide the required quantity of power. The ability to reduce weight is also a critical consideration in automobile applications.

1.3. What is The Battery?

Electrochemical power sources, commonly known as batteries, are devices of directly converting the energy that is generated as a result of a chemical reaction into electrical energy. These devices can also be referred to by their other name, electrochemical power sources. The movement of electrons from one substance to another in the course of an electrical circuit is what causes these kinds of reactions to take place. There are two distinct classes of batteries that can be examined. The first kind, known as primary batteries, are designed to be used just once and then thrown away, while the second kind, known as secondary batteries, can be recharged and discharged multiple times (Aktaş, 2007). Today, it is feasible to create batteries in any size that a customer desires, and experts are constantly working to improve the functionality of batteries. In recent years, nickel cadmium, nickel metal hydride, lead acid, alkaline, and lithium-based batteries have been the types of batteries that have seen the most widespread use.

1.4. The Evolution of Batteries

The invention of the first battery dates back 200 years by Alessandro Volta (1745-1827). Its name is referred to by the unit of electrical potential, the volt. Volta's famous experiment involved the pili created by separating discs of silver and zinc with a piece of cloth saturated with salt water. In the battery, one end of which is terminated with a silver disc and the other end with a zinc disc, a continuous electric current is produced by connecting these two ends with a conductive wire. The first galvanic or primary battery designed in this way is known as the "Volta battery". Waldmar Jungner, a Swedish engineer, created the first Ni-Cd battery in the end of the seventeenth centuries. Then, in response to developing technology and increased demand, nickel-based batteries e.g. Ni-Zn batteries, silver-zinc batteries, Ni(OH)₂ batteries, and lithium batteries were developed. (Linden 2002).

Although they were first marketed in 1991, li-ion battery has since been utilized in a large range of applications including medical and military equipment as well as electronic devices that can be carried in a pocket (such as telephones, electronic watch and video cameras and laptop computers). Li-ion batteries can be used for many things., including in electric cars that are likely to take over automobile manufacturing in the near future. Lithium-ion batteries, as a result, are now found in practically every home, making them an essential part of modern electronics. Market growth for lithium-ion batteries is predicted to be 12.5% each year through 2024, from a 2015 high of \$30 billion. When it comes to energy storage, lithium-ion batteries have the biggest market share and most investments, thanks in large part to the growing usage of electric and hybrid cars (Pillot 2013). The usage of LiCoO₂ as a cathode material in consumer electronics has dropped since due to the fact that costly and hazardous of cobalt, even though the material was first marketed in 1991. (Goodenough and Kim 2010). The specific energy density of LiFePO₄s lags behind that of LCO, despite the fact that they are a safer cathode material. Both Li₂MnO₄ and LiFePO₄ were selected because of their high nominal voltages (Chen, et al. 2011). By the reason of its long cycle life and high energy density, LFP is utilized in electric cars while LCO is used in consumer electronics. For electric cars, two novel cathode materials, LiNiCoAlO₂ and LiNiCoMnO₂, have been discovered that have high capacities and nominal voltages (Nitta, et al. 2015).

Within the business of lithium-ion batteries, which sees significant levels of investment, NMC cathode materials rich in lithium are among the most consumed of all cathode materials. Because of their ability to function within a high voltage range, lithium-rich NMC cathode materials offer a high capacity in addition to a high energy density (Rozier, Tarascon 2015).

1.5. The Use of Lithium Ion Batteries

Li-ion batteries are classified as secondary batteries, which can be charged/discharged, in addition to this characteristic, they have a high energy density relative to their size and weight, so all of these properties have given a more popular to them. As a result of these qualities, they find applications in a variety of industries, including the defense industry, the medical profession, the automotive industry, and electronic devices. The gravimetric and volumetric energy densities of the secondary batteries are shown in Figure 1.1, it can be said that lithium-ion batteries have the greatest gravimetric and volumetric energy densities of all of the secondary batteries when considered Fig.1.1. In the periodic table, Li has more oxidation potential, low atomic weight as metal, and it also has roughly a specific capacity of $3680 \text{ Ah}(\text{kg})^{-1}$ because of its electropositivity. These properties give an advantage to lithium metal to be used in battery systems.

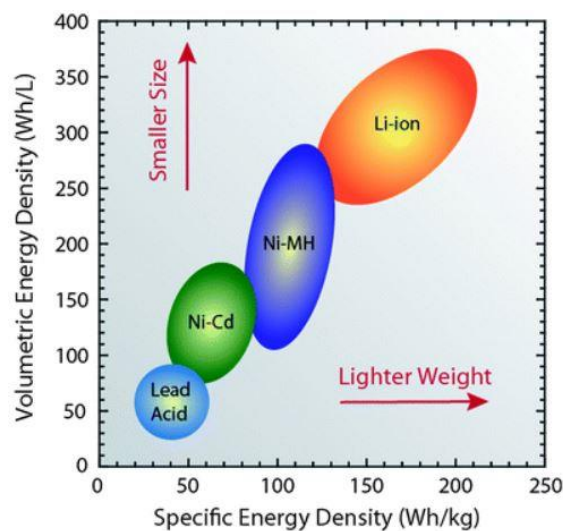


Figure 1.1. Gravimetric and volumetric energy density for secondary batteries (Source: Epec Energy Tech., 2022)

Li-ion batteries, in comparison to other secondary batteries, do not exhibit a memory effect. There is no considerable loss of capacity if they are recharged before they are completely depleted. The capacity loss is little and gradual while they aren't in use. Additionally, they do not need any upkeep or affect the environment. Lithium-ion batteries, on the other hand, lose capacity when they are overcharged, and they degrade in hot environments. Lithium-ion battery materials are also prohibitively costly, which is a serious disadvantage(Linden 2002). A comparison of lithium-ion batteries' benefits and weaknesses may be seen in Table 1.1.

Table 1.1. Lithium-ion batteries: advantages and disadvantages.

Advantages	Disadvantages
High energy density and the ability to be charged	The use of costly materials in its construction
Low intensity	Capacity loss due to overcharging
Able to work at both low and high voltages	Needs a safety circuit
Work in a wide range of temperatures	Thermal breakdown at high temperatures
High accuracy in coulombs	Low degree of security dangers
Low effect on memory and quick charging	
Low loss of power when not being used	
Easy to use and lasts a long time	

1.5.1. Utilization

Thanks to its remarkable features such as high energy and power density, longer life, low self-discharge rate, no memory effect, the lithium-ion battery is one of the most popular and widely used in mobile phones, laptops, tablets, electric vehicles and hybrid electric vehicles today. It is one of the rechargeable batteries. (Liu X. et al., 2018)

Li-ion batteries are now widely used as the main energy source for mobile devices such as smartphones and laptops, as well as hybrid vehicles due to their high power output and high energy characteristics. Lithium-ion batteries, which are planned to be widely used in electric vehicles and expected to be used in electrical energy storage, can store and use energy through oxidation and reduction reactions associated with the anode and cathode. (Hwang D. et al., 2016)

For their use as a medium to large energy source, lithium-ion batteries, such as EVs in modern small electronic devices, and energy storage systems have been extensively studied in recent years. Cathode active material is required for these applications, which include high energy density, high voltage, charge / discharge efficiency, high stability and environmentally friendly materials. (Kang K. et al, 2011), (Lee H. et al, 2013). In addition, they are used to store energy from renewable energy sources, such as wind turbines and solar panels. Figure 1.2 sums up the different ways li-ion batteries can be applied.



Figure 1.2. Li-ion battery applications

(Source: Pastic Energy, 2022.)

1.5.2. Principal Lithium-Ion Battery Components

Li-ion batteries are made up of the cathode, anode, electrolyte and separator. In this configuration, the anode (negative), the cathode (positive) electrode, the separator is the sheet between both the electrodes, and the electrolyte is the fluid that carries ions between the electrodes. Figure 1.3 provides an illustration of the primary constituents of lithium-ion batteries.

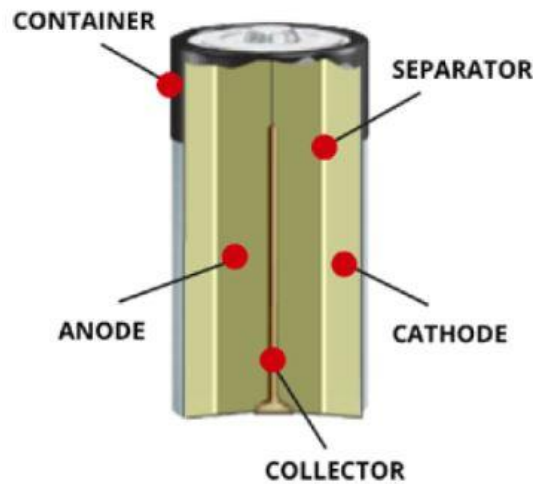


Figure 1.3. Li-ion battery components.
(Source: Energizer, 2022.)

1.5.2.1. Anodes

There are studies that look at the anode, cathode, and electrolyte separately, and there are also studies that look at the whole system. There have been a lot of patents on this subject, and research done in the US patent office showed that there are almost 10,000 patents on lithium ion alone.

Lithium metal was initially employed in the 1970s, according to the historical evolution of anode materials. Although lithium metal has a high energy density, it is an alkali metal that has a rapid and combustible reaction with water. As a consequence of the rapid heating caused by the exothermic reaction, the battery has lost its dependability.

Eventually, carbon began to be used as an anode material. However, carbon's poor capacity has compelled scientists to explore for alternate anode materials. Among the parameters considered during the research are the capacity, the availability of the

material to be chosen on earth, the reliability of the battery during its lifetime, the impact of the battery on the environment (whether it has toxic effects, its suitability for the recycling process), the cell production cost, and its manufacturability via various techniques. (Inoue and Zou 2005, Shukla 2008).

In the years that followed, it was discovered that lithium metal formed alloys with many metals (Mg, Ca, Al, Si, Ge, Sn, Pb, As, Sb, Bi, Pt, Ag, Au, Zn, Cd, etc.), and lithium alloy anodes were manufactured. Examining the capacities of materials that may be used as anodes reveals that carbon-based anodes have a capacity of 372 mAhg^{-1} ; metals such as Sn, Si, Pb, As, Sb, and Al have greater capacitance values. (Hummel 2011).

There are several carbon-based compounds that may be found in modern technology. These include graphite and graphite oxide, as well as graphene and its composites. Because of its low price, one of the most widely utilized carbon based anode substance is graphite in industry. Graphite has a theoretical capacity of 372 mAhg^{-1} and a single-track capacity of 330 mAhg^{-1} . Furthermore, generally li-ions can be readily absorbed into graphite and separated, these ions generate an interface which is known as solid electrolyte interface (ISE) with electrolyte solutions (Endo, et al. 2000). Li-ion batteries with graphite anodes, on the other hand, suffer from permanent capacity reductions (Yao and Cojocaru 2013). In contrast to graphite, strong carbon has a greater oneway capacity and less expensive. The commercial usage of graphite in lithium-ion batteries has been replaced by hard carbon (Li 2012).

While the theoretical capacities of li-ion materials like silicon (4200 mAhg^{-1}) - (9944 mAhg^{-1}) are large, the fact that lithium ions may readily attach and detach from their structures makes these materials unsuitable for widespread use. In an effort to improve these materials' resistance to volume change, the scientific community is working hard (Zhang 2011). Anodes used in li-ion batteries must meet certain specifications.

1. Alteration in volume with the entry and departure of li-ions from the structure
2. Li-ions may go in and depart the structure without difficulty.
3. Superior beginning capability
4. High capacity for irreversible processes and
5. High-capacity protection
6. Simple manufacture
7. It is inexpensive.
8. It reacts little with electrolytes.

1.5.2.2. Cathodes

The cathode, in li-ion batteries, serves both positive (+) electrode and the negative (-) electrode during charging/recharging. There are commercial applications for spinel, olivine, and layered cathode materials. LiMO₂ or LiMP are the most common compounds. Today, there are five primary cathode materials. As a group, they include LiCoO₂, LiMn₂O₄, LiFePO₄, LiNiCoAlO₂, and LiNiCoMnO₂ (NCA) (NMC). There are comparisons between these different cathode materials in Table 1.2 in terms of their crystal structures and electrochemical characteristics.

Table 1.2. Electrochemical performance of five common cathode materials used in commercial products (Yuan 2013).

Material	Li/Li+ Rated Voltage (V)	Capacity (mAhg ⁻¹)	Energy Density (Whkg ⁻¹)
LiCoO ₂	3,6	120-150	432-540
LiMn ₂ O ₄ (LMO)	3,8	100-135	380-520
LiFePO ₄ (LFP)	3,45	150-170	510-590
LiNi _{0,8} Co _{0,15} Al _{0,05} O ₂ (NCA)	3,8	180-200	680-760
LiNi _{0,5} Co _{0,2} Mn _{0,3} O ₂	3,8	160-190	610-730
Li _{1,2} Ni _{0,2} Co _{0,08} Mn _{0,52} O ₂	3,7	220-260	820-970

Because cobalt is costly and hazardous, LithiumCobaltOxide was used as the first cathode material to be commercially available in 1991. Also it was utilized by user devices. In spite of the fact that LFP cathodes were safer, they were unable to match the LithiumCobaltOxide with regard specific energy density. Due to their high nominal voltage and extended cycle life, LMO and LFP stand out from the crowd (Yuan 2013). Because of its long cycle life and high energy density, LFP is utilized in electric cars

while LCO is used in consumer electronics. New cathode materials were also created by modifying existing ones with varied additions.

$\text{LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4$ and other high-performing materials were used. Lithium-Nickel-Cobalt-Aluminium-Oxide with a rated voltage and high capacitance as well as Lithium-Nickel-Cobalt-Manganese-Oxide materials, were identified also studied for application in electric cars. Using these two cathode materials in electric cars is becoming more popular nowadays.

The cathodes used in li-ion batteries should have these properties;

1. High beginning capability
- 2 It is secure.
3. Superior irreversibility
4. Inexpensive.
5. Capacity for high conservation.
6. It is environmentally friendly.
7. It is simple to create.
8. Great energy density.
9. Capability to operate in a wide voltage range.
10. The ease with which a high number of lithium ions may bind to the crystal structure.

1.5.2.2.1. Layered Cathode Components

LiCoO_2 has still been used well in the battery systems since commercially the first available. It is made up of layers of -NaFeO_2 . Figure 1.4 shows how LiCoO_2 is made up of crystals. LiNiO_2 has a similar structure to LiCoO_2 , but it has low-cost and can hold more than LiCoO_2 . LiNiO_2 do not play an important role in business, because it has low thermal stability, and its structure decomposes when it is exposed to temperature (Demiray 2007). Figure 1.5 shows, in general, how the layered LiMO_2 s are made.

Two-layered cathode materials are $\text{LiNi}_{0.8}\text{Co}_{0.15}\text{Al}_{0.05}\text{O}_2$ (NCA) and LiNiCoMnO_2 (NMC), these two structure are being more studied, because they have a high capacity and a high nominal voltage, so these two cathode structures are more used in electric vehicles. They attract the attention from cobalt-based batteries with high energy density because they have long-life. But NCA batteries have problems like losing

power at high temperatures between 50–70°C, because the SEI layer grows, and tiny cracks form at the particle boundaries. (Bloom, et al. 2003).

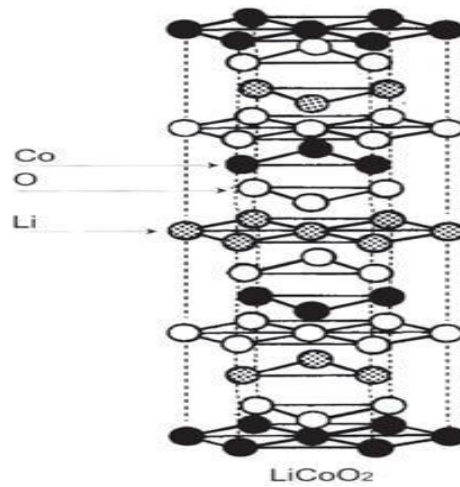


Figure 1.4. Crystal structure of Lithium-Cobalt-Oxide. (Linden 2002).

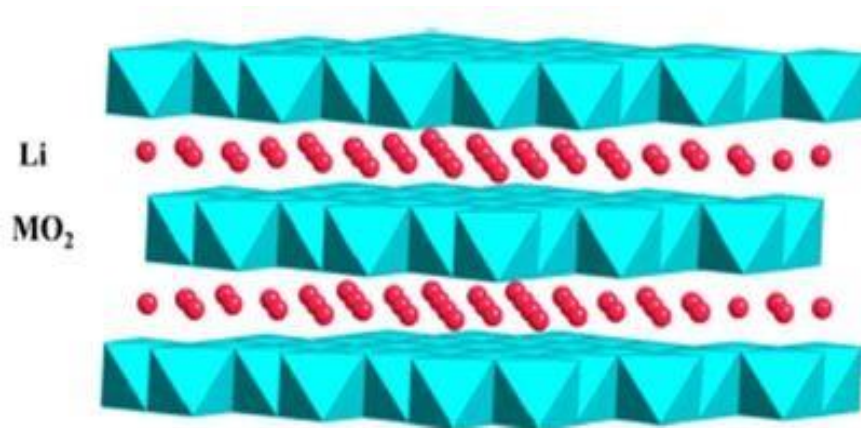


Figure 1.5. Illustration of the structure of cathode materials based on LiMO₂. (Xu, et al. 2012).

Despite the fact that NMC cathodes have a high density and a long cycle-life, capacity loss and poor cycle life may occur because of structural degradation for several causes. Studies are being conducted on Li-NMC cathode materials in an effort to mitigate the structural deteriorations that have been observed. Because they are composed of two phases structural that are Li₂MnO₃ and LiMO₂ (where M = Ni, Co, and Mn), respectively—Li-NMC cathodes have a good capacity as a result of their composition. As a result of its large capacity and high nominal voltage, it has a high energy density, which makes it an excellent candidate for usage in electric cars. But in order to do this,

the issue of capacity loss has to be substantially reduced. You may also mention Li_2MnO_3 , LiVO_2 , LiCrO_2 , and LiTiS_2 as other layered cathode materials that have been utilized and investigated (Nitta, et al. 2015).

1.5.2.2.2. Spinel-Structured Cathode Materials

LiMn_2O_4 is a significant spinel cathode materials that could used (Figure 2.6). Although LiMn_2O_4 cathode has a bed specific capacity and its energy density is comparable to that of other cathodes owing to its high rated voltage.

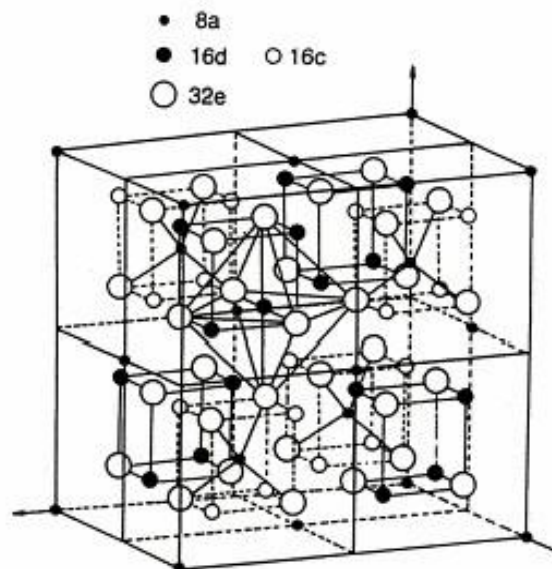


Figure 1.6. The spinel structure's appearance. (Source: Linden, 2002).

$\text{LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4$ has a spinel structure, and this structure has a higher specific capacity ,approximety 147 mAh/g, than LiMn_2O_4 (Hu, et al. 2013). Also, the fact that these two materials are cheap and good for the environment is a big plus. But because of things like reactions that separate Mn^{+3} from the structure, Jahn-Teller distortions that happen when the Mn^{+3} concentration goes up, and capacity loss because of these things, the studies are being worked on to improve this cathode (Kang, et al. 2001). The oxygen atoms that is in the structure of LiMn_2O_4 are similar to a layered structure. However, unlike the layered structure, the Mn atoms is in the octahedral position. (Xu, et al. 2012, Hu, et al. 2013, Bloom, et al. 2003, Demiray 2007, Liu, et al. 1996, Kang, et al. 2001).

1.5.2.2.3. Cathode Materials With The Structure of Olivine

Cathode material having an olivine structure is LiFePO_4 , the most widely used commercial cathode material in the world. Nominal voltage for the cathode of this device is 3.45 V, and its theoretical capacity is 170 mAhg^{-1} . But even though it has a larger capacity, this cathode material has a lower nominal voltage than most other cathode materials (Chung, et al. 2002). Due to the fact that LiFePO_4 has great a steady temperature and a long cycle life with no capacitance deterioration without deterioration, it is not extensively employed. (Armand 2004). LiMnPO_4 and LiCoPO_4 cathode materials in the olivine structure are also being studied (Delacourt, et al. 2005), (Orendorff and Doughty 2012). LiFePO_4 has an octahedral FeO_6 structure and tetrahedral PO_4 s in its crystal structure (Okada, et al. 2001). Figure 2.7 depicts the olivine crystal structure of LiFePO_4 in a solid solution. In the b and c planes, these octahedra and tetrahedra touch each other, but the PO_4 -tetrahedra do not (Zhang 2011). LiFePO_4 is stable at high temperatures because of the powerful Phosphorus Oxygen covalent bonds in its crystal structure, which prevent the $\text{Fe}^{3+}/\text{Fe}^{2+}$ redox pair from breaking down.

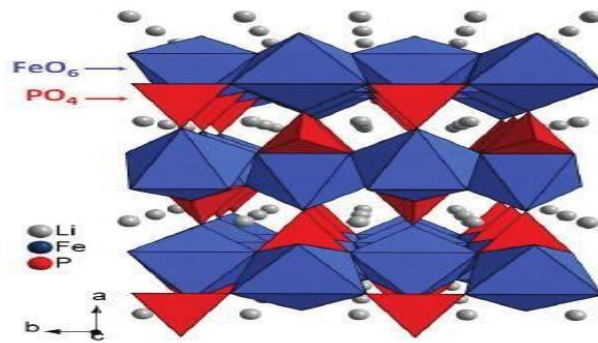


Figure 1.7. Structure of LiFePO_4 s. (Source: Daniel, et al. 2014)

1.5.2.3. Electrolyte

The electrolyte's job is to move electricity between the electrodes. Most Li-ion battery electrolytes are made of salt dissolved in liquid electrolyte solvents. The electrolyte, which is in between the positive and negative electrodes, is a key part of how

the battery works. Comparative tests between the electrode and the electrolyte should be done to look at the properties of the new electrolyte solutions.(Linden 2002).

Li-ion batteries have four types of electrolytes: liquid electrolytes, gel electrolytes, polymer electrolytes, and ceramic electrolytes. Most of the time, liquid electrolytes are solutions of lithium salt in organic solvents made of carbonates. The polymer electrolyte is a liquid with no solvents. The ionic conducting phase is made by dissolving the salt in the high molecular weight polymer. A gel electrolyte is an ionically conductive material made from a salt, a solvent, and a high molecular weight polymer that is mixed or dissolved together.

Most Li-ion batteries need electrolytes that have the following basic qualities.(Vincent, 2003)

- ✓ To dissolve lithium salt at the right concentration, it must have a high dielectric constant.
- ✓ It should be fluid in the working temperature range so that it is easier to move ions.
- ✓ Solvents should have a high point at which they boil and a low point at which they melt.
- ✓ It should be stable when it comes to electricity.
- ✓ It must not react with any part of the battery.
- ✓ It must be safe, not harmful, and cheap.

1.5.2.4. Seperator

Most commercially available Li-ion batteries use microporous polyolefin separators like polyethylene (PE) and polypropylene (PP), which have a standard pore size.

The separator is an important part of batteries with liquid electrolyte. It is placed between the positive and negative electrodes to keep them from touching and to stop electrons from flowing through the battery by letting free ions pass through. Most of the time, it is made up of a microporous layer with a polymeric membrane or non-woven fabric inside. It must be chemically and electrochemically stable against the materials in the electrolyte and electrodes, and it must be strong enough to withstand high pressure

(tension) when the battery is being installed. For high ionic conductivity, the separator must have enough holes in its structure to soak up the liquid electrolyte (IHM, 2002).

The separator makes the electrical resistance go up and takes up a small amount of space in the battery, which hurts the performance of the battery. Because of this, choosing the right separator is important for the energy density, power density, cycle life, and safety of the battery. For high energy and power densities, the separator needs to be very thin and have a lot of holes while still being strong. For the safety of the battery, the separator should be able to stop the battery from working if it gets too hot. This could happen if there is a rare short circuit. The stop function comes from the fact that the separator has more than one layer. In this case, at least one layer melts below the thermal runaway temperature to close the pores, and the other layer gives it strength to keep the electrodes from touching (Zhang, 2007).

1.5.3. Principle Of Operation Of Lithium-Ion Batteries

The parts of a cylindrical lithium-ion battery are shown in Figure 1.8. Lithium-ion batteries have a metal shell, just like most other batteries. This metal box holds a long spiral of three thin plates that are packed together tightly. This is the anode, the cathode, and the separator. This organic solvent, which acts as an electrolyte, is poured over these three thin plates.

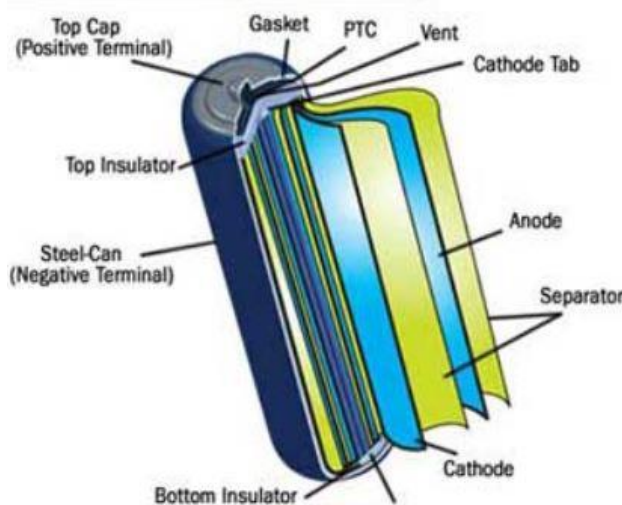


Figure 1.8. General structure of the lithium-ion battery (Source: Physics Central, 2012.)

As with other electrochemical batteries, the way rechargeable lithium-ion batteries work is based on the idea that chemical energy can be turned into electrical energy.

Like other batteries, lithium-ion batteries have three main regions where electrochemical reactions happen. These are:

- I. the positive electrode,
- II. the negative electrode, and
- III. the electrolyte.

During the charging and discharging process, Li^+ ions move between the anode and the cathode. This is what makes the battery work. Lithium-ion batteries are called "swing chair batteries" because of this. Lithium cobalt oxide and graphite are used as electrodes in lithium-ion batteries. The electrolytes are made by dissolving lithium salts, like LiPF_6 , in an organic solvent. The reaction mechanism in commercial lithium-ion batteries. (Linden 2002).

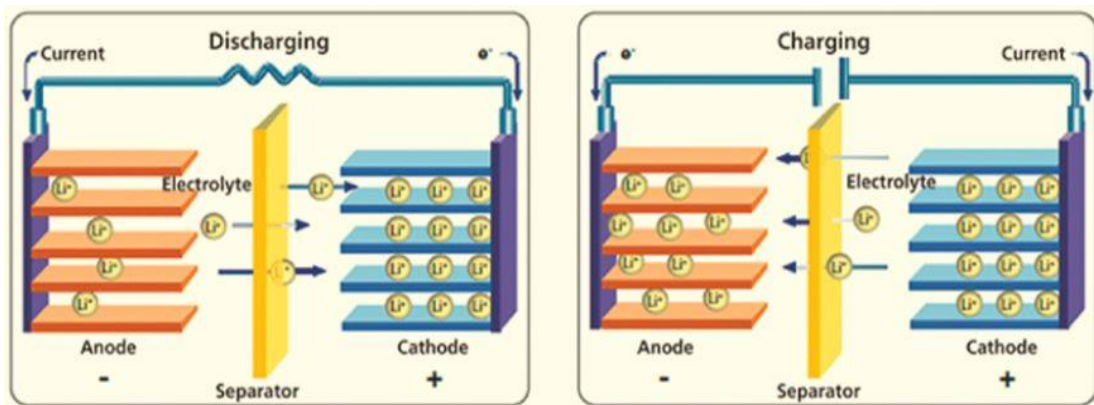
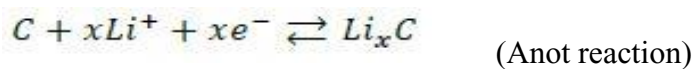
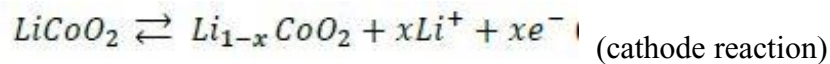


Figure 1.9. Li-ion battery charging and discharging processes. (Source: Buchmann, 2012)

1.6. Materials For NMC and Li-Rich NMC Cathodes

In recent years, NMC cathode materials have been utilized extensively in lithium-ion batteries. It possesses a high specific capacity (170 mAhg⁻¹), a high nominal voltage (3.8 V), and therefore a high energy density and a long cycle life. Due to these characteristics, its application in electric cars is growing. In the investigations, several NMC cathode compositions are being investigated. NMC cathode materials feature α -NaFeO₂ type layered structure, and the most often investigated NMC compositions are LiNi₁ / 3Co₁ / 3Mn₁ / 3O₂ (NMC111), LiNi_{0,5}Co_{0,2}Mn_{0,3}O₂ (NMC523), and LiNi_{0,8}Co_{0,1}Mn_{0,1}O₂ (NMC524) (NMC 811).

Table 1.3 presents a comparison of the findings gained from the research conducted on the most common types of NMC cathode materials. Additionally, the compositions of NMC622, NMC712, and NMC721 are being investigated (Choi and Lee 2016, Pan, et al. 2016)

Table 1.3. demonstrates the initial discharge capacities and capacity retention ratios of the most experimentally investigated NMC cathode materials.

NMC Composition	First Discharge capacity (ms/g)	Capacity Conservation (%)
NMC111 (Oljaca, et al. 2014)	160 (0,2C)	61 (After 80 cycles with 1C)
NMC523 (Kong, et al. 2014)	201,2 (0,1C)	87.4 (After 60 cycles with 2C)
NMC811 (Lu, et al. 2013)	195,7 (0,1C)	85.2 (at 0.1C after 50 cycles)

Divalent nickel, trivalent cobalt, and tetravalent manganese ions are found in NMC cathode materials. Ni²⁺ and Co³⁺ and Co⁴⁺ redox reactions only occur between 2.5

and 4.2 volts, whereas Mn stays tetravalent. Because of this, Mn is included into the design to help keep it stable (Johnson, et al. 2004). Charging the NMC layer over 4.2 V keeps it steady. Due to the fact that the structure is disrupted during charging, Ni and a little amount of Co are able to enter the tetrahedral lithium sites that lithium discharge, causing the structure to break down (Mizushima, et al. 1980). Oxidation of the electrolyte or the release of oxygen from the cathode may also be caused by an increase in oxygen activity on the electrode's surface. In the wake of these occurrences, the cycle life is shortened and capacity is reduced (Wang, et al. 2011).

Lithium-rich NMC is a layered cathode material with Li_2MnO_3 and LiMO_2 (M = Ni, Co, Mn) phases. The Li_2MnO_3 phase is what makes it different from NMC cathode materials. The formulas $x\text{Li}_2\text{MnO}_3(1-x)\text{LiMO}_2$ (M = Ni, Co, Mn) or $\text{Li}_{1+x}(\text{NiMnCo})_{1-x}\text{O}_2$ can be used to describe lithium-rich NMC cathode materials. Studies are being done to make lithium-rich NMC cathode material widely used in electric vehicles because it works at high voltages, has a high capacity, a high energy density, a low cost, and is safe. The structure of lithium-rich NMC is like that of $\alpha\text{-NaFeO}_2$. NaFeO_2 is made up of Li in the Na positions and Ni, Co, and Mn in the Fe positions. If you think of this structure as a modified NaCl structure, you can see that tightly packed planes of oxygen atoms separate the lithium layers from the layers that are rich in transition metals.

Transition metals and lithium occupy octahedral pores . (Brinkhaus 2015).

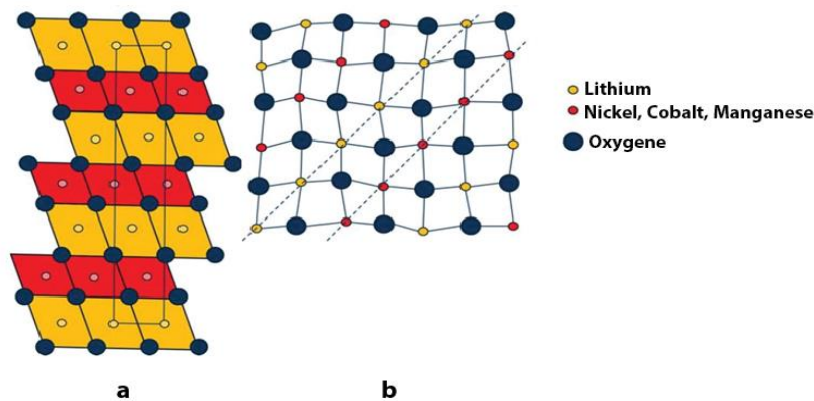


Figure 1.10. a) 2-D octahedral arrangement in NMC b) the position and arrangement of all the atoms in it. (Source: Brinkhaus, 2015).

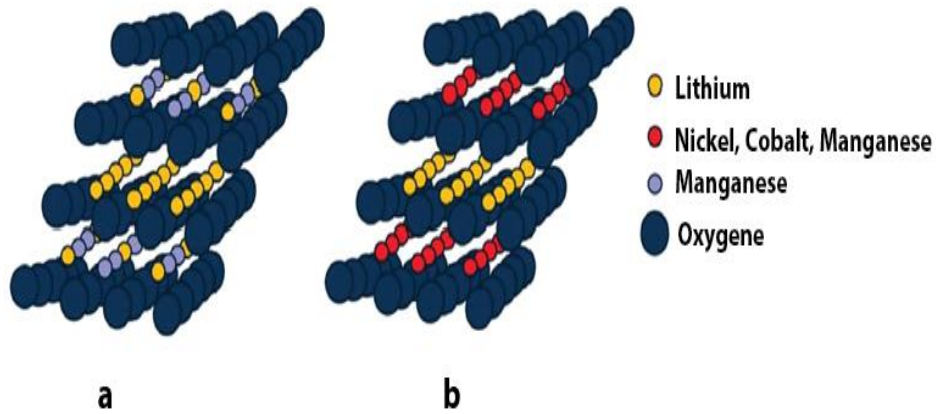


Figure 1.11. a) Li-rich NMC 2-D octahedral array in its structure. b) Show how the atoms are put together. (Source: Brinkhaus, 2015).

Manganese endures over the whole surface from the earth to atoms in the formation of Li_2MnO_3 as shown in figure 1.12, deny the reality that the structure is formed of atoms on the earth in a layer with rigid planetary facades in stage. . Coulomb, a block in terms of position between superlattice and manganese in a hexagonal form. This array's symmetry is $C2(m)^{-1}$. According to the studies, the XRD study has uncovered 2 degrees worth of health data. Therefore, this is the case, the formula for $x\text{Li}_2\text{MnO}_3(1-x)\text{LiMO}_2$ may be written as "Li₂MnO₃ possesses monoclinic C2/m symmetry, whereas LiMO₂ possesses rhombohedral symmetry."

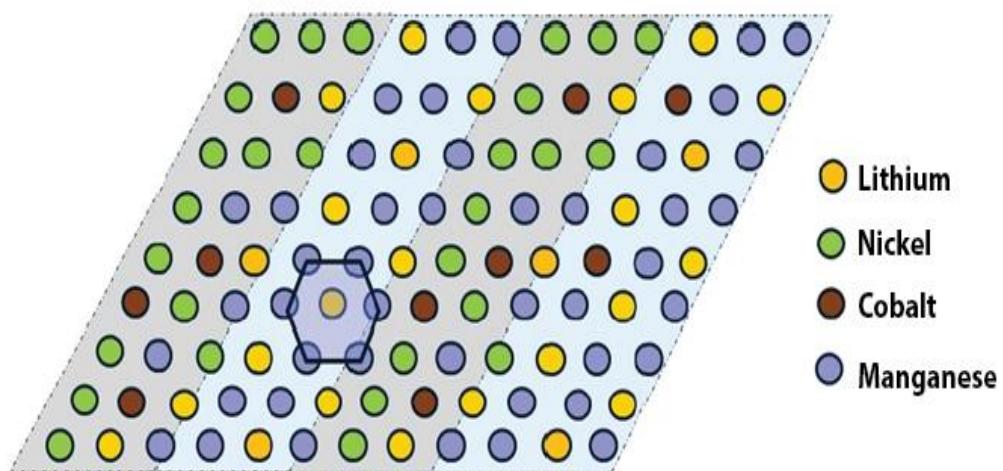


Figure 1.12. Balpetic (flower) block display with uniform dispersion of 2 stages of li-rich NMCs on the transition metal plane. (Source: Brinkhaus, 2015).

During the first charge of li-ion batteries with cathode (NMC), ions of li with a voltage of up to 4.4 V are made from LiMO₂. Between 3.5-3.9 V, divalent nickel (Ni²⁺) changes into tetravalent nickel (Ni⁴⁺), while trivalent cobalt (Ni³⁺) changes into tetravalent cobalt (Ni⁴⁺) at 4.4 V. Manganese remains tetravalent (Mn⁴⁺). When whole of Ni and Co have been oxidized to 4.4 V, the theoretical capacitance is 130 mAhg⁻¹ (Yu, et al. 2012). Li₂MnO₃ gives off Li₂O (as Li⁺ and O₂) when the voltage goes above 4.3–4.4 V. As much as five volts may this. As a result of this, going to high voltages is a way to get high capacities (Ates et al. 2013). This is especially true for the first charge. The first cycle charge is also a very important part of understanding how Li₂MnO₃ and LiMO₂ keep their shape. This process was described by Ateş et al. as the release of oxygen during the initial charge and permanent capacity decreases after the initial cycle owing to the interaction between the electrolyte and the active ingredient.

1.6.1. Techniques for Preperation of Li-Rich NMC Cathode Materials

There are many different processes that may be used to generate lithium-rich NMC cathode materials. The production method has an impact on particle size, purity and dispersion of the material that is produced. The Pechini approach, coprecipitation technique, solid state method, the, and the sol gel technique are the four procedures that are utilized the most frequently in the creation of lithium-rich NMC.

1.6.1.1. Pechini Technique

In the Pechini technique, the precursors are mixed together, which results in the production of a polymer matrix, which is then pyrolyzed at a high temperature. Creating a polymer matrix is the basis of this strategy. Citric acid and ethylene glycol may polymerize when metal ions are introduced and the temperature is elevated to 100 degrees Celsius. After then, pyrolysis and oxidation occur as a consequence of a rise in the temperature of the pechini technique process. The pyrolysis reaction produces the substance that was intended to be the end product of the process. It is not necessary to heat the product that is generated by using this approach (Kakihana et al. 1999).

1.6.1.2. Synthesis In The Solid State

The simplest technique of powder synthesis is solid state synthesis. It's preferable to grind together the oxides of TMs and the lithium precursor before heating the mixture to high temperatures. Due to the risk of crystallization impurities, this synthesis process is not extensively employed for multilayer materials. NMC produced by the solid state technique was able to attain a 141.3 mAh/g initial discharge capacity in the 2.5–4.5 V range. (Eker 2006). Using this approach, the oxides, that are the building blocks of the substance to be made, are ground and mixed to make sure that the powder particles are evenly distributed. Then, if the grinding was done while it was still wet, it is dried and pressed. The samples that have been pressed are then sintered at the correct temperature and in the correct way. The product that comes out of sintering doesn't need to go through any other steps (Arikan 2010).

1.6.1.3. Technique of Coprecipitation

The coprecipitation procedure involves dissolving the metal salts in clean water before adding them to the substance to be produced. After that, adding a fundamental solution and at the right pH, a precipitate forms. In the supersaturated solution, a reaction product is made that can't be dissolved. This is what causes the precipitate to form. The final product is made by heating the precipitate that has already formed. Here are some of the good things about this method; (Darab 2010).

- 1- Temperature of synthesis that is not very high
- 2- Particles of small size
- 3- The ease of the procedure

1.6.1.4. Sol-gel Technique

Commercially, the sol-gel technique is frequently utilized manufacturing techniques because of its manageability of chemical reactions, the ease and low energy consumption, but because of its long processing time and expensive starting ingredients. The following table 1.4 shows the positive and negative features of the sol gel approach.

When particles smaller than 500 nm in diameter are suspended in water under the effect of Van Der Waals forces, the solution goes by the name "sol." In the sol-gel technique, several methods are used based on the kind of supplies that is needed to be generated.

Table 1.4. The positive and negative features of the sol gel process

Positive	Negative
A simple method.	The process is time-consuming.
Possible chemical reactions can be managed.	expensive starting materials
obtaining ultra-pure, submicron-sized powder submicron size	Hydroxyl and carbon continue to exist in the structure.
Does not need much effort	Destruction of resources
Application at a low temperature	Sensitivity to wetness
Appropriate for surface modification and doping	Difficult to construct on a large scale.

Sol is created once the solution achieves the necessary pH and temperature, and then gelation commences. Following gelation, the required material is created by applying product-specific operations for example thermal treatment, drying and extrusion to the gel.

The main stages of the sol-gel process may be summarized in the below;

- ✓ Gelling
- ✓ Condensation
- ✓ Hydrolysis
- ✓ Polymerization

In the sol-gel technique, condensation processes produce monomers, which polymerize into particles, and these particles grow. A gel is generated when the larger particles establish a network structure between one another. "Http://Www.Lehigh.Edu/Imi/Teched/Lecbasic" is the result of drying and heating the created gel to produce the required product. Composition, chelating agent concentration, pH, and temperature are only a few of the variables that play a role in the development of these phases.

1.6.2. Methods for the Production of Li-Rich NMCs

NMC powders that are high in lithium are usually coated on metal surfaces. Suitable coating techniques;

- Laminar coating
- Print coating
- Lamination
- Spin coating
- Roller coating
- Spray coating
- Dipped coating

Dip coating, lamination and spin coating are the three coating techniques that are used the most often for the purpose of coating lithium-rich NMC.

1.6.2.1. Spin coating

The coating materials are put in the middle of the substrate that needs to be coated, and the substrate is turned at the right speed to get the coating on the surface called as spin coating technique.

The basis for distribution is (Figure 1.13). The coating operation is done by letting the dissolver evaporate then the solution has been spread over the surface.

Following the trickling procedure, the substrate's spinning speed is increased incrementally until it achieves the target value. After that, the coating solution is distributed evenly over the surface by the centripetal power and any surplus coated mixture is removed.

The coating material can be spread out on the surface in either a static or a dynamic way. In static distribution, the surface is turned after the liquid has been dripped, but in dynamic diffusion, the liquid is dripped when the surface is turning. The coating's thickness relies on things like how fast it spins, how long it spins, its viscosity and how it is evaporated. As the speed and time of rotation go up, the coating gets thinner (Higgins 1992).

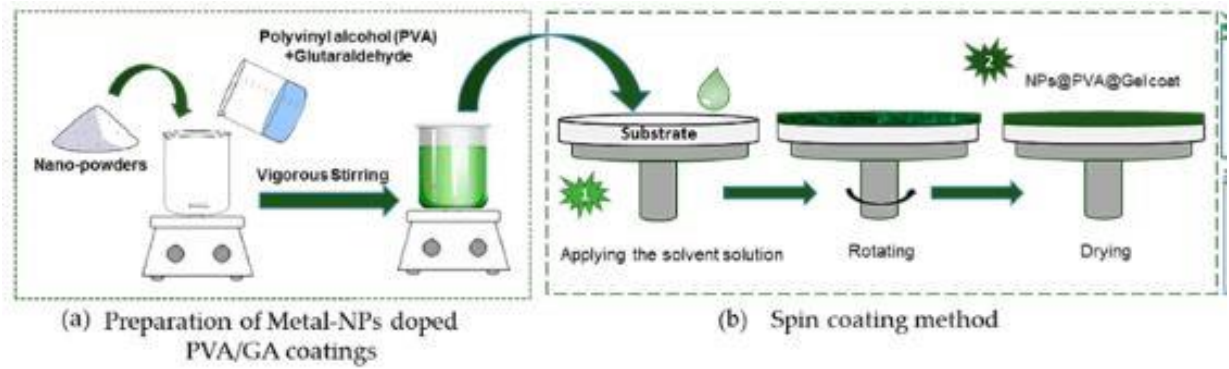


Figure 1.13. Shows how the spin coating method works. (Source: MDPI, 2018)

1.6.2.2. Dipped coating

The dipping coating technique is a very common things to ways to coat a substrate with a thin film (shown in Figure 1.14). The best things about this method are that it is easy to do, that it can be done in a series, and that it can be done again on top of the coating. While an area needs to be coated, a mask is put from the another area, which makes it hard for the coating to stick to the surface. Surface adhesion is one of the most important issues with this technique. It is affected by three force, that is called as the force of gravity, the force of friction, and the surface tension.

The logic behind the dip coating method is simple, like coating the surface besides having to dip it into coating mixture. For a more detailed explanation, the substrate is put into a solution of the material that is to be used and left there for the amount of time that is needed. Then required period for the litter to remain in the solution is up, it's taken out at a consistent rate to avoid the unevenness that would happen if it were taken out at a faster rate. How quickly the substrate is taken out of the solution also affects how thick the coating is. By letting the solvents in the coated substrate evaporate, the right film is made (Brinker, et al. 1991).

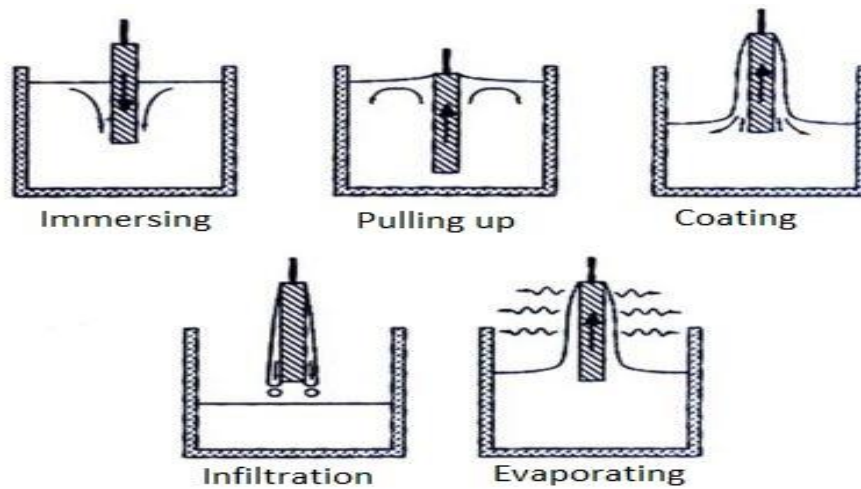


Figure 1.14. The fundamental stages of the dip coating process.

1.6.2.3. Lamination

Figure 1.16 depicts a special knife called a "Doctor blade," which is used in the lamination method. This knife is used to apply the the substance to be used as a surface coating of the substrate, and Figure 1.15 depicts a device that is used to dry the material after it has been applied. The lamination method is based on this principle. The simplicity and low cost of the lamination procedure make it one of the most attractive alternatives to other approaches. A fluctuation on the coated surface is one of its disadvantages, additionally to benefits like consistent mass production and, coating which are both advantages of this method.

To be able to procure a more in depth explanation of the lamination process, I will explain that once the pad material has been sanitise, it's then secured to the lamination machine. First, the coating material's binder, solvent and powder are mixed together, and then the combination is dripped over to part of the substrate that is nearest to the blade. The mixture is driven into the surface of the substrate at the appropriate speed and at the required thickness by the blade, which is known as the Doctor blade. After this procedure, the solvent will have evaporated, and the substrate will then be covered. In the procedure known as lamination, the thickness may be altered by moving the blade up and down, which enables coating to produced at a variety of different thicknesses. Even though the coating ranges that may be manufactured diversify depending on the blade that is worked, they are often anywhere from 10 to 1000

micrometers. When the coating is thinner, even little imperfections on the bottom surface might cause the underlay to distort and become wavy. (Bierwagen, 1992)

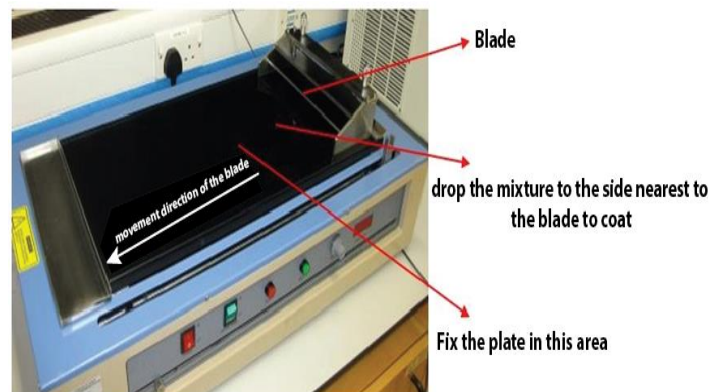


Figure 1.15. Laminating machine



Figure 1.16. Laminator's blade.

(Source: Doctor Blade, 2022)

Lithium-rich NMC films are almost exclusively made via the lamination process when looking at the various coating techniques currently in use. For the most part, this is because it is the simplest and quickest approach, while also avoiding the issues that emerge with dip coating and spin coating. Table 1.5 shows the pros and drawbacks of the most commonly used coating processes.

Table 1. 5. Positives and negatives of the coating method

Method	Advantage	Disadvantage
Lamination	<ul style="list-style-type: none"> ○ The coated mud sticks more easily to the substrate surface. ○ Coating That Is Uniform ○ Thickness may be regulated within specified parameters ○ A simple and affordable strategy. ○ Suitable of mass production. 	<ul style="list-style-type: none"> ○ If the coating thickness is not correctly controlled, the backing foil can tear. so that the coating thickness cannot fall below a certain minimum. The appearance of surface waves.
Rotation Coating	<ul style="list-style-type: none"> ○ The thickness of a material may be controlled. ○ Coating That Is Uniform 	<ul style="list-style-type: none"> ○ Yield is modest ○ Coating is influenced by the litter size.
Dip Coating	<ul style="list-style-type: none"> ○ It is feasible to apply coating layers on top from each other. 	<ul style="list-style-type: none"> ○ Coating-application sludge has difficulty adhering to the substrate's surface.

1.6.3. Li-Rich NMC Cathode Materials Challenges and Possible Solutions

Researchers are putting significant work and energy into studying li-rich nickelmanganesecobalt cathode. Li rich NMC cathodes are receiving the greatest attention from researchers. Lithium-rich NMC cathode materials have unique challenges that must be addressed before any progress can be made toward a solution.

These issues may be enumerated as follows;

- In cycles, there are structural alterations.
- Oxidation of electrolytes and persistent Li loss
- Dissolution/loss of metal ions from the structure (W. Mehrens, et al. 2004)

It is possible to change the structure of the li rich NMC cathode materials by cycling them. The reason for this is that Mn atoms rapidly occupy the tetrahedral spaces in the Li plane as Li ions escape the cathode, whereas Li atoms settle in the tetrahedral pores onto opposite face of the octahedral pores vacated by the manganese atoms during this process (Reed, et al. 2001). Lithium inflow and outflow are prevented by the change in structure from layered to spinel as a result of these atom movements. Li intake and outlet blockage has a detrimental impact on speed and cycle time. Alkali metals like Na, K, and Mg are often employed to address this issue. (He, et al. 2013) According to the research, alkali metals do not move from the octahedral spaces to the tetrahedral spaces, making the transition from a layered to a spinel structure challenging (Li, et al. 2014, Jin, et al. 2014).

Additionally, lithium-rich NMC cathode materials have trouble with O₂ losses from the cathode, which leads to oxidized of the electrolyte. When the voltage during the initial charging increases beyond 4.4 volt and the Li₂MnO₃ phase is turned on, this problem happens. When the activated Li₂MnO₃ gave off Li₂O into the electrolyte, it stopped Li from going back to the cathode and caused an oxide layer to form between the cathode and the electrolyte by reacting with the oxygen that was moving into the electrolyte. As a result of these things, the capacity drops a lot, considerably after the initial charge owing to irreversible lithium loss. There is also oxidation of the electrolyte by oxygen getting into the electrolyte and the creation of an oxide at the cathode

electrolyte interfaces, which causes capacity and speed performance drops in subsequent cycles (Zhou, et al. 2016).

When NMC622 was synthesized by the classical method, the crystalline nickel manganese cobalt ions are in a homogeneous distribution. This situation creates some problems during battery charging and discharging process involving high concentration. The most prominent of these problems is that after the nickel ion is oxidized from nickel +3 to nickel +4 during charge discharge, the high amount of concentration of nickel +4 in the environment reacts irreversibly with the electrolyte in the battery. And it ends the battery life early. Therefore, in a spherical structure, a reaction has to be made that the inner layer must be nickel and the outer layer must be manganese. In this case, all of the nickel ions pass into the electrolyte again.

1.6.3.1. Surface-Modified Metal Oxide

Lithium-rich NMC cathode materials have yet to be commercialized in part because of rapid capacity losses. The cathode materials are treated with metal oxide surface modifications for this purpose. Several MO₂ surface changes performed to NMC cathode materials with li rich have shown promising results, as shown in Table 1.6.

Kong and his colleagues used the ALD method to make a thin ZrO₂ coating. In their study, they tried to find a way to stop the capacity loss that happens when the temperature goes up. At the conclusion of this research, they reported that the surface changes they produced had stopped the loss of metal ions that had been caused by hydrogen fluoride attacks and had expanded the capacity retention at both 25°C and 55°C by sustaining the surface structure. This was done by increasing the capacity retention at both temperatures. (Kong, et al. 2016).

Japanese scientists Jin et al. changed the surface of MnO₂ by using the sol gel technique. They said that its surface change decreased the loss of capacity that can't be fixed after the first charge. They said that this was because the MnO₂ surface modification made it so that Li₂O couldn't separate from the layered structure. They also said that changing the surface of MnO₂ makes Li⁺ move through it faster (Jin, et al. 2016).

Table 1.6. Comparison of li-rich NMC cathodes with different surface modifications.

Name	Coating Made	Applied Technique	Electrochemical Performance
$\text{Li}_{1.2}\text{Mn}_{0.54}\text{Ni}_{0.13}\text{Co}_{0.13}\text{O}_2$ (Zhou, et al. 2016).	La_2O_3	Sol gel technique	Capacity retention rate of 71% after 100 cycles, discharge capacity of 201.4 mA/g
$\text{Li}_{1.2}\text{Mn}_{0.54}\text{Co}_{0.13}\text{Ni}_{0.13}\text{O}_2$ (Li, et al. 2016).	SnO_2	liquid phase technique	Capacity retention rate of 86.8% after 150 cycles, 214 mAs/g discharge capacity
$[\text{Li}_{0.2}\text{Mn}_{0.56}\text{Ni}_{0.16}\text{Co}_{0.08}]\text{O}_2$ (Shi, et al. 2013).	Sm_2O_3	Sol gel technique	91.5% capacity retention rate at the end of 80 cycles, 214 mAh / g discharge capacity
$\text{Li}[\text{Li}_{0.2}\text{Mn}_{0.54}\text{Ni}_{0.13}\text{Co}_{0.13}]\text{O}_2$ (He, et al. 2016).	V_2O_5	Sol gel technique	80.2% capacity retention rate after 50 cycles, 202.2 mAs/g discharge capacity

1.7. Thesis Overview

In this thesis, we investigated the interaction of ammonia concentration with morphology and electrochemical properties in the synthesis of cathode materials for high nickel content li-ion batteries. NMC622 powder was produced by using different concentrations of ammonia solution by co-precipitation method. XRD and SEM analyzes and battery capacity tests were performed on these samples. As a result of the studies, it was observed that low ammonia concentration deteriorated the desired morphology and

increased the stability despite decreasing the specific capacity. It has been observed that high ammonia concentration improves the specific capacity and surface morphology while decreasing the stability. The positive or negative effect of ammonia concentration on NMC cathode materials is clearly explained.

CHAPTER 2

EXPERIMENTAL

2.1. Experimental Introduction

Under the primary heading of "experimental studies," this thesis will provide in-depth knowledge about the cathode materials that were utilized in the experiments, as well as the status under which that studies were conducted, the fabrication of the experiments, then the applied classification of the experiments. Figure 2.1 presents the study's methodology in the form of a flow chart. The primary steps of this flow chart are broken down as follows: manufacturing of $\text{Ni}_{0.6}\text{Co}_{0.2}\text{Mn}_{0.2}\text{O}_2$ powder; fabrication of cathode film; electrochemical testing and characterizations; and final product. The coprecipitation technique is used to produce the precursors of the hydroxide. After that, the characterization and electrochemical testing of the NMC 622 cathodes that were generated by calcining the hydroxide precursors comes later.

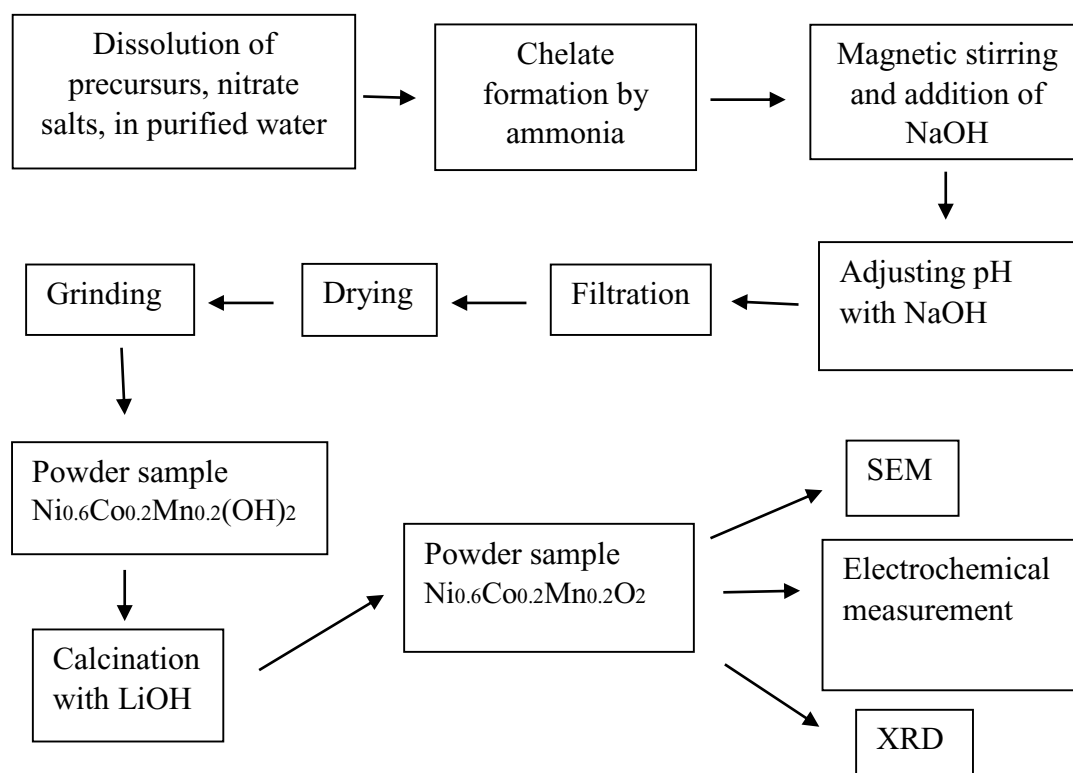


Figure 2.1. Chart of experiment

2.2. Fabrication of Nanoporous Hierarchical Nickel-Hydroxide.

Through a process known as redeposition self-template, hierarchical nickel hydroxide may be manufactured. In the very first stage of the method, 20 milliliters of distilled water are used to dissolve 0.1 mole of nickel nitrate, 0.5 and 1 mole of urea. At room temperature and for a period of half an hour, and solution is allowed to be stirred in three neck balloon. After waiting for half an hour, it was discovered that the solution had become green. After that, the solution is placed inside of a balloon jug that has three separate necks. In this setting, it is stirred for a total of six hours at a temperature of 60 degrees. It has been noticed that the color of the solution transforms into a bluish hue as a direct outcome of the operation of mixing. After this step, a solution of naoh is added to the solution such that the pH is between 10.5 and 11.00, while mixing is maintained at a temperature of 60 degrees. Then about two and a half hours and a half an hour, the color of the solution changes to translucent, and the process of adding the base is complete. After collecting the solution that was produced by centrifugation, it is next cleaned with ethanol and water that has been deionized. After that, it is heated to 70 degrees Celsius and let to dry.

2.3. Cobalt-Manganese Doped Nickel-Hydroxide Synthesis

Another stage of the process is that, 40 ml distilled water is used to dissolve 0.08mol $\text{Ni}(\text{NO}_3)_2$ and 0.50 mol $\text{CH}_4\text{N}_2\text{O}$. At room temperature and for a period of half an hour, solution is allowed to be stirred in three neck balloon. After waiting for half an hour, it was discovered that the solution had become green. After that, the solution is placed inside of a balloon jug that has three separate necks. In this setting, it is stirred for a total of six hours at a temperature of 60 degrees. It has been noticed that the color of the solution transforms into a blue as a direct outcome of the operation of mixing. Following the observation, the colour changes to navy blue, 0.01 mol $\text{Co}(\text{NO}_3)_2$ and 0.01 mol $\text{Mn}(\text{NO}_3)_2$ are put in three necked balloon containing the solution. And then, a solution of naoh is added to the solution such that the pH is between 10.5 and 11.00, while mixing is maintained at a temperature of 60 degrees. When the colour changes into a colorless state, the reaction comes to an end. After obtaining the product, it is then cleaned in pure

water and allowed to dry in oven at a temperature of 70°C. Around 10 hours are needed to finish the whole operation.

2.5. The Manufacturing of Coin Cell

In the course of this era, The coin cell would be constructed, and then it will be performed. To begin, the mixture containing 90% NMC622 cathode, 5% PVP binder and 5% conductive additive was mixed in NMP solvent until homogeneous sludge was obtained. Then the mixture was laid on Al-foil and dried in a vacuum oven at 60 oC for 12 hours. The coating amount is 22 mg NMC/cm². No cleaning process was applied to the Al-foils before coating. The produced cathodes were used as cathodes in the CR-2032 type cell after they were cut to certain sizes. Metallic Li foil was used as the anode in the cell, and 1.0 LiPF₆ dissolved in EC:DMC (1:1) was used as the electrolyte. The NMC622 cathode was tested using commercial carbon black as conductive additive.

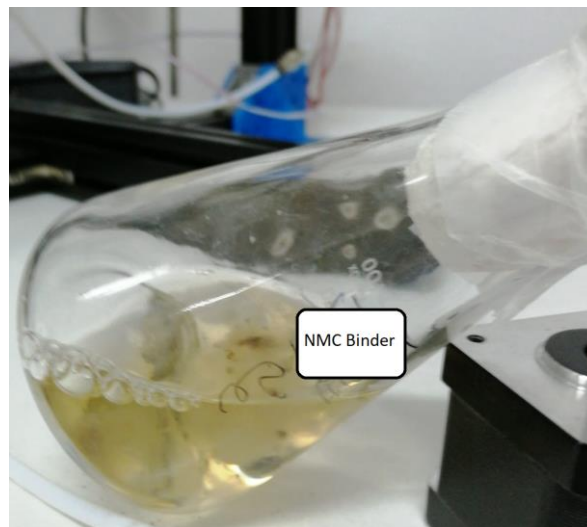


Figure 2.2. Binders for NMC cathode material

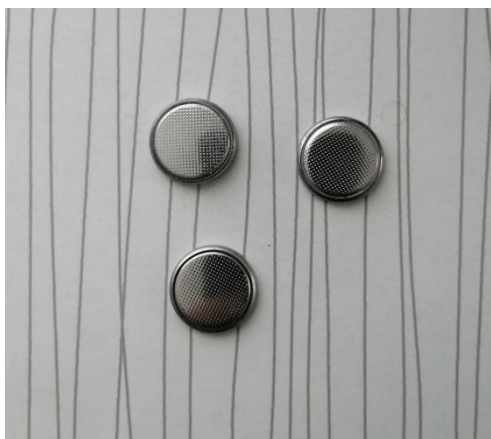


Figure 2.3. Manufactured coin cells



Figure 2.4. Coin cell components

2.6. Techniques of Characterization

In this work, on the side to substance characterization techniques, for example scanning electron microscope (SEM) and X-Ray diffraction (XRD) electrochemical characterization techniques have been used to assess the electrochemical performance of the generated cathode materials. These methods were used to determine how well the cathode materials performed in terms of their electrochemical properties.

2.6.1. Tap Density Analysis

The Tap Dense-TD 101 instrument was utilized in order to carry out measurements of tap density. It was decided to go with the ASTM technique. Tap density was assessed by doing 300 taps per minute, that's 3 thousand hits in total and the results were recorded.

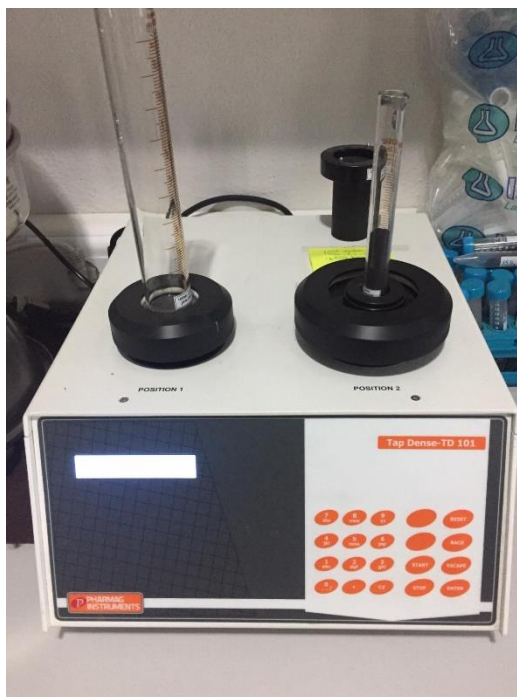


Figure 2.5. Tap Dense-TD 101 device

2.6.2. XRD Analysis

The X-Ray diffraction technique (XRD) is based on the idea that each crystal phase refracts X-rays in a distinct pattern due to their unique atomic arrangement and does not harm the sample. In quantitative phase analysis, it determines the phases of the material by X-Ray Diffraction, as well as temperature, pressure, etc. It is used to determine phase transitions based on physical factors, particle size and orientation, and chemical content. Using this approach, the crystal structure of Li-NMC powder was examined.

The Philips X'Pert Pro is the instrument that is utilized in XRD analysis. The product's crystal structure was analyzed with the assistance of measurements. scanned at an angle of 15-80 degrees.

2.6.3. Sem Analysis

SEM analysis is one way to find out what the surface of samples made with the solid state reaction technique looks like, as well as the size and shape of the particles in these samples. Detailed information about the general morphological properties of the examined Li-NMC powder and the structural and textural properties of the components that make up the internal structure can be provided by image analysis performed in electron microscopy (SEM).

It is able to depict the architecture of materials at the microscopic and nanoscale levels. The SE detector generates a topographic picture that is three dimensions in length, whereas the BSE detector generates an image that is only two dimensions long and is depending on the contrast of the atoms. Otherwise, the elemental composition of the structures may be determined quantitatively as well as qualitatively by using the EDX detector, and the mapping function allows for the monitoring of the distribution of elements across the image. During the SEM measurements, an equipment known as the FEI QUANTA 250 FEG was utilized. The morphology of the surface was looked at. The EDX analysis was used to identify the chemical composition.

2.6.4. Electrochemical Measurements

All battery test analysis were performed with Neware BTS CT-4008T battery tester model. All results were obtained by scanning the batteries in the range of 2.5V-4.3V, using the CC-charge/CC-discharge method, between 100-280 mA.



Figure 2.6. BTS CT-4008T battery tester

CHAPTER 3

RESULT AND DISCUSSION

3.1. The growth process of $\text{Ni}_{0.6}\text{Mn}_{0.2}\text{Co}_{0.2}(\text{OH})_2$

The production of NMC622 may be broken down into two distinct stages: the first is the synthesis of the Template- $\text{Ni}(\text{OH})_2$, and the second is the regrowth by NaOH deposition. The hydrolysis of urea occurred during the first step, which resulted in the production of hierarchical porous Template- $\text{Ni}(\text{OH})_2$. This procedure will continue until the reaction achieves equilibrium, which should take around six hours. Equilibrium will be reached when the yield of Template- $\text{Ni}(\text{OH})_2$ does not rise with increasing the amount of time the reaction is allowed to proceed. At this point in the process, the two most important steps toward obtaining high specific surface area NMC622 were completed: first, Template- $\text{Ni}(\text{OH})_2$ was formed in the reactant solution; second, NH_3 was released as a side product of the hydrolysis of urea using the following equation: $\text{CO}(\text{NH}_2)_2 + \text{H}_2\text{O} \rightarrow \text{CO}_2 + 2\text{NH}_3$, which will form the $[\text{Ni}(\text{NH}_3)_x]^{2+}$ complexes with the residual In the second step, the generated hierarchical porous Template- $\text{Ni}(\text{OH})_2$ served as a template, and NaOH was used as a precipitating agent (the third important component in achieving a large specific surface area for $\text{Ni}_{0.6}\text{Mn}_{0.2}\text{Co}_{0.2}(\text{OH})_2$). By slowly lowering the concentration of the NaOH solution, the equilibrium of the saturated solution was rapidly disturbed, and a new precipitate phase was produced. We synthesized NMC622 by adding Co^{2+} and Mn^{2+} in the second-stage process in order to improve the conductivity of $\text{Ni}(\text{OH})_2$. The new precipitate phase was combined with a large number of nucleation sites presented by hierarchical Template- $\text{Ni}(\text{OH})_2$, which are favorable for the nucleation and the formation of ultrathin $\text{Ni}(\text{OH})_2$ platelets along the surface of hierarchical Template- $\text{Ni}(\text{OH})_2$.

3.2. SEM Images

The specific morphology of NMC622 synthesized with 0.5 M ammonia concentration and NMC622 synthesized with 1 M ammonia concentration were characterized by scanning electron microscopy (SEM). We can see that for NMC synthesis at 1 M ammonia concentration ($<10\ \mu\text{m}$) it exhibits a similar uniform spherical morphology with a diameter of about $10\ \mu\text{m}$. But in the synthesis of NMC622 with 0.5 M ammonia concentration, the particle size homogeneity is rather poor and there are two morphological structures. There are mostly amorphous spherical particles and some small pieces. This situation shows us that low ammonia concentration disrupts the surface morphology and enables us to obtain a more amorphous structure.

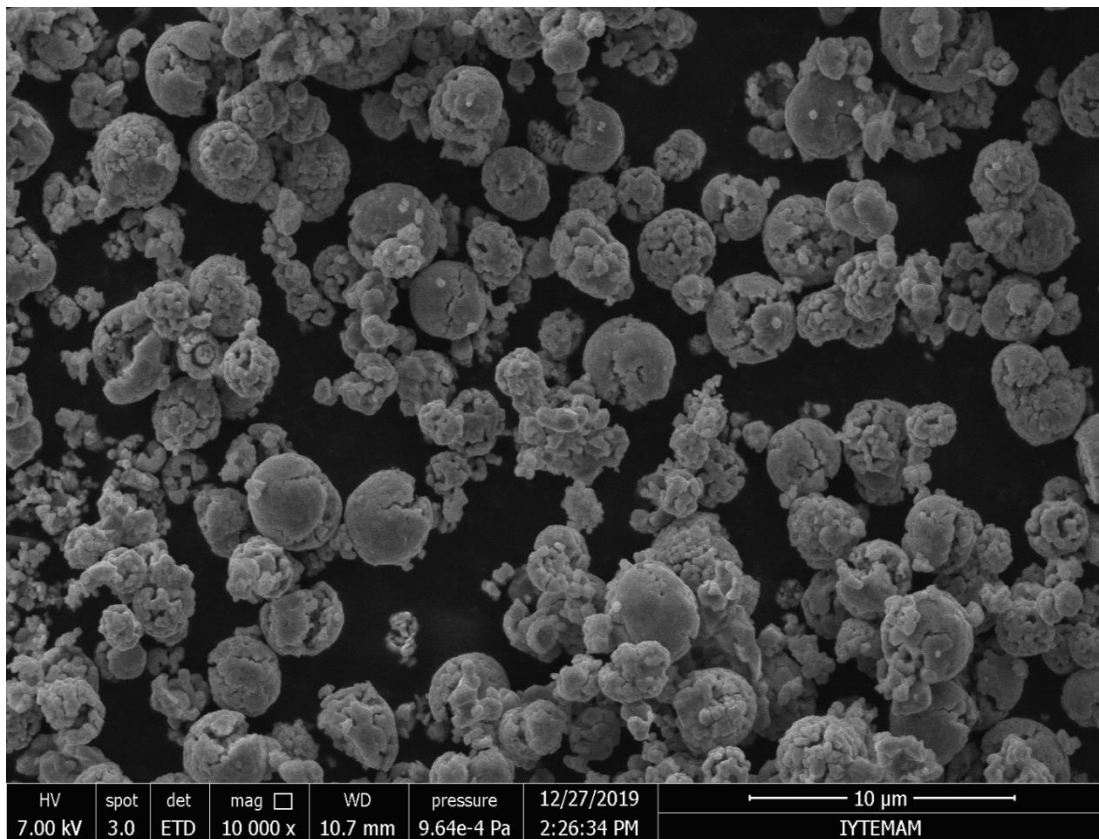


Figure 3.1. SEM image of NMC622 that synthesized with 1 M ammonia concentration

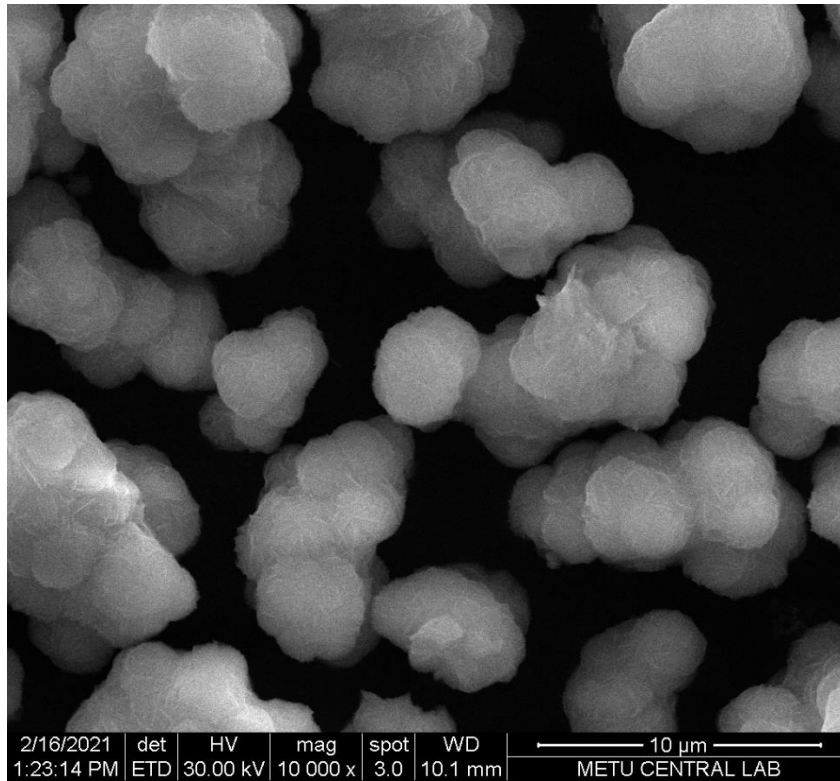


Figure 3.2. SEM image of NMC622 that synthesized with 0.5 M ammonia concentration

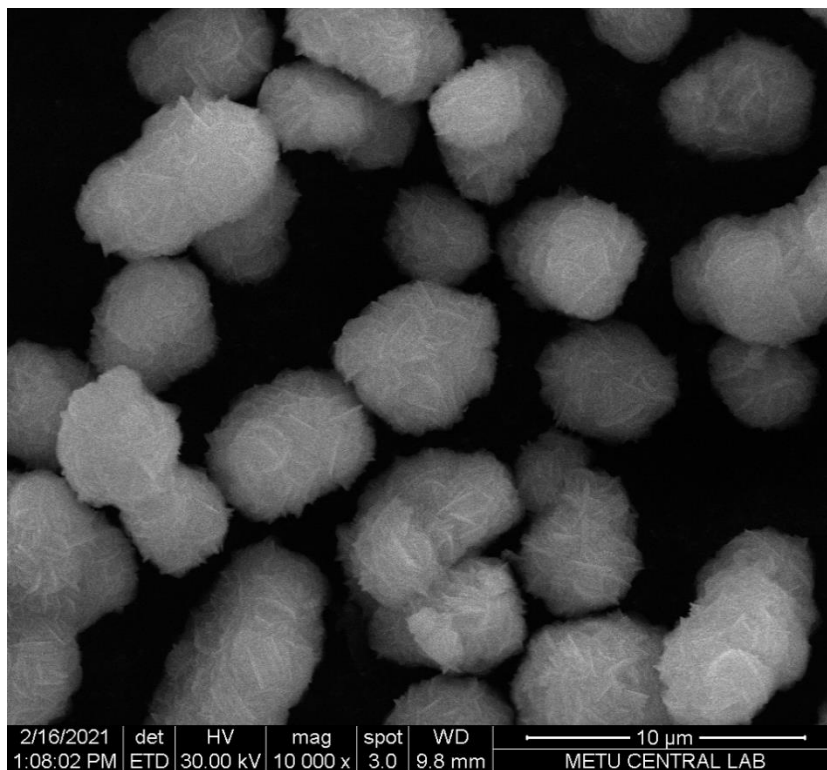


Figure 3.3. Second SEM image of NMC622 that synthesized with 0.5 M ammonia concentration

At a reduction of 10 micrometers, it was noticed that the particle size was regularly distributed and almost completely spherical for NMC622 that produce with 1 M ammonia concentration. Particle sizes are spread homogeneously and evenly. It is accepted that the global structure has been achieved in its totality. The ideal structure for NMC cathodes is a spherical shape. Additionally, it is important that the spherical structures be both big and tiny, since this guarantees that there are no gaps when they are stacked. This provides for a greater density of buildings per unit square. Moreover, the spherical form guarantees that the cathode material is placed appropriately on the collector.

3.3. XRD Patterns

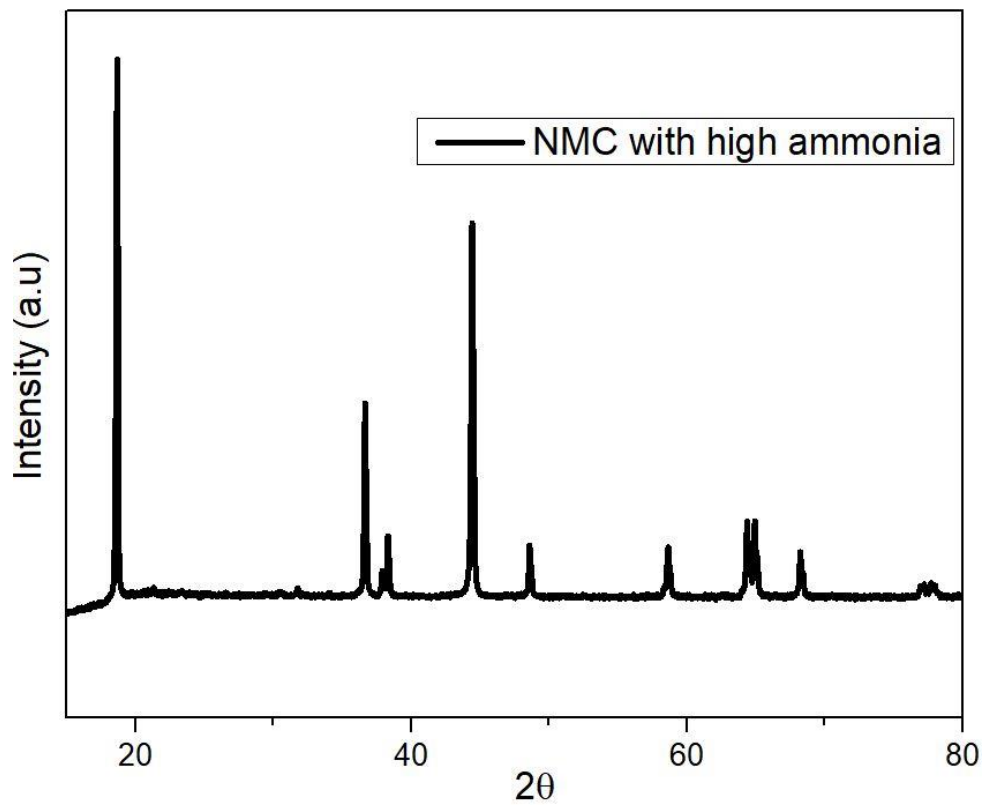


Figure 3.4. XRD result of NMC synthesized with low ammonia

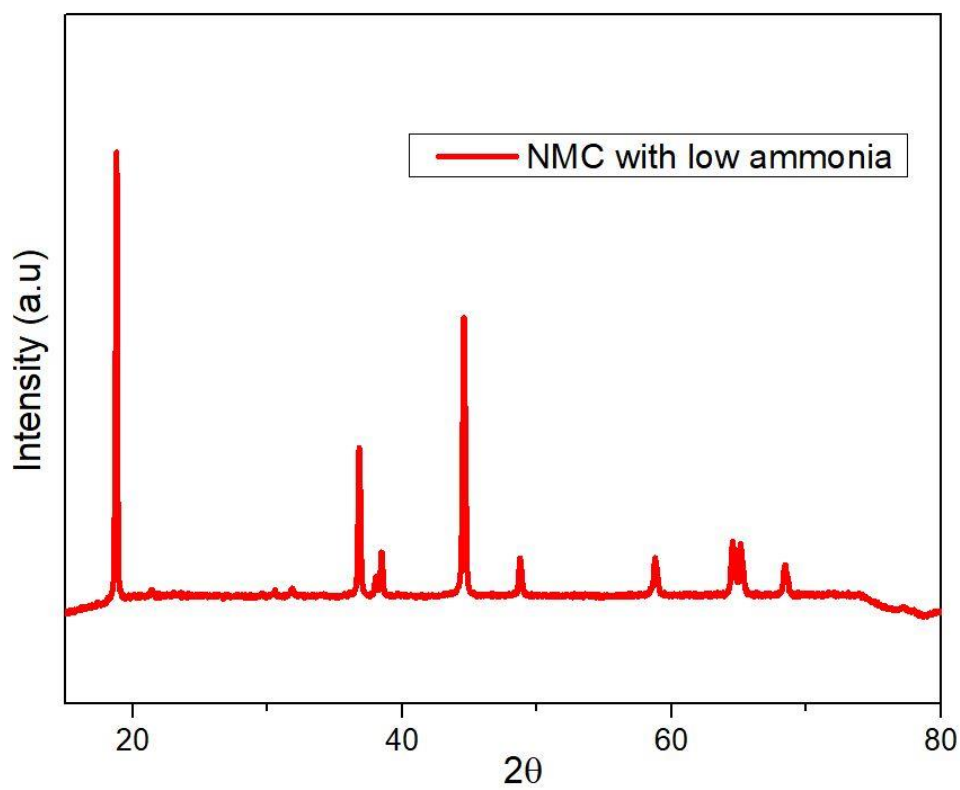


Figure 3.5. XRD result of NMC synthesized with high ammonia

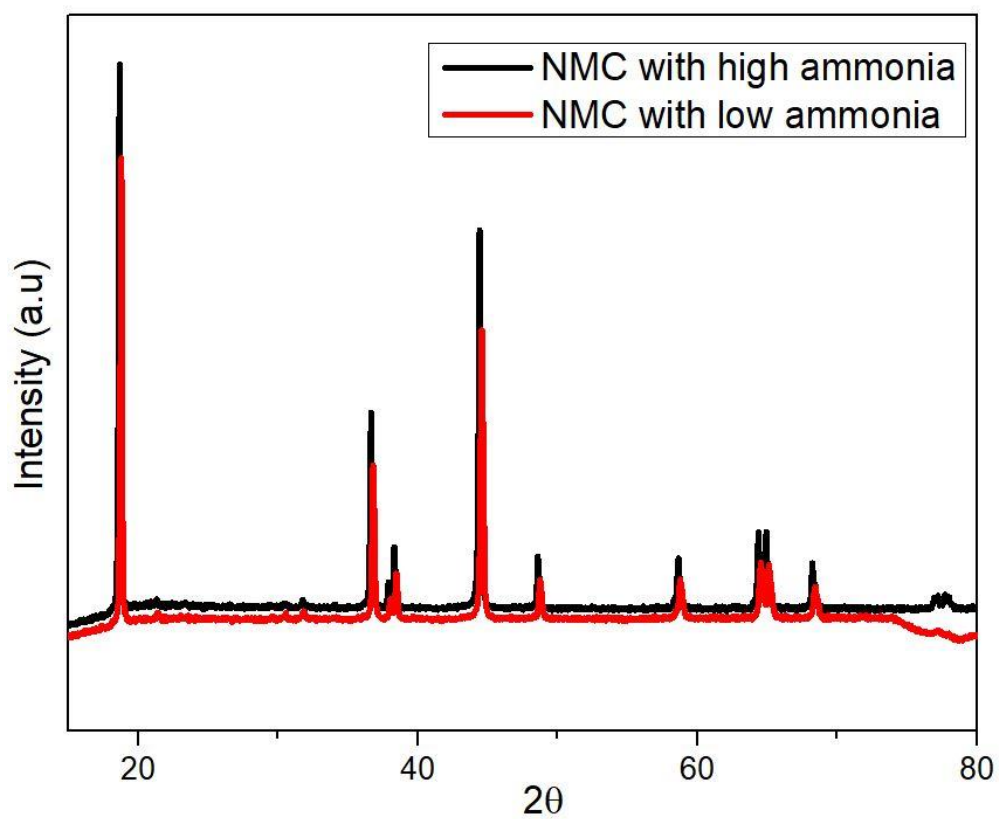


Figure 3.6. XRD results of NMC622

When we look at the XRD results, the high peak (the black one) in the graphics belongs to NMC622 synthesized with high ammonia concentration. The low peak (red one) belongs to NMC622 synthesized with low ammonia concentration. There is no difference in the crystal structures of the materials synthesized by both methods. The only difference seen is in the peak intensity. Therefore, the NMC622 structure synthesized by both methods was successfully synthesized.

3.4. Battery Tester

The charge/discharge curves of the first 10 cycles obtained at 0.5 C current density in the 2.4V-4.8V range for the NMC622 cathode active material produced with 0.5 M and 1 M concentration of ammonia are presented in figure 3.6 and figure 3.7. In the first figure, the specific capacity of NMC622 obtained with 0.5 M concentration of ammonia against the potential is given. In the second figure, the specific capacity of NMC622 obtained with 1 M concentration of ammonia against the potential is given. According to these graphics, it was seen that the material obtained with 1M concentration reached approximately 280 mAh/g. On the other hand, it is seen that the cathode active material obtained with 0.5 M concentration can reach up to 150 mAh/g. It is understood that the decrease in the amount of ammonia is directly proportional to the decrease in the specific capacity.

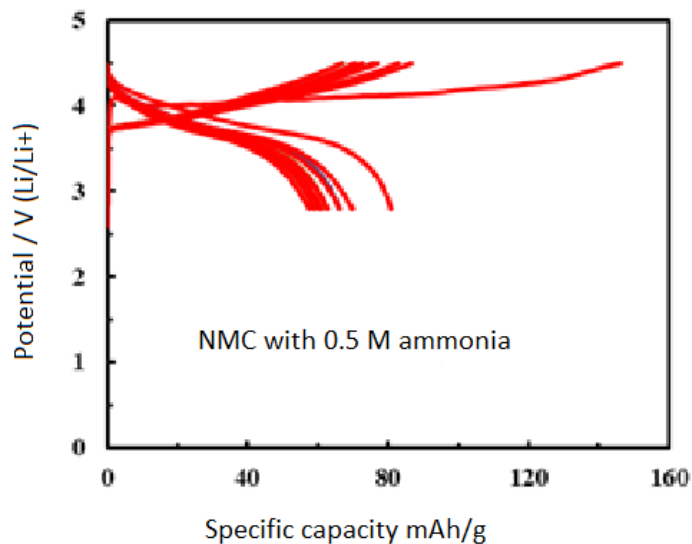


Figure 3.7. Charge/discharge curves for the first 10 cycles for NMC synthesized with low ammonia

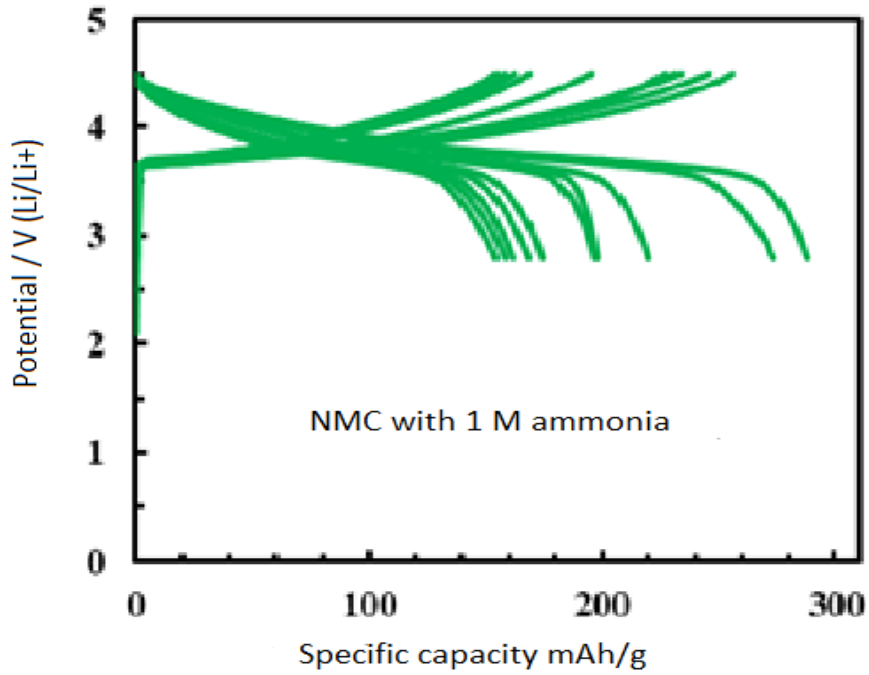


Figure 3.8. Charge/discharge curves for the first 10 cycles for NMC synthesized with high ammonia

The graph of the specific capacity versus the cycle number of NMC 622 cathode active material produced with 0.5 M and 1 M concentration of ammonia is presented in the graphic below. In studies with NMC 622 obtained with 1 M concentration of ammonia, it is observed that the specific capacity decreases as the number of cycles increases. On the other hand, in the study conducted with NMC 622 obtained with 0.5 M concentration of ammonia, it is seen that the capacity does not decrease much and remains stable as the number of cycles increases. The conclusion we will draw from here is that as the ammonia concentration that we will use during the synthesis of NMC 622 decreases, the stability remains almost constant.

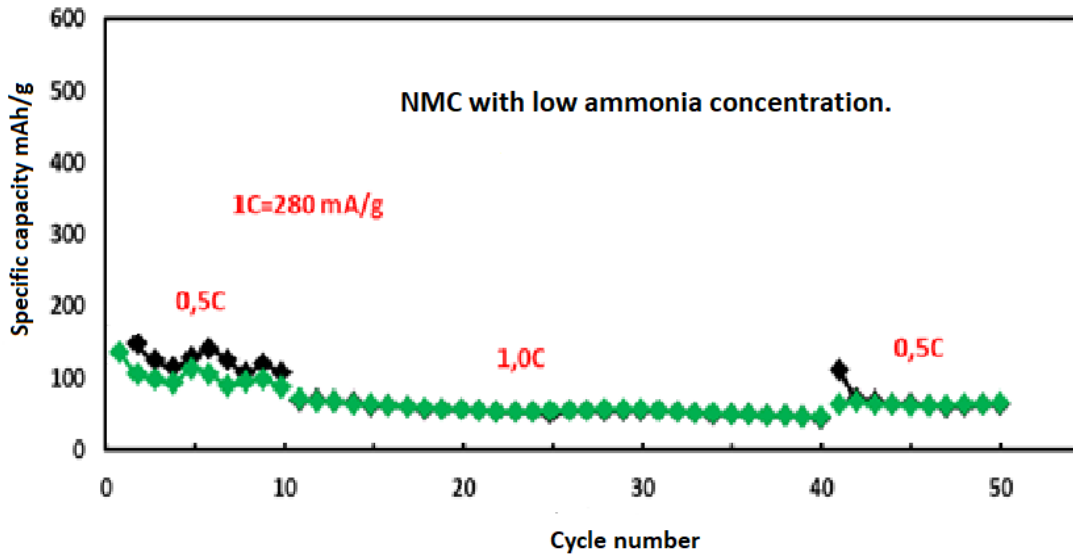


Figure 3.9. Graph of capacity versus number of cycles for NMC synthesized with low ammonia

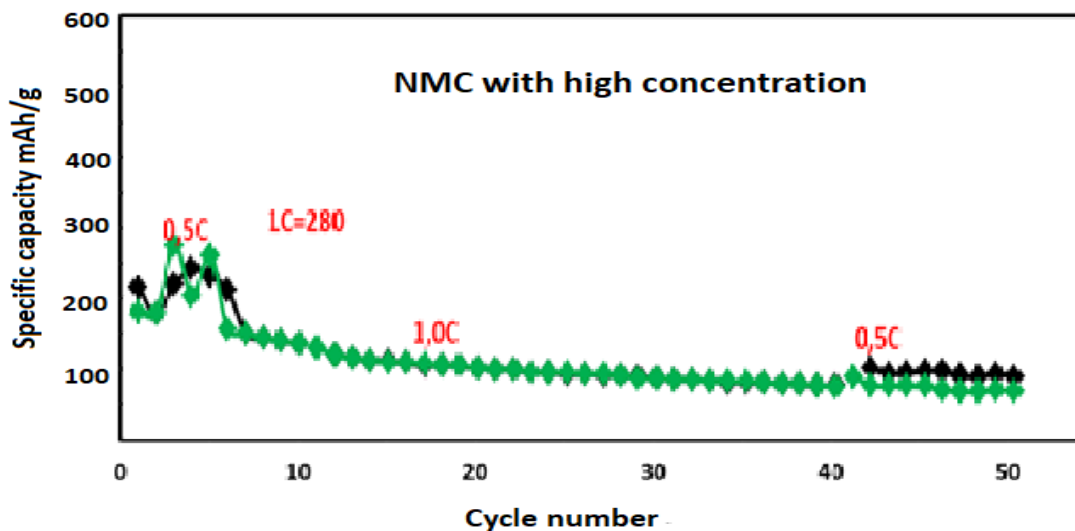


Figure 3.10. Graph of capacity versus number of cycles for NMC synthesized with high ammonia

The data obtained from the study showed that the battery performance depends on the ammonia concentration used during the synthesis of NMC 622. Although the use of low concentration ammonia decreases the specific capacity, it is seen that it increases the stability. However, the use of high concentration ammonia increased the specific capacity, it decreased the stability.

CONCLUSION

In this thesis, we investigated the interaction of ammonia concentration with morphology and electrochemical properties in the synthesis of cathode materials for high nickel content li-ion batteries. NMC622 powder was produced by using different concentrations of ammonia solution by co-precipitation method and its effect on electrochemical performance was observed. XRD and SEM analyzes and battery capacity tests were performed on these samples. According to the XRD results, the crystal structures of the materials synthesized by both methods are identical. The only noticeable difference is the peak intensity. Therefore, the NMC622 structure was successfully synthesized using both methods. In the SEM analysis, it was understood that the particles were in the range of 10 to 20 nanometers and the particle shapes were closer to the spherical structure of the NMC622 powder produced with 1M concentration ammonia. In the SEM analyzes performed on NMC622 powder produced with 0.5M concentration ammonia solution, the surface morphology of the samples was distorted and lost their spherical shape, and some clusters were observed. Cell coin batteries were made from the produced powders and subjected to electrochemical tests. As a result of these tests, the specific capacitance of the cathode material produce with high concentration ammonia was better, but the stability was lower than the NMC622 synthesized with low concentration ammonia. It has been observed that the cathode material synthesized with low concentration ammonia also supports the preservation of structural stability. According to all these results, the positive or negative effect of ammonia concentration on NMC cathode materials during synthesis was clearly understood, and it was observed that the material synthesized with high concentration improved the specific capacity and surface morphology, while decreasing the stability.

FUTURE PERSPECTIVES

In this study, NMC622 product was successfully synthesized. Testing products in a more sensitive battery test may yield more realistic results.

Also, it has been observed that the high ammonia syntheses clearly improve the surface morphology and capacity. When the obtained SEM images were examined, it was observed that although the high concentration NMC622 cathode material improved the spherical structure, its stability was impaired. Further studies are needed to increase the stability, and the product obtained in this direction promises much better results in the future. In order to achieve these goals, first of all, the synthesis conditions need to be improved. With more detailed studies on the product, features such as specific capacity and stability on the battery can be improved.

In addition, the synthesis method applied on NMC622 can be tested on other NMC forms. Better results can be obtained by applying this method for the NMC811 cathode material, which is the trend of the world battery market.

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