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EFFECT OF DIMENSIONS ON PRISMATIC POUCH BATTERY PERFORMANCE WITH NMC541 CATHODE MATERIAL

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ABSTRACT

Electrochemical reactions and ion exchange processes in Li-ion batteries not only depend on the properties of the electrode material and electrolyte, but also greatly depend on the geometric structure and size of the electrode. The distance between positive and negative current collector might have a significant effect. A battery with dimensions of 6x5 has the highest efficiency value in the 3rd cycle of 96.75657%, where two samples with larger dimensions have efficiency values below 80%. The maximum discharge capacity of the 6x5 sample is also the largest, namely 171.41829 mAh.g^{-1} with an rct value of 1.4675 Ω. Cyclic voltammetry test result also shows that NMC541-6x5 has the lowest potential difference (ΔE) which lead to better reversibility and kinetics of Li ions during charge transfer.

Keywords: *Cathode, Dimension, NMC541, Rct , Pouch Battery.*

1. INTRODUCTION

Battery performance not only depend on the material properties and electrolyte but also its size. By increasing the number of electrodes while keeping the volume constant, the surface area of the electrodes increases, which helps increase the efficiency of electrochemical reactions and facilitates ion exchange between the electrodes and the electrolyte [1] Due to the growing demand, research is ongoing to improve the performance of lithium-ion batteries. In the energy storage sector, rechargeable lithium-ion (Li-ion) batteries are crucial [2] [3]. Rechargeable battery technology enables widespread deployment of backup power for electric vehicles (EVs) and intermittent energy sources (such as solar and wind power), and reduces dependence on fossil fuels [4]. To meet the high energy demand especially for long-range electric vehicles, high-energy LIBs with long cycle stability need to be developed. Li-ion battery performance depends on cathode performance. The NMC offers a high specific energy density, leading to a higher reversible capacity [5]. One of the drawbacks is that high temperatures accelerate LIBs to degrade [6]. The process of delivering electric current to a battery takes place through a lithium intercalation-deintercalation mechanism in the structure of the cathode material [7]. Apart from the cathode, the dimensions of the battery also have a significant influence. In general, battery size is directly related to its storage capacity. Larger batteries have the capacity to store more energy than smaller batteries of the same type. It is important to note that the size of a battery is not directly related to its energy density, which is the amount of energy stored in relation to the weight or volume of energy. Battery cells come in many forms. The shape can be divided into cylindrical and prismatic, while the prismatic shape can be further divided according to the stability of the container into prismatic hard-case cells and prismatic pouch cells [8].

The research was conducted to determine the performance of batteries using NMC541 cathode material and graphite anode with fixed electrode sizes for varying battery dimensions. From this research, we aim to find out the optimal dimensions of the battery based on its electrochemical performance. Performance testing is carried out using LCR (Inductance, Capacitance and Resistance), EIS (electochemical impedance spectroscopy), CV (cyclic voltammetry), and CD (charge discharge) tests.

2. METHODOLOGY

This research consists of the process of making NMC541 cathode sheets, graphite anode sheets and the process of assembling and testing prismatic pouch batteries. Fig 1 below shows how the graphite anode sheets were made

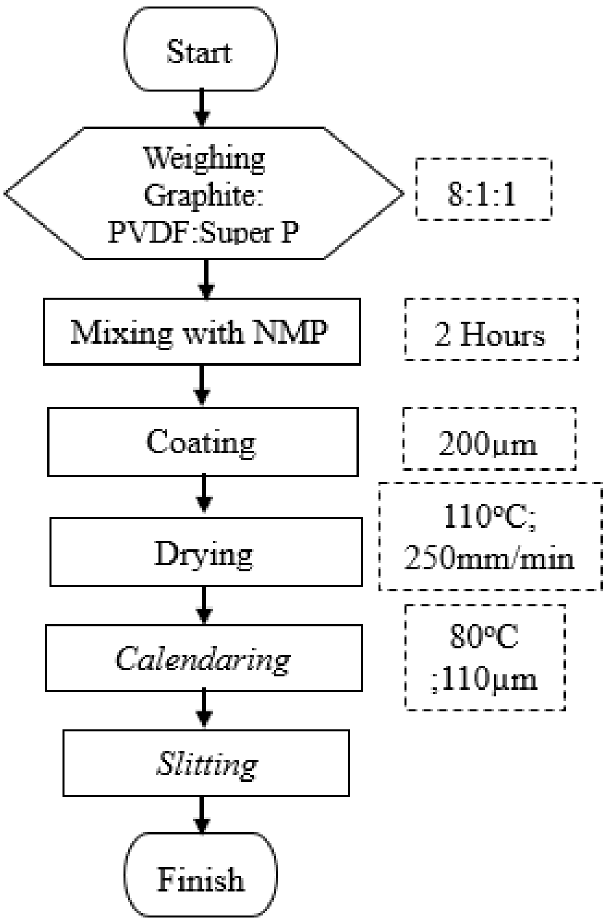

Fig 1. Making of graphite anode sheet

The manufacture of NMC541 cathode sheets is carried out in the same way as the flow diagram above, only using NMC541 material and a weighing ratio (active material: PVDF: super P) of (9:0.5:0.5). The finished cathode and anode sheets are then subjected to LCR testing first to determine the conductivity of the material.

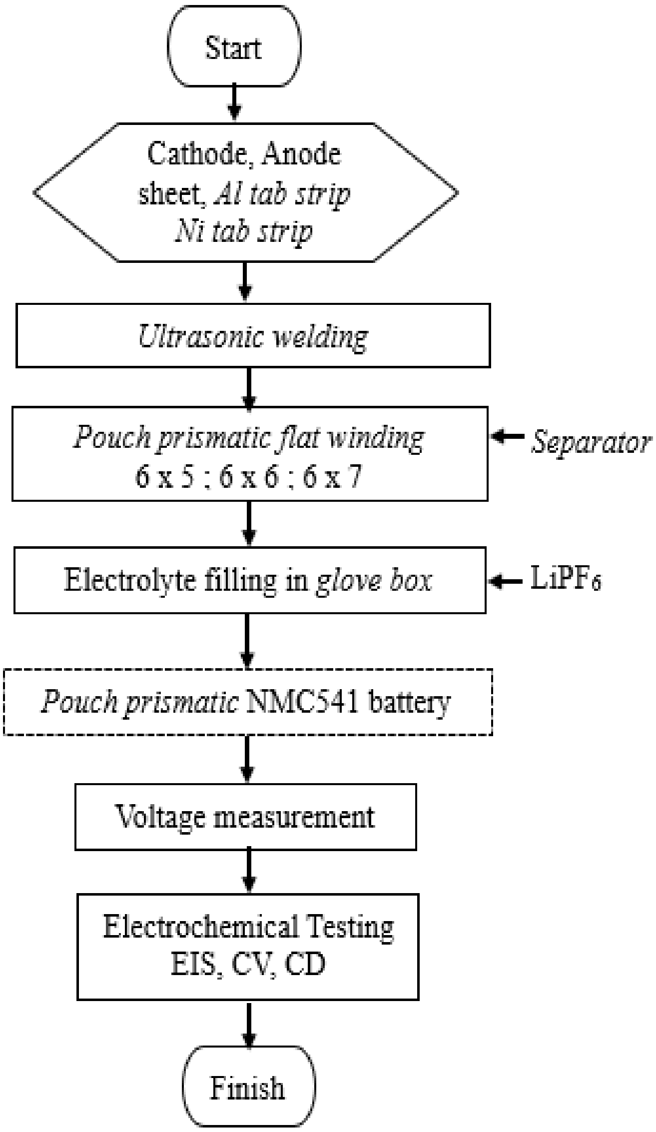

Fig 2. Assembly and pouch battery testing

3. RESULTS AND DISCUSSION

3.1. LCR Measurement

Conductivity is a value that affects the transfer of lithium ions from anode to cathode (discharge) and from cathode to anode (charge). If the conductivity of the material is good, the transfer of lithium ions will be fast. Conversely, if the conductivity of the material is poor, the transfer of lithium ions will be slower [9]. The cathode sheet formed was then tested by LCR to determine its conductivity with a test frequency variation of 42 Hz – 1 MHz and a clamp probe with an area of 0.785 cm^2 at room temperature. For each electrode two measurements were carried out.

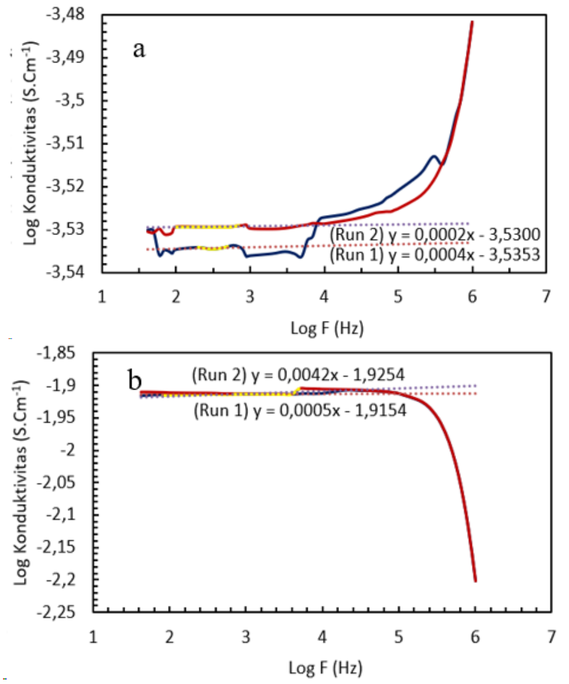

Fig 3. LCR measurement results a) cathode, b) anode.

Results of measuring the cathode conductance using the HIOKI LCR twice, values were obtained of 0.0002915 and 0.0002951 S.cm^{-1} with an average conductance value of 0.0002933 S.cm^{-1}. The thickness of the double side coated cathode was measured at 0.0228 cm. Results of measuring the anode conductance using the HIOKI LCR twice, the values obtained were 0.0121507 and 0.0118741 S.cm^{-1} with an average conductance value of 0.0120124 S.cm^{-1}. The measured thickness of the double side coated anode is 0.0181 cm.

3.2. Cyclic Voltammetry (CV)

To investigate the insertion and release of Li-ions in the sample cells, cyclic voltammetry (CV) testing was performed in the voltage range of 2.5 to 4.3 V at a scan rate of 60 µV/s.

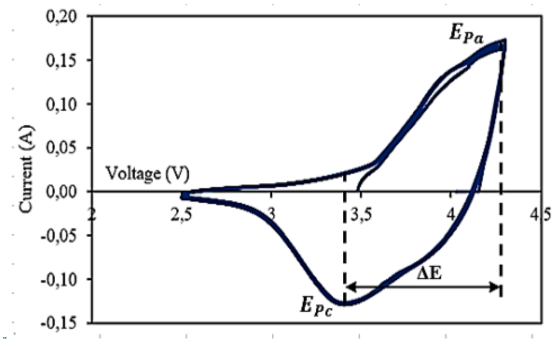

Fig 4. CV Result of NMC541-6x5

Fig 4. shows the results of CV NMC541-6x5, the anodic current is highest when it reaches the oxidation peak (Epa) of 4.2956 V. After experiencing oxidation, the current will decrease and turn towards negative to carry out the reduction process. The reduction process occurs at cathodic current, with a reduction peak (Epc) of 3.4836 V. This is carried out several cycles to determine the stability of the results obtained. The potential difference (ΔE), also known as the intercalation distance, indicates the speed of transfer of lithium ions in the electrolyte. This can affect the specific capacity of the battery. The potential difference or intercalation distance obtained in a sample measuring 6x5 cm is 0.8120V.

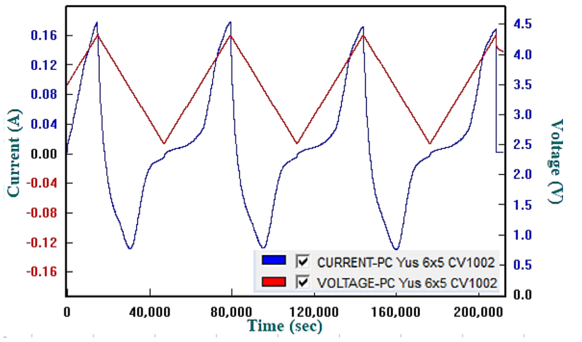

Fig 5. NMC541-6x5 CV test time

Fig 5. shows test time for this sample was around 58,3 hours. The potential difference of oxidation and reduction events indicates the reversibility of the battery material. The voltage potential difference (ΔE) qualitatively determines the degree of reversibility and kinetics of Li ions during charge transfer. The smaller ΔE value indicates better reversibility and high ion transfer kinetics during intercalation and deintercalation.

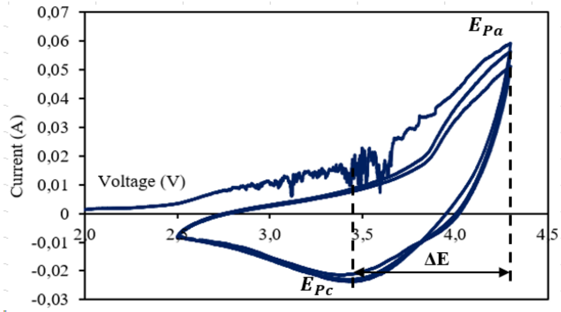

Fig 6. CV Result of NMC541-6x6

The image above shows the results of CV NMC541-6x6, the anodic current is highest when it reaches the oxidation peak (Epa) of 4.2997 V. After experiencing oxidation, the current will decrease and turn towards negative to carry out the reduction process. The reduction process occurs at cathodic current, with a reduction peak (Epc) of 3.4561 V. This is carried out several cycles to determine the stability of the results obtained. The potential difference (ΔE) or intercalation distance obtained in a sample measuring 6x6 cm is 0.8436 V. Fig 7. Below shows test time for this sample was around 72,2 hours.

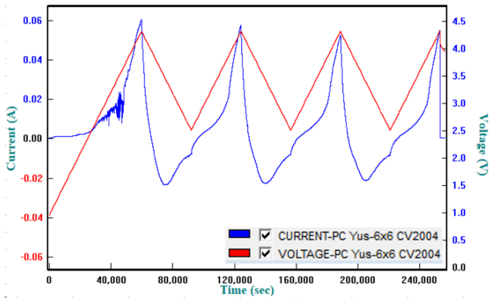

Fig 7. NMC541-6x6 CV test time

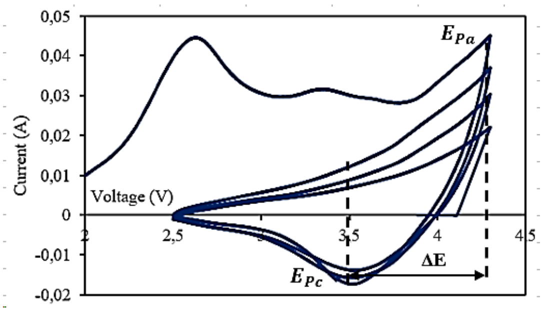

Fig 8. CV Result of NMC541-6x7

The image above shows the results of CV NMC541-6x7, the anodic current is highest when it reaches the oxidation peak (Epa) of 4.2994 V. After experiencing oxidation, the current will decrease and turn towards negative to carry out the reduction process. The reduction process occurs at cathodic current, with a reduction peak (Epc) of 3.4298 V. This is carried out several cycles to determine stability and ensure the results obtained. It can be seen that there is degradation in the battery cells which is indicated by the lines not stacking in the CV results. A line with a voltage below 2.5 V appears because the battery was initially at a low voltage condition. Another thing that might affect the results is poor contact between the electrodes, separator and electrolyte in the battery. The potential difference (ΔE), also known as the intercalation distance, indicates the speed of transfer of lithium ions in the electrolyte. This can affect the specific capacity of the battery. The potential difference or intercalation distance obtained in a sample measuring 6x7 cm is 0.8696 V. Fig 9. Below shows test time for this sample was around 75 hours.

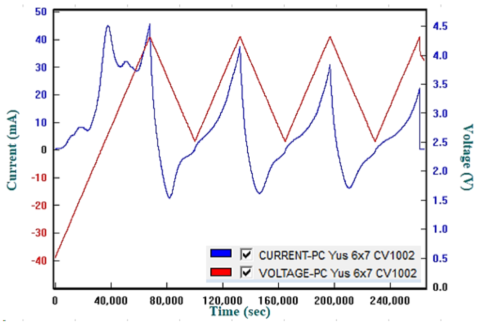

Fig 9. NMC541-6x7 CV test time

Table 1. Cyclic Voltammetry Results

Sample Code	Epa (V)	Epc (V)	ΔE (V)
NMC541-6x5	4,2956	3,4836	0,8120
NMC541-6x6	4,2997	3,4561	0,8436
NMC541-6x7	4,2994	3,4298	0,8696

Based on Table 1, it can be seen that the NMC541-6x5 sample has the lowest ΔE value so that the level of reversibility and kinetics of Li ions during charge transfer is said to be better than other samples.

3.3. Charge Discharge (CD)

The charge/discharge profile used and the time required differ for each battery manufacturer. In general, this process takes several hours to a day to complete because charge/discharge is carried out at a slow current rate ranging from 0.1C to 0.2C [10]. Initially, all batteries were tested for charge-discharge using cut-off voltage 2.8-4.6V and constant current of 0.0947 A or 0.1C using the theoretical capacity calculation of NMC532 which is closest to NMC541. The theoretical capacity of the NMC532 varies across sources from 160-186.675 $mAh.g^{-1}$. This is done to determine the maximum discharge capacity obtained by each sample, so that the C-rate calculation can be carried out according to each capacity. Below are the charge-discharge test results for the NMC541-6x5 sample:

Table 2. CD first 3 cycles of NMC541-6x5

Cycle	Cap Chg ($mAh.g^{-1}$)	Cap Dchg ($mAh.g^{-1}$)	Efficiency (%)
1	323.890	171.418	52.924
2	177.222	162.320	91.591
3	158.915	153.760	96.756

For the NMC541-6x5 sample in the first cycle charging stage it was able to achieve a charge capacity of 323.89050 $mAh.g^{-1}$, the discharge capacity was only 171.41829 $mAh.g^{-1}$, with an efficiency of 52.92476%. The highest efficiency value was achieved in the 3rd cycle, with a value of 96.75657%.

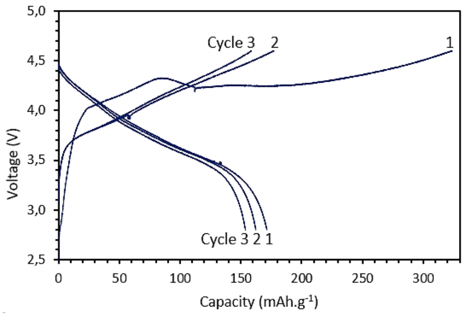

Fig 10. CD first 3 cycles of NMC541-6x5.

The charge capacity or Coulombic efficiency (CE) as a battery parameter to monitor the magnitude of side reactions, has been of great interest in recent years [11]. The charge/discharge efficiency is the ratio of the energy can be taken out from a battery (discharging capacity) divided by the energy can be put in (charging capacity) [12]. The charge-discharge results in the first cycle usually have low efficiency because the battery is not yet stable. Therefore, in the second and third cycles, it can be seen as in the picture below that the charge capacity value is getting closer to the discharge capacity value, so that the efficiency value increases. Generally, batteries are tested for tens or even hundreds of cycles to ensure the results obtained are stable.

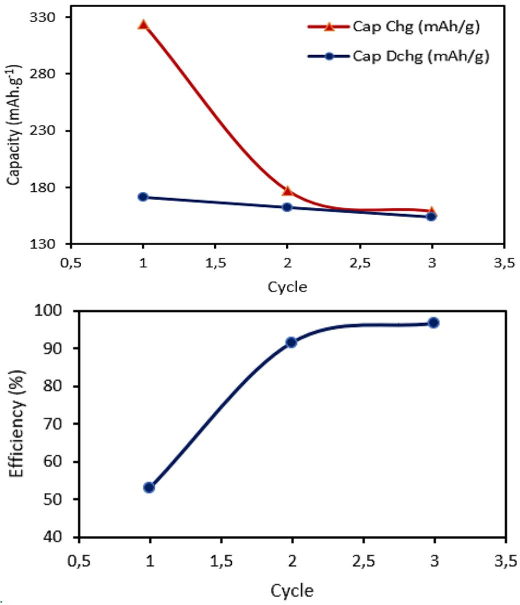

Fig 11. Specific capacity and Efficiency of the first 3 cycles of NMC541-6x5.

Below are the charge-discharge test results for the NMC541-6x6 sample:

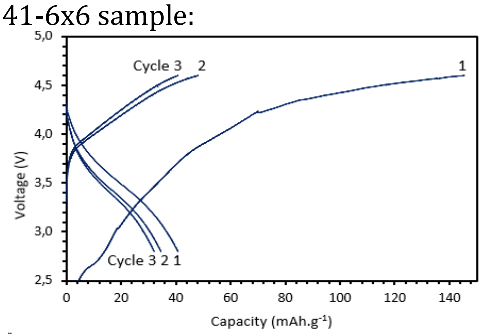

Fig 12. CD first 3 cycles of NMC541-6x6

For the NMC541-6x6 sample, in the first cycle charging stage, it was able to achieve a charge capacity of 145,467 mAh.g^{-1}, a discharge capacity of only 40,772 mAh.g^{-1}, with an efficiency of 28,029%. This efficiency value is 50% lower than the first cycle NMC541-6x5 sample. In the second and third cycles the NMC541-6x6 sample also shows the same thing, the charge and discharge capacity values begin to approach and efficiency also increases. The 3rd cycle efficiency value is 78,621%.

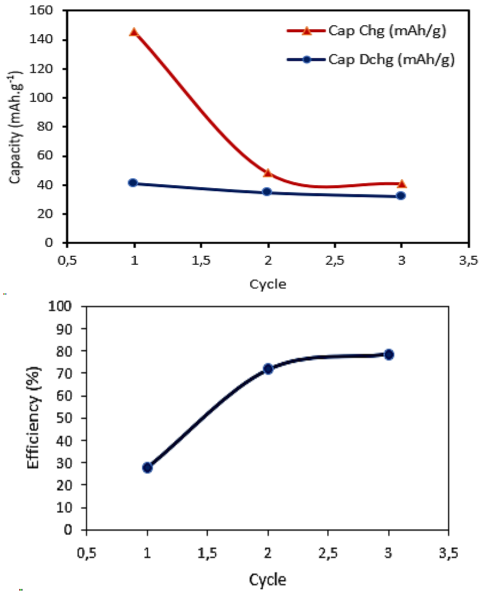

Fig 13. Specific capacity and Efficiency of the first 3 cycles of NMC541-6x6

Table 3. CD results of the first 3 cycles of the NMC541-6x6

Cycle	Cap Chg (mAh.g^{-1})	Cap Dchg (mAh.g^{-1})	Efficiency (%)
1	145,467	40,772	**28,029**
2	48,163	34,554	**71,744**
3	40,753	32,040	**78,621**

Below are the charge-discharge test results for the NMC541-6x7 sample:

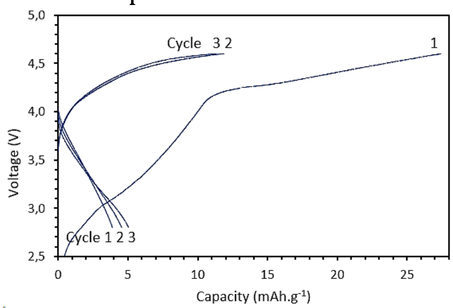

Fig 14. CD first 3 cycles of NMC541-6x7

The NMC541-6x7 sample in the first cycle charging stage achieved a charge capacity of 27.425082 mAh.g^{-1}, a discharge capacity of only 3.8802374 mAh.g^{-1}, with an efficiency of 14.148498%. This efficiency value is 73% lower than the first cycle NMC541-6x5 sample. In the second and third cycles, the NMC541-6x7 sample also showed the same thing, namely the charge and discharge capacity values began to approach and efficiency also increased, although it was still smaller than the other two samples. The greatest efficiency value was achieved in the 3rd cycle, namely 44.406368%.

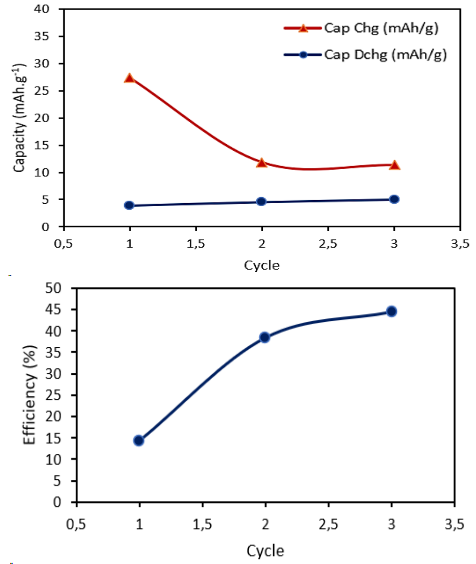

Fig 15. Specific capacity and Efficiency of the first 3 cycles of NMC541-6x7

Table 4. CD results of the first 3 cycles of the NMC541-6x7

Cycle	Cap Chg ($mAh.g^{-1}$)	Cap Dchg ($mAh.g^{-1}$)	Efficiency (%)
1	27,425	3,880	14,148
2	11,854	4,548	38,364
3	11,356	5,042	44,406

The NMC541-6x5 sample was tested for 50 cycles with a range of 2.8 – 4.3 Volts with a current of 0.0868A or 0.1C based on the first 3 cycle tests. The voltage range used corresponds to the anodic (oxidation) peak obtained from CV testing. Below is a graph of the charge-discharge results for 50 cycles.

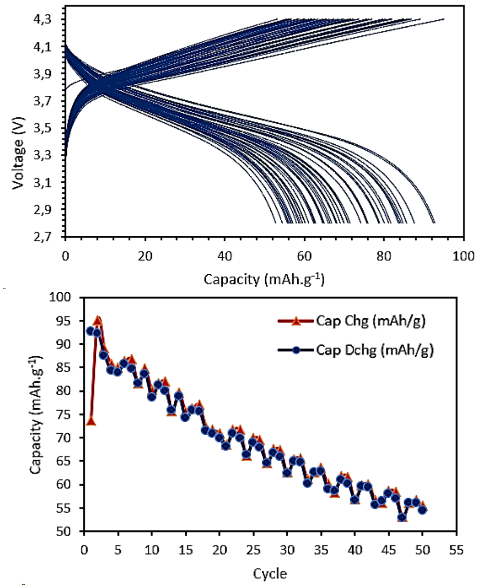

Fig 16. 50 cycles Charge discharge result of NMC541-6x5

From the data above, it can be seen that the higher the test cycle, the charge and discharge capacity values appear to decrease simultaneously. The efficiency value looks quite constant, approaching 100%, even though in the first cycle, the charge capacity value is smaller than the discharge capacity so that the efficiency value obtained exceeds 100%, namely 125.9%. This occurs because the battery condition is initially at a fairly high voltage approaching the cut off voltage limit of 4.3V, so that the recorded capacity during charge is small, then immediately enters the discharge stage.

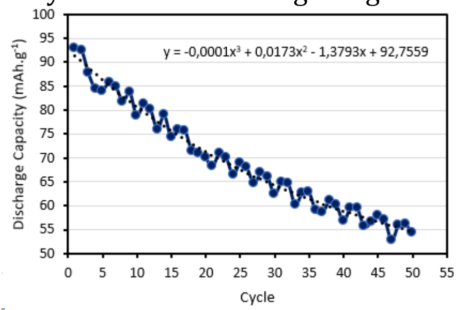

Fig 17. Capacity trend of NMC541-6x5

By using Ms.Excel we can display a trendline from existing specific discharge capacity data. We get the equation $Y=-0{,}0001X^3+0{,}0173X^2-1{,}3793X+92{,}7559$. We can use this equation to estimate in what cycle the battery will run out and cannot be used again. The Y value represents the capacity in $mAh.g^{-1}$ and the X value represents the cycle. In the 122nd cycle, the remaining capacity is 0,389 $mAh.g^{-1}$.

The NMC541-6x6 sample was tested for 50 cycles with a range of 2.8 – 4.3 Volts with a current of 0.0206 A or 0.1C based on the first 3 cycle tests. Below is a graph of the charge-discharge results for 50 cycles.

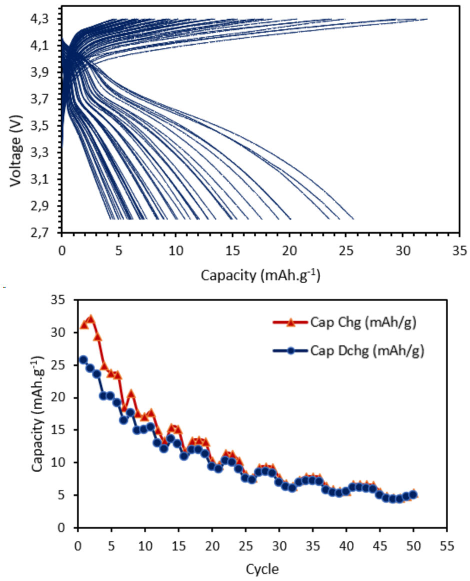

Fig 18. 50 cycles Charge discharge result of NMC541-6x6

The image shows that the charge capacity value of the NMC541-6x6 is not much different from its discharge capacity, so the efficiency value is around 80-90%. What is thought to be the cause of the low capacity value is because the contact between the electrode, electrolyte and separator is poor, the further the distance between the current collectors, the greater the resistance that must be passed.

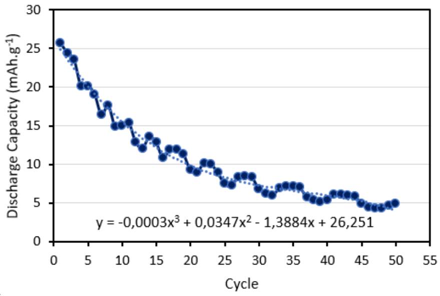

Fig 19. Capacity Trend of NMC541-6x6

By using Ms.Excel we can display a trendline from existing specific discharge capacity data. We get the equation Y=-0.0003X3+0.0347X2-1.3884X+26.251. We can use this equation to estimate in what cycle the battery will run out and cannot be used again. The Y value represents the capacity in mAh.g 1 and the X value represents the cycle. After calculating it was found that in the 65th cycle the remaining capacity was 0.225 $mAh.g^{-1}$

The NMC541-6x7 sample was tested for 50 cycles with a range of 2.8 – 4.3 Volts with a current of 0.0126A or 0.5C based on the first 3 cycle tests. A current value of 0.1C of 0.0025A is too small to be input on the charge discharge machine used, because the minimum current value that can be input is 0.01A or 10 mA.

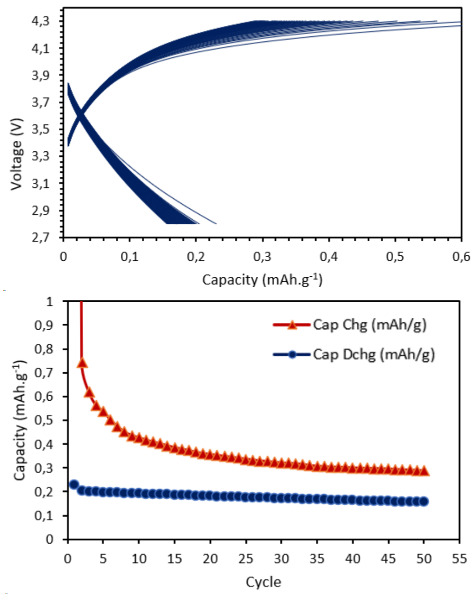

Fig 20. 50 cycles Charge discharge result of NMC541-6x7

This happens because the resulting battery capacity is too small. Below is a graph of the results of charge-discharge for 50 cycles. From Fig 20. it can be seen that the charge capacity value of the NMC541-6x7 is always greater than its discharge capacity, so the efficiency value is only around 50%, even lower in the first 10 cycles. One of the things that is thought to be the cause of the low capacity value is because the contact between the electrode, electrolyte and separator is poor, the farther the distance, the greater the resistance and diffusion distance that must be traversed.

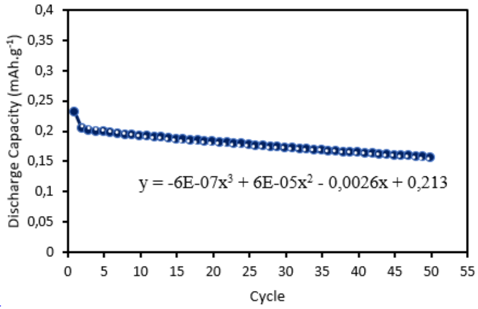

Fig 21. Capacity Trend of NMC541-6x7

By using Ms.Excel we can display a trendline from existing specific discharge capacity data. We get the equation $Y=-6x10^{-7}X^3+6x10^{-5}X^2-0{,}0026X+0{,}213$. We can use this equation to estimate in what cycle the battery will run out and cannot be used again. The Y value represents the capacity in $mAh.g^{-1}$ and the X value represents the cycle. In the 94th cycle, the remaining capacity is very small at 0.0004 $mAh.g^{-1}$. So that it can be compared, the remaining capacity is limited to 0.2-0.3 $mAh.g^{-1}$. In the second cycle of the 6x7 sample size, the capacity was 0.204 $mAh.g^{-1}$ From Table 5. it can be seen that the NMC541-6x5 sample has better capacity and efficiency performance than other samples.

Table 5. NMC541 Long Cycle Test Tabulation

Sample Code	Avg Cap Chg ($mAh.g^{-1}$)	Avg Cap Dchg ($mAh.g^{-1}$)	Avg Efficiency (%)	Life Cycle	Remaining Cap ($mAh.g^{-1}$)
NMC541-6x5	69.586	69.188	99.436	122	0.389
NMC541-6x6	11.878	10.349	89.487	65	0.225
NMC541-6x7	0.632	0.178	48.816	2	0.208

Rate capability or Charge discharge testing with varying currents is carried out to determine the characteristics of the battery at different current values. This test was carried out with sample NMC541-6x5. C-rates of 0.05 C, 0.10 C, 0.20 C, 0.30 C, 0.50 C or 0.043, 0.086, 0.174, 0.260, 0.434 A are used for 3 cycles each, which in total we did 15 cycles of charge discharge.

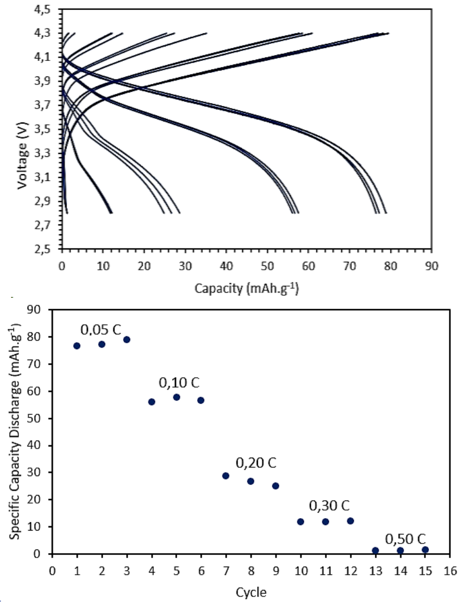

Fig 22. Effect of C-rate on battery capacity

As can be seen in the graph above, the higher the C-rate used, the lower the capacity value, at 0.5C the discharge capacity value obtained is only around 1.2–1.3 mAh,g^{-1}. Decreased capacity due to increasing current possibly caused by thickening of the solid electrolyte interface (SEI). [13].

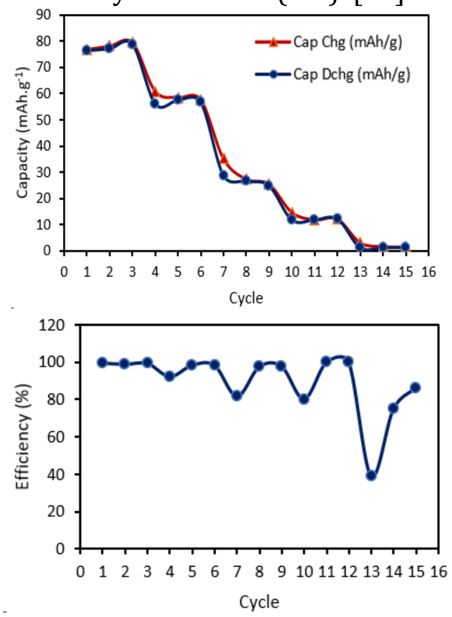

Fig 23. Effect of C-rate on specific capacity and efficiency

The rapid fading of the cell capacity at a charge and discharge rate of 0.5C is mainly caused by the loss of Li supply from the cathode structure and the metallic Li coating on the graphite electrode when the charge rate is high. Another analysis states that there is an increase in salt molarity in the electrolyte liquid at high charging rates. By increasing the charging rate, more capacity is lost due to limitations of Lithium ion transport [14]. Apart from that, other things happen when we increase the charging rate, such as increasing gas production, the gas produced by one electrode can migrate to the other electrode [14].

3.4 Electrochemical Impedance Spectroscopy (EIS)

Electrochemical Impedance Spectroscopy (EIS) is a non-destructive testing method commonly used to characterize lithium-ion batteries. Battery internal impedance is one of the characteristics that has a direct impact on battery voltage, efficiency and battery capacity. In general, impedance increases as battery cells age and the quality of the electrode material decreases [15] [16]. EIS (Electrochemical Impedance Spectroscopy) is used to characterize the electrical properties of the sample. The output data, the impedance value Z and the phase angle θ, are expressed in the form of a Nyquist plot as the relationship between the real part and the imaginary part of the impedance [17]. Below are the EIS measurement results for each sample:

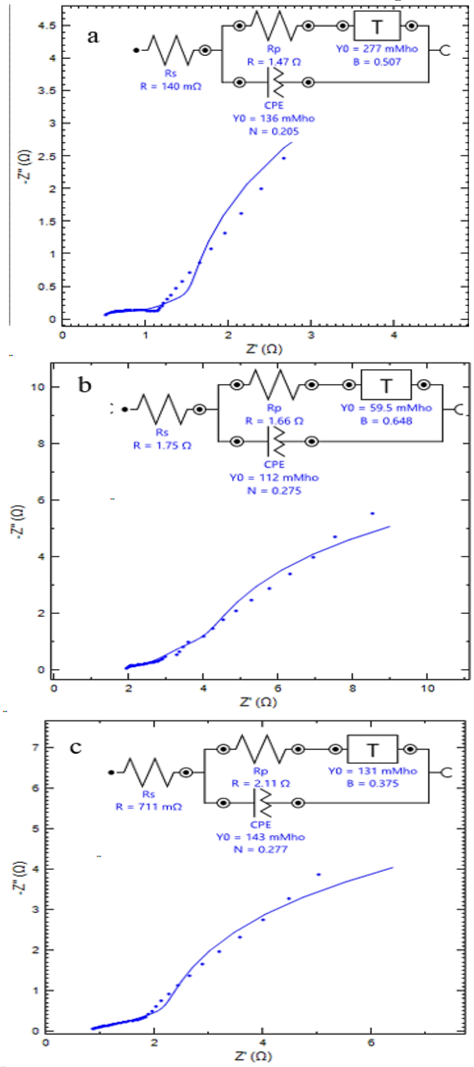

Fig 24. Nyquist plot a) NMC541-6x5, b) NMC541-6x6, c) NMC541-6x7

The adjusted EIS parameters are shown in Table 6. below. Generally, solution resistance (Rs) and charge transfer resistance (Rct) represent the Li^+ transport resistance and charge transfer resistance in the electrolyte, and are important parameters that greatly affect the electrochemical performance of cathode materials. At the initial stage of aging, the charge transfer resistance tends to decrease, probably due to an increase in double layer capacitance [18]. When studying coating phenomena, constant phase elements (CPEs) are often used instead of double layer capacitances (CDLs). The thickness of the protective layer on the metal surface is indicated by the decrease in the CPE value. A change in CPE value indicates that the water molecules on the metal surface have changed. Increasing the bilayer thickness after lowering the surface permittivity reduces the CPE value [18].

Table 6. EIS simulation and fitting test results

Sample Code	Rs (Ω)	Rct (Ω)	CPE (mMho)
NMC541-6x5	0,1402	1,4675	277
NMC541-6x6	1,747	1,656	60
NMC541-6x7	0,7112	2,1107	131

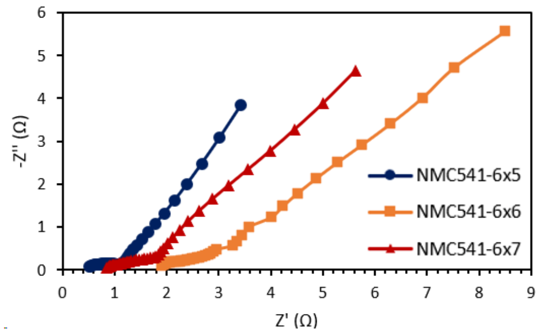

Fig 25. Combined Nyquist plot curve

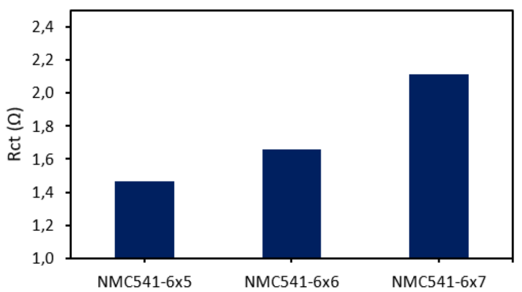

Fig 26. Graph of the influence of dimensions on resistance

The resistance results of all samples are shown in Fig 26. NMC541-6x7 has the largest Rct value, while NMC541-6x5 has the lowest Rct value. Based on Figure 25, the results of the combined Nyquist curve above show that NMC541-6x6 is further to the right compared to the other samples, however, based on Fig 26, Rct is 1.656 Ω. NMC541-6x7 has the largest Rct value, namely 2.1107 Ω. The greater the resistance, the smaller its ability to conduct electricity. The lowest Rct value is owned by NMC541-6x5 compared to other samples which have higher charge transfer resistance values. The low resistance is due to low polarization, which has been shown to improve electrochemical properties [19].

4. CONCLUSION

From the test results it was found that batteries with a fold size of 6x5 cm showed better electrochemical performance than batteries with larger fold sizes, namely 6x6 and 6x7 cm. A 6x5 battery has the highest efficiency value in the 3rd cycle of 96.75657%, where the other two samples have efficiency values below 80%. The maximum discharge capacity of the 6x5 sample is also the largest, namely 171.41829 $mAh.g^{-1}$ with an rct value of 1.4675 Ω. Cyclic voltammetry test result also shows that NMC541-6x5 has the lowest potential difference (ΔE) which lead to better reversibility and kinetics of Li ions during charge transfer.

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