AIP Conference Proceedings

RESEARCH ARTICLE | OCTOBER 06 2023

Understanding insight of commercial li-ion battery samsung 25R cylindrical type 18650

Evvy Kartini; Muhammad Fakhrudin; Alan J. Drew; Sigit Aryo Kristianto

AIP Conf. Proc. 2932, 020012 (2023)
https://doi.org/10.1063/5.0174814

Understanding Insight of Commercial Li-Ion Battery Samsung 25R Cylindrical Type 18650

Evvy Kartini[1, 2, a)], Muhammad Fakhrudin[1, 2], Alan J Drew[1, 3], Sigit Aryo Kristianto[1]

[1]*National Battery Research Institute, Indonesian Life Science Center, Technology Business Zone BRIN Puspiptek Area, Bogor, 16340, West Java, Indonesia*
[2]*Nano Material Research Organization, National Research and Innovation Agency, Puspiptek, Setu, South Tangerang 15314, Indonesia*
[3]*School of Chemical and Physical Sciences, Queen Mary University of London, 327 Mile End Road, London E1 4NS, United Kingdom, UK*

[a)] *Corresponding author: evvy.kartini@n-bri.org*

Abstract. Commercial Samsung 25R lithium-ion battery has been used as object of reverse engineering study. The aim of this study was to observe the physical parameters and insight of the cathode materials, that became a benchmark for developing the new materials. The first method was to dismantled the cylindrical cell, into the battery components. The cathode was coated on the Al-foil with the length of 875 mm and 570 mm width, and the thickness of about 1.41 mm. Meanwhile the anode was coated on the Cu-foil with the length of 930 mm and 570 mm width, and the thickness of about 1.28 mm. The length separator was 1820 mm, twice longer from the length of electrodes, and its width wider to prevent the short circuit. The separator was the thin sheet only 0.15 mm. The inside of the cylindrical cell was almost similar to other lithium-ion battery. The XRD pattern of the cathode material was identified as layered α-$NaFeO_2$ hexagonal structure belonged to the layered $LiNi_{1/3}Mn_{1/3}Co_{1/3}O_2$. According to the database, the diffraction pattern is the most compatible with NMC111 (ICSD 98-016-2295). It was found that, the peaks (006) and (012), as well as (018) and (110), are separated indicating the ordering of the layered hexagonal structure. The optimal c/a ratio was more than 4.899 exhibited the ideal c/a values, indicating the presence of cation mixing. A ratio of I(003)/I(104) was 2.5796, indicating desired cation mixing occurs. The sample's R value was low enough to indicate that it had good lattice ordering. In conclusion, the commercial Samsung 25R 18650 has proven to be good crystalline materials with layer NMC111.

INTRODUCTION

Lithium-ion battery (LIB) has become important issue on achieving the sustainable development goals through the clean energy [1][2]. The LIB has been applied not only in a daily life, for electronic devices such as mobile phone, laptop, iPad, camera, drone etc, but also in various emerging technology, from small scale in energy storage system to large storage system in electric vehicles [3][4]. Various developing technology of this lithium-ion battery mainly concerned to increase the performance in terms of capacity and working voltage window, reliability of usage of the battery in vehicle application, cost-effective manufacture, and the safety concerns [5].

From materials to cell fabrication, there are many aspects that affect the performance of the battery [6][7]. Such that aspects must be determined by trial-and-error experiments to obtain the parameters for optimizing performance of the battery. Given that in mind, each battery producers have their own recipe for achieving the best battery performance given from different material produced [6][8][9].

Recently there are significant increase of researches on cathode materials based on nickel, such as the $LiNi_{1-y-z}Mn_yCo_zO_2$ (NMC) and $LiNi_xCo_yAl_zO_2$ (NCA) with x + y + z = 1 [10]. This is due their better performance and higher capacity in comparison to the non-nickel based cathode [11][12]. The research showed also that the nickel rich cathode

The International Conference on Battery for Renewable Energy and Electric Vehicles (ICB-REV) 2022
AIP Conf. Proc. 2932, 020012-1–020012-7; https://doi.org/10.1063/5.0174814
Published by AIP Publishing. 978-0-7354-4692-2/$30.00

materials even exhibited better capacities, and among them, Ni-rich layered oxides, $LiNi_{0.8}Co_{0.15}Al_{0.05}O_2$ (NCA) and $LiNi_{0.8}Co_{0.1}Mn_{0.1}O_2$ (NCM811) are increasingly popular active cathode elements used in commercial LIBs for electric cars and planes [10][7][12].

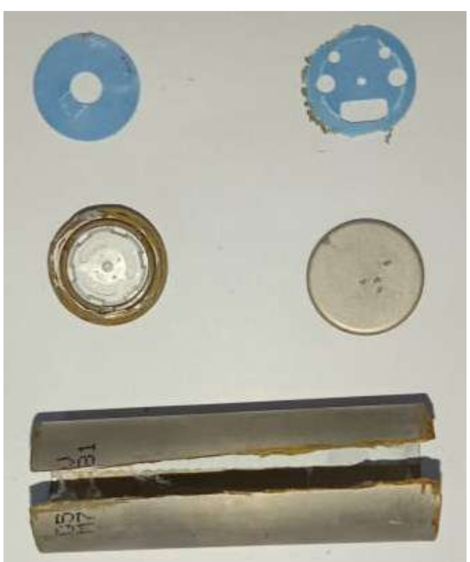
This paper gives insights of one of the commercial lithium batteries in market, Samsung 25R 18650. The reverse engineering process was conducted to study the insight of physical parameters, and the structural analysis of the materials that gave characteristic of the battery. By dismantling the cell, it will bring the information on the physical parameters and actual dimension in the cell [13][14]. It is also important to investigate the crystal structure of the cathode materials of this commercial lithium-ion battery and its morphology, so that more understanding what the market's need. The characterization was performed by using an X-ray diffractometer (XRD) and the morphology was conducted by a Scanning Electron Microscopy (SEM). It is expected that the result can be used as a bench mark for developing new materials with better battery performance. Aim of this paper would discuss comprehensively on the physical properties and insight of cathode materials, meanwhile the battery performance has been separately discussed in the other article in this issue.

METHODOLOGY

A commercial Samsung 25R lithium-ion battery was used as an object of reverse engineering study, which has a cylindrical shape with a diameter 18 mm and length 650 mm. It was obtained from commercial market. The cylindrical cell was dismantled to obtain the components inside such as anode, cathode, separator sheet, current collector, Tab and isolator as shown in Figure 1(a)-(d). The inside materials and physical parameters of the cell was observed. The dimension (length, width, thickness) of calendar roll of each anode and cathode were measured. The cathode material was measured by X-ray diffractometer to determine its crystal structure, and then it was analysed by using full proof software. The morphology of the cathode was observed by a Scanning Electron Microscope (SEM).

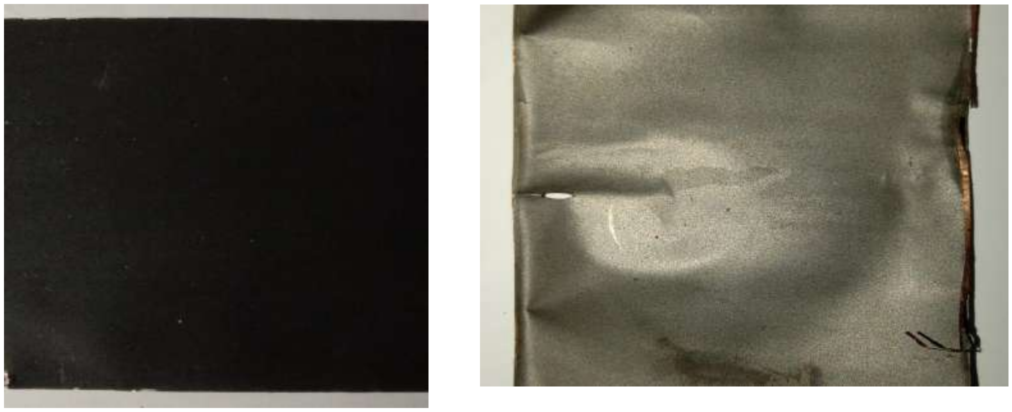
RESULTS AND DISCUSSION

Physical Parameters of Cylindrical Battery Components



(a)

(b)



(c) (d)

FIGURE 1. Disassembled results of Samsung 25R commercial battery 18650 (a) cylindrical parts, (b) battery components of the cylindrical cell, (c) cathode sheet materials, (d) anode sheet materials.

The disassembly process was conducted to study the physical parameters inside the commercial battery that gave the overall information of the battery. It is believed that every battery manufacturer has their own recipe of their battery that gives a good performance to be sold in market. Therefore, it is important to study this reverse engineering. Figure 1(a) show the parts of cylindrical cell from Samsung 25R 18650 that consisted of the Al-case, cover, isolator, connector and tabs. The Al-case has been cut to take off the battery components in rolled form, as seen in Fig.1(b). Then the cathode and anode sheets were opened from the rolled form, that respectively shown in Figs.1 (c) and 1(d). The cathode as a positive electrode was coated on the Al-foil with the length of 875 mm and 570 mm width, and the thickness of about 1.41 mm. Meanwhile the anode as negative electrode was coated on the Cu-foil with the length of 930 mm and 570 mm width, and the thickness of about 1.28 mm. The separator was 1820 mm, twice longer from the length of electrodes, and its width wider to prevent the short circuit. The separator was the pore material with the tiny sheet only 0.15 mm. The inside of the cylindrical cell was almost similar to other lithium ion battery, except the dimensions and the formula of cathode [15][16].

TABLE 1. Physical parameters of components inside the 18650 lithium-ion battery.

Components	**Length (mm)**	**Width (mm)**	**Thickness (mm)**
Negative electrode	930	57	1.28
Positive electrode	875	57	1.41
Separator	1820	60	0.15

Crystal Structure Observation of Cathode materials

Further analysis was to observe the insight of cathode materials from the commercial Samsung 25R 18650. The cathode materials were peeled from coated positive electrode on the Al-foil sheet. Then the crystallography characterization of cathode was conducted by an X-Ray Diffraction. The analysis is performed to determine the structure of the cathode material and also to retrieve the nickel-manganese-cobalt composition of the analysed material. The typical NMC cathode material with different composition can have similar XRD pattern so it needs to be analysed further to obtain the right composition of each materials [12].

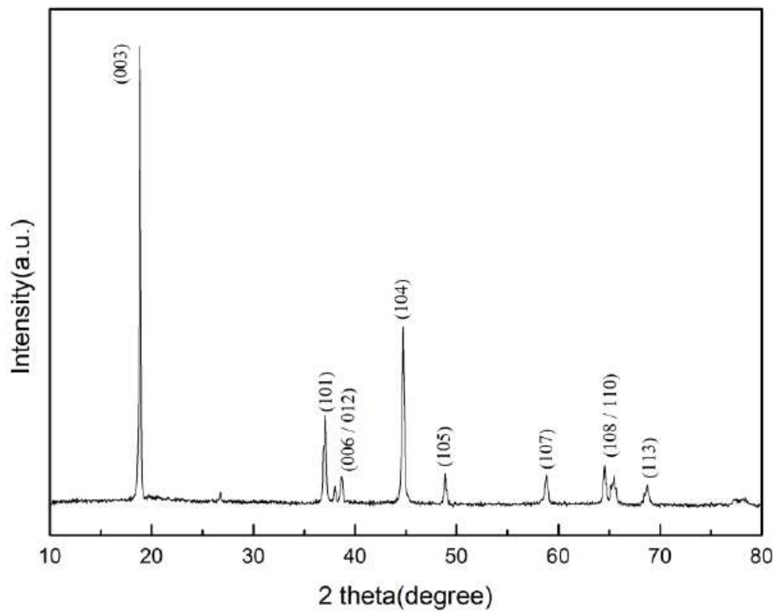


FIGURE 2. XRD analysis of the cathode material inside the Samsung 25R 18650 cell.

Figure 2 shows XRD patterns of Samsung cathode active material. The pattern was identified as layered α-$NaFeO_2$ hexagonal structure. According to the database, the diffraction pattern is the most compatible with NMC111 (ICSD 98-016-2295). It is then further analysed from the pattern that shows the Samsung cathode material composition of nickel-manganese-cobalt belongs to 1:1:1 (NMC 111). The earlier study of NMC111 have also been conducted by Feng Wu et.al [17]. The study showed that, the layered $LiNi_{1/3}Mn_{1/3}Co_{1/3}O_2$ materials with good crystalline have successfully been synthesized by a novel method of hydrothermal method followed by a short calcination process. The layered $LiNi_xMn_yCo_{1-x-y}O_2$ composites have attracted many attentions due to their high capacity, good cycling stability and excellent thermal stability

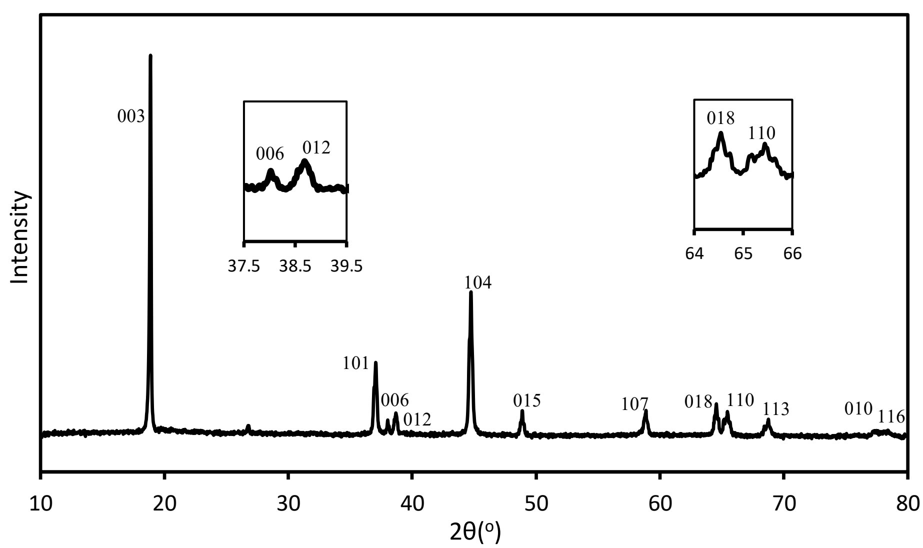


FIGURE 3. Diffraction pattern of Cathode Samsung 25R 18650 with the magnification of two pairs of nearby peaks is seen in the inset image.

The magnification of two pairs of nearby peaks is seen in the inset image in Fig.3. The separation of the peaks (006) and (012), as well as (018) and (110), indicates the ordering of the layered hexagonal structure [18][19] . Table 2 displays the Rietveld refinement and crystallite size analysis results. Despite cation mixing, the optimal c/a ratio is more than 4.899. The samples exhibit the ideal c/a values, indicating the presence of cation mixing while remaining within ideal limits. When the ratio of I(003)/I(104) is ≥1.2, desired cation mixing occurs. The cathode commercial material's I(003)/I(104) ratio was 2.5796, indicating appropriate cation mixing. The sample's R value was low enough to indicate that it had good lattice ordering. The lower the R value, the higher the lattice ordering of the NMC crystal structure [11][19].

TABLE 2. Rietveld refinement and size analysis results of XRD patterns.

Parameters	Samsung	Information
R_{exp}	2.38	
R_{wp}	3.66	
a (Å)	2.8606	
c (Å)	14.2687	
c/a	4.9880	Ideal: > 4.899
$I_{(003)}/I_{(104)}$	2.5796	Ideal: ≥1.2 (desirable cation mixing)
$R=[I_{(012)}+ I_{(006)}]/I_{(101)}$	0.4568	Lower → better
Crystallite size (Å)	27913	

Those parameters obtained from the refinement results of the measured commercial cathode corresponded to the layer NMC111 with an ideal condition. It has shown almost the ideal value of c/a that larger than 4.899; the ratio of $I_{(003)}/I_{(104)}$ is higher than 1.2 and the R value is lower. Showing the higher ordering. Similar results were also observed for the NMC111 cathodes synthesized by a novel method of hydrothermal method followed by a short calcination process. In their XRD pattern, the integrated peaks of (006)/(102) and (108)/(110) split, regardless of short calcination time. Indicating the layered $LiNi_{1/3}Mn_{1/3}Co_{1/3}O_2$ materials are successfully synthesized [17][19].

Morphology of the Commercial Cathode

The microstructural morphologies of the cathode material after several steps of charge-discharge process are shown in Figs.4(a)-(c). The SEM images of the Samsung commercial $LiNi_{1/3}Mn_{1/3}Co_{1/3}O_2$ are presented in Fig. 4(a),(b) and (c) with the enlargement of 1000x, 2500x and 5000x, respectively. It can be seen that the surface morphology was not smooth and homogenous. There are distinction composites, it agglomerated in several clusters but some paths existed in between surrounding the area (Fig.4(a). Further observed in more detail, the path seems like the crack, after the cathode being charge and discharge for 100 cycles. The average particle size of LiNi1/3Mn1/3Co1/3O2 was 300–500nm, which are greatly agglomerate.

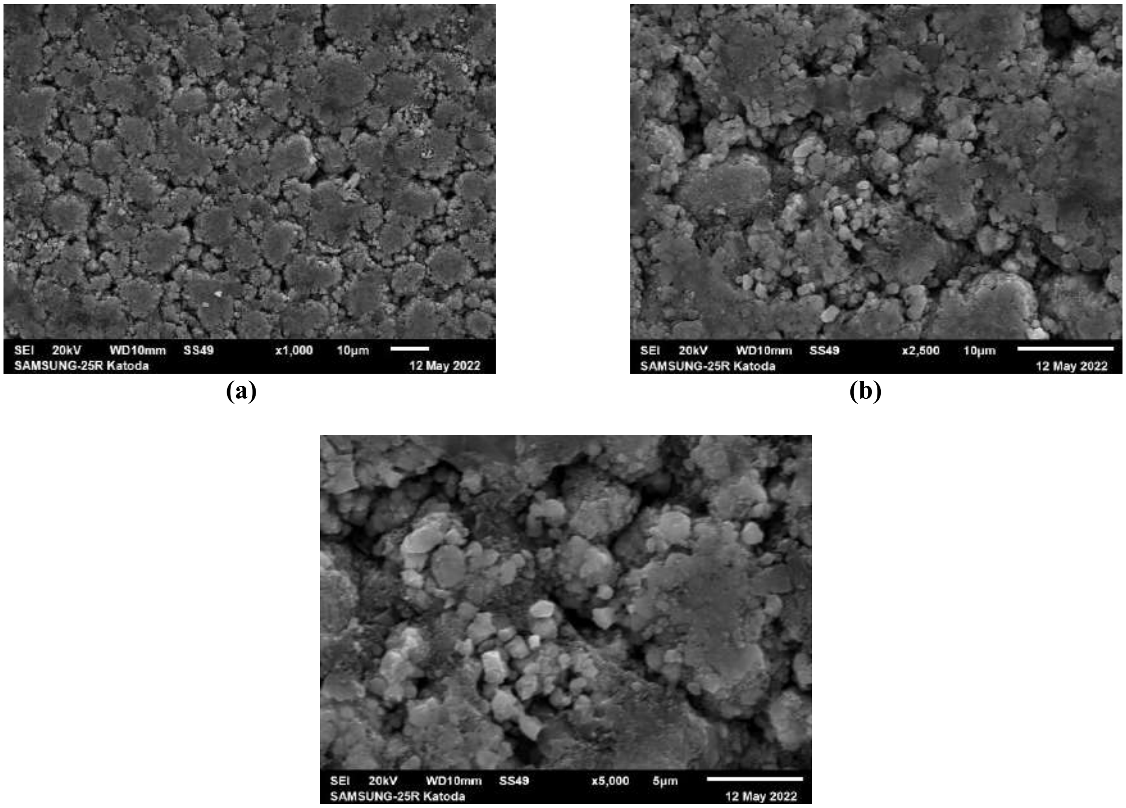


FIGURE 4. SEM observation of cathode material inside the lithium-ion battery in different zoom scaling (a) 1000x, (b) 2500x and (c) 5000x.

There is an evident distinction in morphology insight the cathodes. The cathodes, that in forms of square are composed of small layered particles (100–300nm) which are greatly agglomerate. After charging and discharging, rock-shaped grains with sharp edges morphology are formed, and the grain size increases to 200–400 nm. Figure 4(c) the images are magnified to 5,000 times, it is clearly to see that the distribution of the particles is much more uniform after charging treatment. It is known that particle shape and size of cathode materials were not only depending on the synthesize method but can also affect the energy density in practical use, so controlling particle morphology is very important [14][20].

CONCLUSION

The commercial Samsung 25R in the form of cylindrical cell 18650 has been successfully investigated via reverse engineering. The study showed that the cathode material corresponded to the the layered $LiNi_{1/3}Mn_{1/3}Co_{1/3}O_2$ materials with good crystalline. Several facts that this crystal has high ordering layered hexagonal structure, it is shown by the separation of the peaks (006) and (012), as well as (018) and (110). The Rietveld refinement and crystallite size analysis results, indicated the cation mixing, with the optimal c/a ratio is larger than 4.899. The ratio of I(003)/I(104) is 2.5796, which fulfilled the desired cation mixing. it had also good lattice ordering whereas R value was low enough to indicate that. It can be concluded that the cathode material of the commercial Samsung 25R 18650 has good crystallinity and high ordering.

ACKNOWLEDGMENTS

This work is financially supported by the National Battery Research Institute (NBRI) in fiscal year 2022.

REFERENCES

1. M.K. Saskia, E. Kartini, Current state of lithium ion battery components and their development, IOP Conf. Ser. Mater. Sci. Eng. 553 (2019) 0–9. https://doi.org/10.1088/1757-899X/553/1/012058.
2. I. Philips, J. Anable, T. Chatterton, E-bikes and their capability to reduce car CO2 emissions, Transp. Policy. 116 (2022) 11–23. https://doi.org/10.1016/j.tranpol.2021.11.019.
3. E. Kartini, S. Sudaryanto, W. Honggowiranto, A. Sudjatno, A. Fadjri, I. Marto, R.N.S. Rofika, Grading of Lithium Ion Battery Module for Public street lighting, IOP Conf. Ser. Mater. Sci. Eng. 553 (2019). https://doi.org/10.1088/1757-899X/553/1/012065.
4. C.P. Grey, D.S. Hall, Prospects for lithium-ion batteries and beyond—a 2030 vision, Nat. Commun. 11 (2020) 2–5. https://doi.org/10.1038/s41467-020-19991-4.
5. F. Schipper, E.M. Erickson, C. Erk, J.-Y. Shin, F.F. Chesneau, D. Aurbach, Review—Recent Advances and Remaining Challenges for Lithium Ion Battery Cathodes, J. Electrochem. Soc. 164 (2017) A6220–A6228. https://doi.org/10.1149/2.0351701jes.
6. D. Oeser, A. Ziegler, A. Ackva, Single cell analysis of lithium-ion e-bike batteries aged under various conditions, J. Power Sources. 397 (2018) 25–31. https://doi.org/10.1016/j.jpowsour.2018.06.101.
7. M.W.S. Mubarok, M. Fakhrudin, E. Kartini, Ce-doped NMC 811 synthesis as cathode material, Int. Conf. Adv. Mater. Technol. 2021. 2708 (2022) 070006. https://doi.org/10.1063/5.0122599.
8. C. Julien, A. Mauger, K. Zaghib, H. Groult, Optimization of layered cathode materials for lithium-ion batteries, Materials (Basel). 9 (2016). https://doi.org/10.3390/MA9070595.
9. A. Manthiram, A reflection on lithium-ion battery cathode chemistry, Nat. Commun. 11 (2020) 1–9. https://doi.org/10.1038/s41467-020-15355-0.
10. C.M. Julien, A. Mauger, NCA, NCM811, and the Route to Ni-Richer Lithium-Ion Batteries, Energies. 13 (2020) 6363. https://doi.org/10.3390/en13236363.
11. E. Kartini, M. Fakhrudin, W. Astuti, S. Sumardi, M.Z. Mubarok, The study of (Ni,Mn,Co)SO4 as raw material for NMC precursor in lithium ion battery, Int. Conf. Adv. Mater. Technol. 2021. 2708 (2022) 070001. https://doi.org/10.1063/5.0122596.
12. M. Fakhrudin, E. Kartini, La-incorporated NMC811 as a new Li-ion battery cathode material, Int. Conf. Adv. Mater. Technol. 2021. 2708 (2022) 070003. https://doi.org/10.1063/5.0123495.
13. Y. Lu, Y. Zhang, Q. Zhang, F. Cheng, J. Chen, Recent advances in Ni-rich layered oxide particle materials for lithium-ion batteries, Particuology. 53 (2020) 1–11. https://doi.org/10.1016/j.partic.2020.09.004.
14. C. Julien, A. Mauger, K. Zaghib, H. Groult, Optimization of layered cathode materials for lithium-ion batteries, Materials (Basel). 9 (2016). https://doi.org/10.3390/MA9070595.
15. V. Muenzel, A.F. Hollenkamp, A.I. Bhatt, J. de Hoog, M. Brazil, D.A. Thomas, I. Mareels, A Comparative Testing Study of Commercial 18650-Format Lithium-Ion Battery Cells, J. Electrochem. Soc. 162 (2015) A1592–A1600. https://doi.org/10.1149/2.0721508jes.
16. J. Liu, Q. Duan, K. Qi, Y. Liu, J. Sun, Z. Wang, Q. Wang, Capacity fading mechanisms and state of health prediction of commercial lithium-ion battery in total lifespan, J. Energy Storage. 46 (2022) 103910. https://doi.org/10.1016/j.est.2021.103910.
17. F. Wu, M. Wang, Y. Su, L. Bao, S. Chen, A novel method for synthesis of layered LiNi1/3Mn1/3Co1/3O2 as cathode material for lithium-ion battery, J. Power Sources. 195 (2010) 2362–2367. https://doi.org/10.1016/j.jpowsour.2009.10.043.
18. N. V. Kosova, E.T. Devyatkina, V. V. Kaichev, Optimization of Ni2+/Ni3+ ratio in layered Li(Ni,Mn,Co)O2 cathodes for better electrochemistry, J. Power Sources. 174 (2007) 965–969. https://doi.org/10.1016/j.jpowsour.2007.06.051.
19. A.M. Hashem, R.S. El-Taweel, H.M. Abuzeid, A.E. Abdel-Ghany, A.E. Eid, H. Groult, A. Mauger, C.M. Julien, Structural and electrochemical properties of LiNi 1/3Co 1/3Mn 1/3O 2 material prepared by a two-step synthesis via oxalate precursor, Ionics (Kiel). 18 (2012) 1–9. https://doi.org/10.1007/s11581-011-0635-2.
20. D.F. Fadrian, M. Wahyu, material prepared via carbonate co- precipitation method Via Carbonate Co-Precipitation Method, 070004 (2022) 4–9.