Research Article

Utilization of Spent Nickel Catalyst as Raw Material for Ni-Rich Cathode Material

Shofirul Sholikhatun Nisa[1,2], Anisa Raditya Nurohmah[1,2], Cornelius Satria Yudha[1,2], Hanida Nilasary[3], Hartoto Nursukatmo[3], Endah Retno Dyartanti[1,2], Agus Purwanto[1,2]*

[1] Department of Chemical Engineering, Faculty of Engineering, Universitas Sebelas Maret, Jl. Ir. Sutami No. 36A, Jebres, Surakarta, Indonesia 57126

[2] Centre of Excellence for Electrical Energy Storage Technology, Universitas Sebelas Maret, Jl. Ir. Sutami No. 36A, Jebres, Surakarta, Indonesia 57146

[3] Research & Technology Center, PT. Pertamina (Persero), Jakarta Pusat, Central Java, Indonesia 10110

* Corresponding Author, email: aguspurwanto@staff.uns.ac.id

Abstract

Spent nickel catalyst will be harmful to the environment if it is not processed or used properly. In fact, this waste still has a high nickel content. The treatment of spent nickel catalysts has been widely reported, but limited to nickel extraction. Since the lithium-ion batteries demand is continued to increase, then nickel is the most sought-after metal. Consequently, nickel from spent nickel catalysts could be developed as secondary source for lithium-ion battery cathode. This study aims to utilize spent nickel catalysts into more valuable materials. Nickel that has been extracted and mixed with Mn and Co has been used as raw material for nickel-rich cathode, namely NMC. Nickel extraction and NMC synthesis were using the acid leaching method followed by co-precipitation. Based on the functional test performed in this work, nickel from spent nickel catalyst can be applied to Li-ion batteries. The sintering temperature that gives good characteristics and electrochemistry was found 820°C. The galvanostatic charge-discharge test gave specific capacity results for NMC of 110.4 mAh/g. The cycle test showed that NMC synthesized from spent nickel catalyst can be carried out up to 50 cycles with a capacity retention of 87.18%.

Keywords: Spent nickel catalyst; NMC; lithium-ion battery

1.   Introduction

Nowadays, nickel is the most sought-after metal. Nickel is a part of the metallurgical, catalyst and even battery industries. As a catalyst, nickel is applied in the hydrogenation, hydrodesulfurization, hydro refining, and methanation processes (Abdel-Aal and Rashad, 2004). This industrial process can cause a decrease in the ability of the catalyst in chemical processes. The catalyst will lose its catalytic activity due to contamination on the external and internal surfaces of the pores, thus clogging the pores. The catalyst that has been contaminated can be regenerated. However, regeneration that is not economically viable is impossible and will cause significant waste disposal problems (Khalid and Athraa, 2017). Moreover, the metal content in the catalyst (especially nickel) has toxic and carcinogenic properties which are very

dangerous for the environment and humans (Genchi et al., 2020). This nickel catalyst solid waste is referred to as a spent nickel catalyst.

According to previous research, the nickel content in spent nickel catalyst in the form of elements and oxides is still high, around 12-50% (Abdel-Aal and Rashad, 2004; Esmaeili and Rahimpour, 2017; Parhi et al., 2013; Sahu et al., 2005). The high nickel content prompted researchers to recover nickel from spent nickel catalyst and succeeded in recovering up to 90% Ni (Hosseini et al., 2012; Le et al., 2017; Parhi et al., 2013; Ramos-Cano et al., 2016; Sahu et al., 2005; Sharma et al., 2015). In general, recovery of precious metals can be carried out by several methods, including electrometallurgy, pyrometallurgy, and hydrometallurgy (Khondabi et al., 2018). Researchers currently use the hydrometallurgical method because it has a simple process and provides high recovery results (Muzayanha et al., 2019). Hydrometallurgical methods include acid leaching using organic or inorganic acids as leachate acids. Among the leachate acids, hydrochloric acid provided the highest percent nickel recovery (Aaltonen et al., 2017; Joulié et al., 2014). This treatment is also well-known for a spent battery and electronic waste (Fornalczyk et al., 2013; Muzayanha et al., 2019). The recovery metal of the spent battery is reused for manufacturing new batteries. However, the treatment of spent nickel catalyst is only limited to the recovery process.

On the other hand, lithium-ion batteries are commonly used as energy storage. Photovoltaics and electric vehicles are a few examples of this application (Dobrzański et al., 2013). Expanding research on Ni-rich material (i.e., $LiNi_{0.8}Mn_{0.1}Co_{0.1}O_2$, $LiNi_{0.85}Co_{0.14}Al_{0.01}O_2$, and $LiNiO_2$) has the main advantage of its high specific capacity. High specific capacity is the main requirement of batteries for electric vehicle applications. Ni from spent nickel catalyst has the potential to be recycled as a Ni-rich cathode material for lithium-ion batteries, especially for $LiNi_{0.8}Mn_{0.1}Co_{0.1}O_2$ (NMC811). Thus, the utilization of nickel from spent nickel catalyst extraction can reduce environmental pollution and be used as a raw material for battery cathodes.

This study aims to utilize nickel recovery from spent nickel catalyst as raw material for NMC811. NMC was selected because it has a longer cycle life, moderate thermal stability and safety level when compared to NCA (Liu et al., 2018). The application of recovery nickel from spent nickel catalyst to NMC811 has not been reported in literature. The methods used include acid leaching for nickel extraction, coprecipitation for precursor synthesis, and sintering for the formation of the NMC cathode material.

2. Materials and Methods

2.1. Materials Synthesis

The synthesis of the $LiNi_{0.8}Mn_{0.1}Co_{0.1}O_2$ (NMC811) cathode material from spent nickel catalyst consists of two steps including the preparation of the precursors and the production of the cathode material. In the first step, the extrudate shape of the spent nickel catalyst (PT. Petro Oxo Nusantara, Indonesia) was crushed and sieved to obtain smaller particles. Then leaching process was carried out by preparing a 4 M HCl solution and heated to 80°C. The spent nickel catalyst powder was added to the HCl solution and leached for 3 hours under stirring at 600 rpm. The dark green filtrate obtained was separated from the residue. Then $MnSO_4.H_2O$ (Yaolong Chem, China) and $CoSO_4.7H_2O$ (Rubamin, India) were mixed into the filtrate with Ni: Mn: Co mole ratio = 8: 1: 1 and heated to a temperature of 60°C under stirring. Equimolar oxalic acid solution and NaOH solution were also prepared as precipitating agents and pH control separately. After that, the NaOH solution was added to the Ni-Mn-Co solution to pH 4. The oxalic acid solution was mixed thereafter to pH 2. The mixing process was carried out for 2 hours. Then, the precipitate material was precipitated, washed with distilled water, filtered, and dried in an oven.

In the second step, the NMC811 precursor was mixed with $LiOH.H_2O$ (Leverton, US) with a mole ratio of 1: 1.1. Then the mixture was pre-sintered at 450°C for 6 hours and sintered at various temperatures of

720°C, 770°C, and 820°C for 15 hours under oxygen flow. The NMC811 cathode material that was formed was crushed, sieved, and characterized.

2.2. Material Characterization

Initial testing was carried out on the composition of spent nickel catalyst using Energy Dispersive X-Ray (EDX) (Jeol JSM-6510LA, Tokyo, Japan). For both precursors and products, material characterization was carried out using X-Ray Diffraction (XRD), Fourier Transform Infra-Red (FTIR), and Scanning Electron Microscope (SEM). XRD (EQ-MD-10 precision mini XRD) was used to identify crystalline phases in materials employing CuKα radiation with λ = 1.5406 at 2θ. FTIR (Shimadzu FTIR Spectrometer, Japan) was used to detect functional groups of materials in the mid-IR region (4000-400 cm^{-1}). SEM (Jeol JSM-6510LA, Tokyo, Japan) was used to determine the morphology material.

2.3. Performance Test

A performance test was carried out on the 18650-type cylindrical cell. The cathode sheet was made by mixing the active material NMC811 (MA), acetylene black (AB), polyvinylidene fluoride (PVDF) with the mass ratio of MA: AB: PVDF = 92: 3: 5 and n-methyl-2-pyrrolidone (NMP) to become a slurry. Then the slurry was coated on an Al foil sheet and dried into a cathode sheet. Graphite and celgard are used as anodes and separators. Then, the cathode, anode and separator are assembled into a cylindrical cell battery. This performance test used the NEWARE Battery Analyzer and BTS Software.

3. Results and Discussion

3.1. Precursor Preparation

The metal composition of spent nickel catalyst was analyzed prior to the process and the results are shown in Table 1. As seen in Table 1, our spent nickel catalyst contains mostly nickel with a composition of 50.52% followed by O with a value of 36.13%. The rest with less amount is Al, Mo, Si, and Ca. The first process in handling spent nickel catalyst was acid leaching. The leaching process will help the co-precipitation process of Ni with Mn and Co. Leaching was carried out using hydrochloric acid as leaching agent because of its ability to dissolve nickel (Bayraktar, 2005). The 4 M hydrochloric acid concentration was used based on Joulie et al, (2012) who conducted research using variations of the 1-4 M hydrochloric acid concentration. The results showed that at the 4 M concentration, Ni was leached out properly and the leaching efficiency reached >80% (Joulié et al., 2014). The characterization of spent nickel catalysts and leached filtrate has been reported in our previous study (Nisa et al., 2021). The treated spent nickel catalyst recovered 90% Ni, and the residue no longer contains Ni. More details can be seen in Table 1. Based on the amount of recovered nickel, the cathode material NMC811 was made to maximize the use of nickel metal in this study.

Table 1. Results of EDX analysis of spent nickel catalyst and the residue

Metal	Spent nickel catalyst (%wt)	Residue (%wt)
Ni	50.52	0
O	36.13	67.32
Al	4.98	7.51
Mo	3.59	0
Si	3.34	25.17
Ca	1.44	0

Filtrate obtained from spent nickel catalyst leaching was mixed with Co and Mn salts and then co-precipitated using NaOH and oxalic acid. The precipitate formed was an oxalate precursor that was characterized using FTIR and the results is shown in Figure 1. As seen, the FTIR spectra shows peaks at 3363 cm^{-1} and 1611 cm^{-1} indicates the presence of an O-H group. Then, peak at 1312 cm^{-1} and 824 cm^{-1} show C-O vibrations (Nandiyanto et al., 2019). The presence of O-H and C-O peaks confirmed that the material formed was an oxalate compound $(C_2O_4.2H_2O)^{-2}$. In addition, there is also a Ni-O peak at 485 cm^{-1} (Jung et al., 2010).
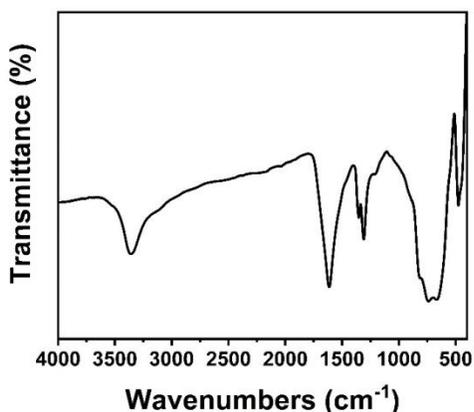
Figure 1. The FTIR analysis of NMC811 oxalate precursor

3.2. Product Analysis

Material characteristics, including structure, functional groups, morphology, and composition, were analyzed after the synthesis process. The pre-analyzed precursor was calcined and sintered under O_2 flow to form NMC811 material. Based on Figure 2, all synthesized NMC811 samples from spent nickel catalyst had the same diffraction peaks as the commercial NMC811 and no impurities were detected. All peaks are also the same as the Joint Committee on Powder Diffraction Standards (JCPDS) no. 20-0781. All samples showed a layered α-NaFeO₂-like hexagonal structure. The sharp peaks at the angle of 2θ 15-20° and 40-46° indicate that the material has high crystalline properties (Hsieh et al., 2016). Furthermore, the structural characteristics of the material are also determined by the lattice parameter and the R value.
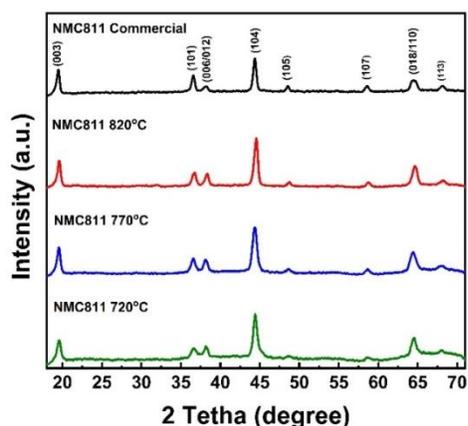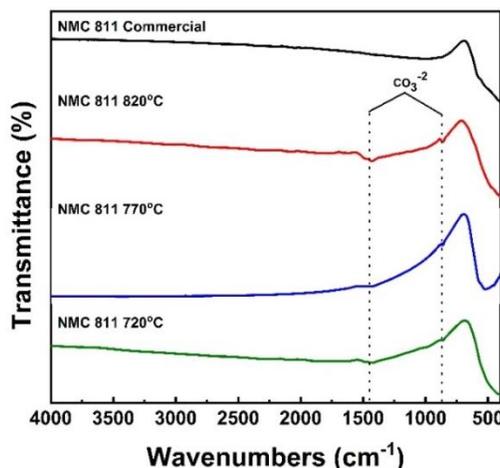

Figure 2. The x-ray diffraction pattern of the NMC811 cathode material at various temperatures

The lattice parameter was calculated by the least squares regression method (Yudha et al., 2019). Lattice parameter of the NMC811 shown in Table 2. The c/a value of the lattice parameter is a direct measurement of the deviation of the lattice from a perfect cubic close-packed lattice. Since the ideal cubic close-packed lattice has a c/a value of 4.899, the greater c/a ratio indicates a more ordered layered structure and the easier the transfer of Li (Xu et al., 2018). From Figure 1**Error! Reference source not found.**, we can conclude that all of the samples have c/a values of less than 4.899. The commercial NMC material has a c/a value 4.877. Eventhough it is less than 4.899, this value indicates that the material has a good ordered layered structure and the Li transfer.

Based on previous studies on Ni-rich cathode material, the higher the value of IR (I (003) / I (104)), the lower the cation mixing level of the material (Qiu et al., 2017; Yudha et al., 2019). All the sample has a relatively similar IR value with the commercial material. In addition, all samples have an R value of less than 0.45, the lower the value of R (I (006) / (I (101) + I (012))) is the better the hexagonal ordering (Kong et al., 2013). As shown in other samples, almost all of the samples seemed to have the optimum conditions for forming a good hexagonal structure of the NMC materials.

Table 2. Lattice parameter NMC811

| Sample	a (Å)	c (Å)	c/a	IR	R
NMC811 Commercial	2.88	14.05	4.88	0.75	0.35
NMC811 720°C	2.87	14.06	4.89	0.58	0.41
NMC811 770°C	2.88	14.16	4.87	0.74	0.46
NMC811 820°C	2.89	14.02	4.85	0.55	0.45

Figure 3. The FTIR peak of the NMC811 cathode material at various temperatures

The results of FTIR analysis to support XRD analysis are presented in Figure 3. Compared to its precursors, this FTIR spectra does not contain O-H because it has been lost during heating. All samples bore similarities to the commercial NMC811 spectra. However, commercial NMC 811 does not have CO_3 groups. Meanwhile, the three samples synthesized NMC811 from spent nickel catalyst at the wavenumbers ~1400 and ~800 cm^{-1} there are carbonate peaks. The presence of a carbonate peak can be attributed to the

excess of Li and prolonged exposure to air (Haik et al., 2010). LiOH can easily be converted to Li_2CO_3 due to the presence of CO_2 in the air and is considered an impurity (Seo and Lee, 2017). These impurities can be removed by washing, but worse for Ni-rich type cathodes washing after sintering can damage the structure of the NMC811. NMC811 is more sensitive to moisture and water will extract Li from the lattice (Langdon and Manthiram, 2021).

Morphological analysis is important to determine the morphology and particle size of the material presented in Figure 4. As shown in Figure 4., all samples consisted of submicron-sized primary particles agglomerated into secondary particles. At the sintering temperature of 720°C and 770°C the primary particles are quasi-spherical and more homogeneous. However, at 820°C the particle shape is less regular. Then, the higher the temperature, the larger the particle size obtained (Drezen et al., 2007). This is because the cathode material with high nickel content has a strong tendency to crystal/particle growth with increasing heating temperature (Ronduda et al., 2020). The primary and secondary particle sizes are presented in Table 3. The larger particles of the material have advantages in electrochemical performance.
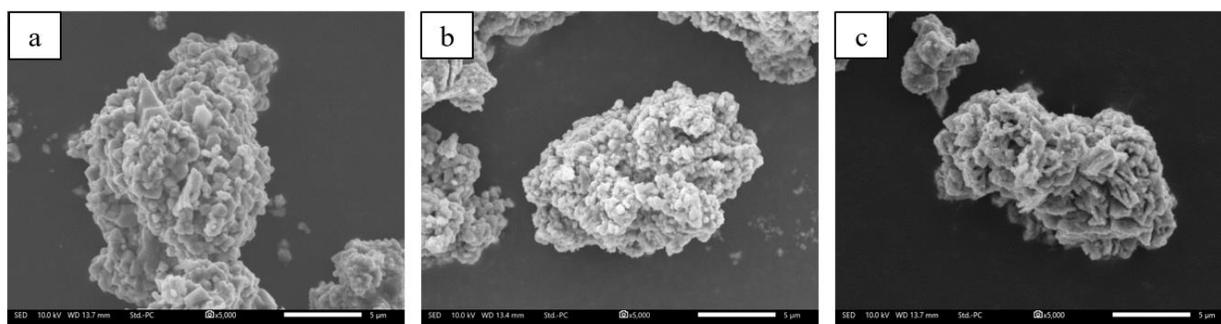
Figure 4. The SEM image of the NMC811 cathode material at various temperatures, (a) 720°C; (b) 770°C; (c) 820°C

Table 3. Particle size of NMC811

Sample	Primary Particle (μm)	Secondary Particle (μm)
NMC811 720°C	0.32-0.56	1.91-2.92
NMC811 770°C	0.41-0.58	3.20-6.00
NMC811 820°C	0.46-1.10	2.62-6.70

3.3. Electrochemical Performance

The electrochemical test was conducted to determine the charge-discharge capability of the NMC cathode. Figure 5 shows the charge-discharge capacity vs voltage of the NMC811 at various temperatures. During the initial cycle, NMC811 at 820°C had the highest charge-discharge capacities of 169.48 and 110.44 mAh g^{-1} at 0.1C. Whereas for temperatures of 770°C and 720°C, the discharge capacity obtained is 82.94 and 72.20 mAh g^{-1} at 0.1C, respectively. The lower discharge capacity at sintering temperatures of 720°C and 770°C maybe because the undispersed primary particles affect the electrolyte infiltration (Zhang et al., 2020). Then, the NMC811 820°C was subjected to a rate-ability test after the cell went through the formation phase for ten cycles at 0.1C. Rate-ability is shown in Figure 6a and carried out at 0.5C and 1C. The increased rate does not cause the discharge capacity to deviate significantly from the initial rate; in fact, it tends to stable.
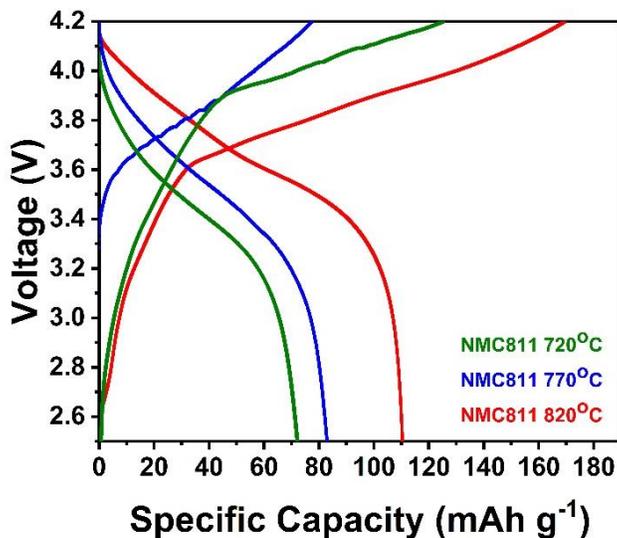
Figure 5. The charge-discharge of the NMC811 cathode material at 0.1C

It was discussed before that the temperatures below 820°C have a smaller particle size. Smaller particles can increase the formation of a solid electrolyte interface (SEI) resulting from unwanted electrode / electrolyte reactions leading to self-discharge and poor cycling ability (Sinha and Munichandraiah, 2009). Therefore, supported by the highest discharge capacity, the cycle-ability test was carried out on NMC811 at sintering temperature of 820°C. The cycle-ability test is carried out at 0.5C and presented at Figure 6b. During 50 cycles the NMC811 820°C has a capacity retention of 87.18% and a coulombic efficiency >98%. This capacity retention can predict the life cycle of the NMC811 battery cells and the results are quite high as the beginning of the application of spent nickel catalyst as a source of nickel for NMC811. The coulombic efficiency is also high and shows good stability.
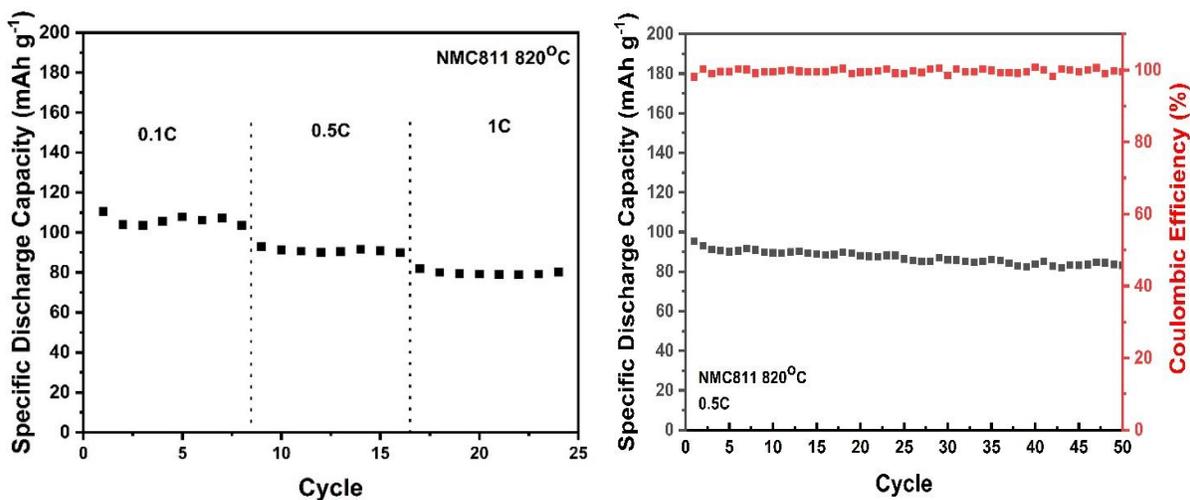

Figure 6. a) The rate-ability of the NMC811; b) The cycle-ability of the NMC811

4.   Conclusion

The utilization of nickel from the extraction of spent nickel catalysts has been successfully carried out in this study. The extracted nickel can be used as raw material for the synthesis of NMC811 cathode material. Based on x-ray diffraction, the NMC811 cathode material has a layered α-NaFeO₂-like hexagonal

structure. The slight impurity of CO_3^{2-} in the FTIR results is negligible. The morphology of the material obtained was quasi-spherical at a sintering temperature of 720°C and 770°C. In this synthesis process, both secondary and primary particles have a submicron size. The electrochemical test shows that material sintered at 820°C exhibited the best results with a discharge capacity of 110.44 mAh g^{-1}. The coulombic efficiency and capacity retention of the samples were >98% and 87.18% after 50 cycles, respectively. It shown that NMC with nickel from spent catalyst comparable with the commercial one because of its stability. The functional test of the NMC811 cathode material from spent nickel catalyst needs to be further investigated to maximize the waste utilization.

Acknowledgements

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