

# Washing Process for Nickel-rich Cathode Synthesized with Excessive Lithium Salts

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Excessive amount of lithium salts such as LiOH or Li<sub>2</sub>CO<sub>3</sub> are usually used when synthesizing lithium nickel manganese cobalt dioxide cathodes such as LiNi<sub>0.6</sub>Mn<sub>0.2</sub>Co<sub>0.2</sub>O<sub>2</sub> (NMC622) or similar to compensate for the loss of lithium salts at high temptations. Correspondingly, there will be salts left on the surface of as-synthesized NMC particle surface. A washing process is therefore necessary to remove the residual lithium salts which otherwise will cause the dehydrofluorination of the poly(vinylidene fluoride) binder[1-3] during the slurry preparation forming “gel”.

## Washing Process

The following process is recommended for laboratory-scale washing and annealing NMC particles without sacrificing its electrochemical performances.

1. Weigh the as-synthesized single crystal NMC powder in a glass beaker.
2. Add deionized water at a 5:1 mass ratio (DI water: NMC) for sufficient dilution to dissolve Li residuals.
3. Stir with a magnetic stir bar for 5 minutes.
4. Ultrasonicate the suspension for 5 minutes.
5. Stir again for 15 more minutes.
6. Set up the vacuum filtration apparatus and turn on the vacuum. Pre-soak the filter paper with DI water to make sure uniform flow, so particles do not pass through dry pores.
7. Pour the suspension onto the filter and allow the solid to collect while the Li residual solution drains.
8. Once the first drain is completed, rinse the filtered NMC with 250 mL DI water per wash. Repeat for a total of 6 washes (1.5L). Allow complete drainage between each wash to make each rinse step effective.
9. After the final rinse, leave the powder on the vacuum filter for an additional 15 minutes so that

it can dry partially to make transfer easier and reduce water content as much as possible before drying.

10. Transfer the filtered NMC (with filter paper) into a glass beaker and dry at 70°C under vacuum for 4 hours.
11. To repair the surface of NMC single crystals, anneal the dried powder at 580°C for 4 hours in oxygen atmosphere (ramp rate: 10°C/min)
12. Grind materials using mortar and pestle and sieve with a 400-mesh sieve. The final products obtained are used for slurry preparation and ready for electrochemical testing.

## References

1. Ross, G.J., et al., *Surface modification of poly(vinylidene fluoride) by alkaline treatment1. The degradation mechanism*. Polymer, 2000. **41**(5): p. 1685-1696.
2. Cho, D.-H., et al., *Effect of Residual Lithium Compounds on Layer Ni-Rich Li[Ni0.7Mn0.3]O2*. Journal of The Electrochemical Society, 2014. **161**(6): p. A920-A926.
3. Bi, Y., et al., *To Pave the Way for Large-Scale Electrode Processing of Moisture-Sensitive Ni-Rich Cathodes*. Journal of The Electrochemical Society, 2022. **169**(2).