The Synthesis and Study of Nanoparticles of Niobium Oxide as Negative Electrodes in Li-ion Batteries

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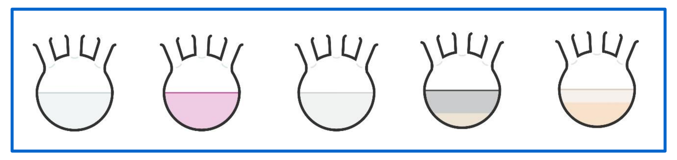
ABSTRACT

The demand for advanced electrode materials has seen an increase in recent years with the complexity of modern battery configurations. This study investigates nanoparticles of niobium oxide for use as negative electrodes in Li-ion batteries. Niobium oxide proves to be an impressive material for its capabilities in redox chemistry, mitigation of dendritic growth, and chemical stability. The presented synthetic route ensures inclusivity and affordability of the discussed materials, while the electrochemical and physical characterization completed uses various types of instrumentation including XRD, TEM, and SEM. The electrodes were tested in Li-ion half-cell batteries to determine capacity, rate performance, lifespan, and possible structural changes. Via a microcosmic approach, the advancement of these electrode materials could provide insight into future improved electrochemical performance.

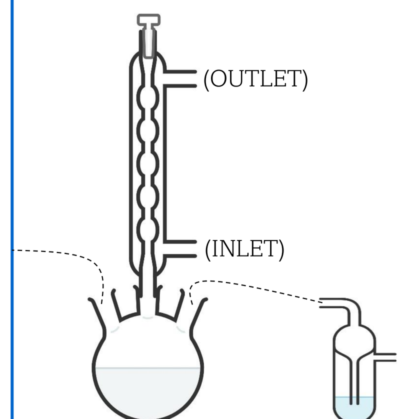
OBJECTIVE

Effectively synthesize $C_{20}H_{50}NbO_5$ precursor and relevant Nb_2O_5 nanoparticles for use as negative electrodes in operational Li-ion batteries, and investigate the performance principles of capacity, rate, life cycle, and structural transformation.

METHODS – (SYNTHESIS)



(Figure 1) – Depicts coloration shifts throughout reflux reaction



REFLUX

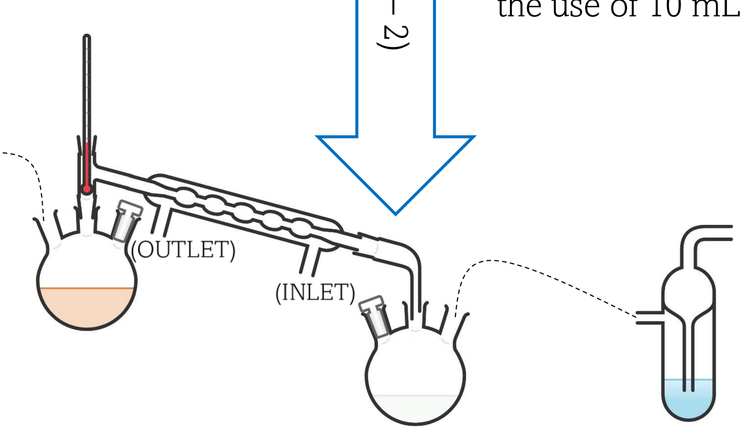
Reaction Conditions:
– Temperature: 110.6°C
– Solvent: Toluene
– Ran under constant argon pressure

Reaction Summary:
– Involves niobium chloride, butyl alcohol, ammonia, and toluene
– Mechanistically, butyl alcohol is deprotonated by ammonia (coloration shift (1-3) – Figure 1)
– Butyl alcohol preforms a nucleophilic substitution on the chlorine of niobium chloride (coloration shift (3-4) – Figure 1)
– Excess ammonia pushes reaction to completion (coloration shift (4-5) – Figure 1)

Balanced Reaction:
$NbCl_5 + 5\,NH_3 + 5\,ButOH \rightarrow Nb(OBut)_5 + 5\,NH_4Cl$
Theoretical Yield: 10.13 g (Calculations based off the use of 10 mL butanol – limiting reagent)

(DAY 1 – 2)

(1) In References



AZEOTROPIC DISTILLATION

Reaction Conditions:
– Temperature: 78.2°C
– Ran under constant argon pressure

Purification Summary:
– Azeotropic mixture: toluene/butyl alcohol
– Resembles the chemical properties of a singular compound under constant pressure
– Reaction vessel contains product w/ excess ammonia salt deposits
– Distillation vessel contain azeotropic mixture that was boiled out of product

(DAY 3)

AUTOCLAVE

Reaction Summary:
– Induces solvothermal synthesis
– Elevates pressure and temperature
– Reacts the various metal ions within the solution to form an amorphous powder

(2) In References

CHARACTERIZATION

The synthesized Nb_2O_5 was analyzed through 4 different variants of instrumentation, with the goal of confirming crystallinity, particle size, and the composition.

GOALS

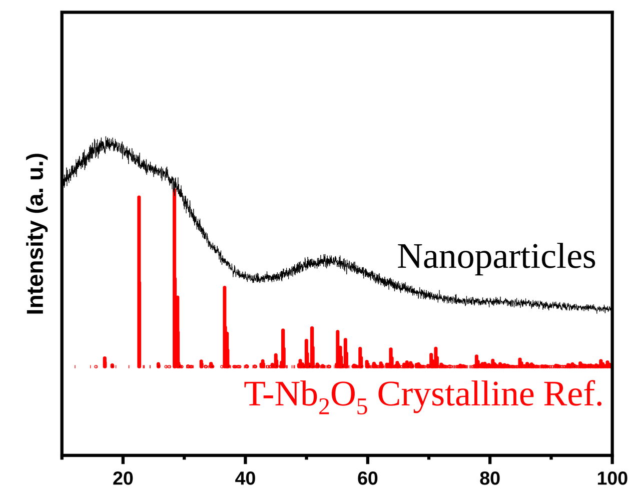
CRYSTALLINITY – XRD followed by HRTEM & SAED
– Structure of Nb_2O_5: amorphous characteristics.

PARTICLE SIZE – SEM followed by TEM
– Particle shape and approximate size.
– Particle size: ($\leq 100\ nm$); Standard Deviation: ($\leq 10\ nm$)

COMPOSITION – SEM; EDS
– Composition of Nb_2O_5
– Presence of Niobium and Oxygen

CRYSTALLINITY

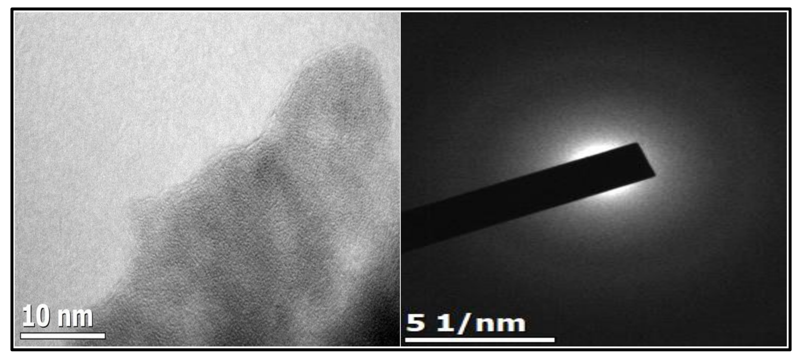
X-Ray Diffraction



(Figure 2) – XRD spectra collected on (Nb_2O_5) nanoparticles

Important Characteristics:
– Lack of definitive peaks; no periodic ray with long range order
– Amorphous; for further confirmation the sample was characterized using TEM:SAED

High Resolution Transmission Electron Microscopy & Select Area Electron Diffraction

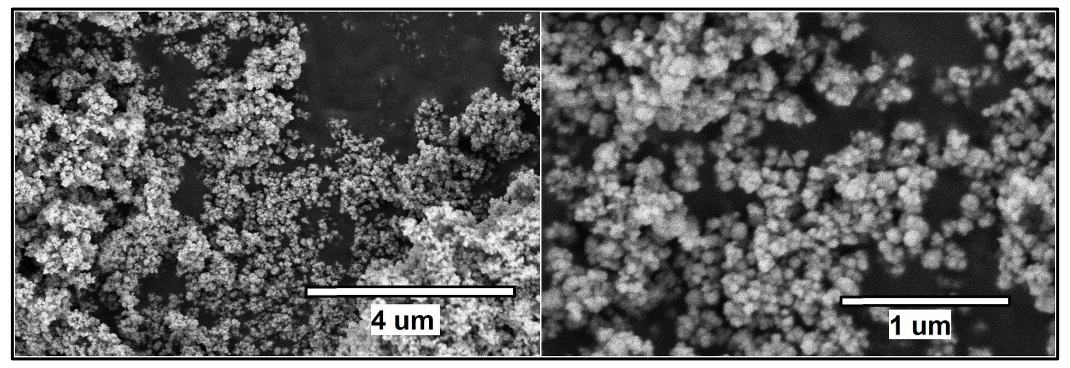


(Figure 3) – HRTEM image lacking latices & an SAED image illustrating diffused rings with no indication of crystallinity

Important Characteristics:
– Transmitted electrons create image in selected area of sample, diffused rings present in both projected images of separate clusters of nanoparticles
– Diffused rings indicative of amorphous crystallinity – confirming XRD suspicions and allowing study to move forward

PARTICLE SIZE

Scanning Electron Microscopy

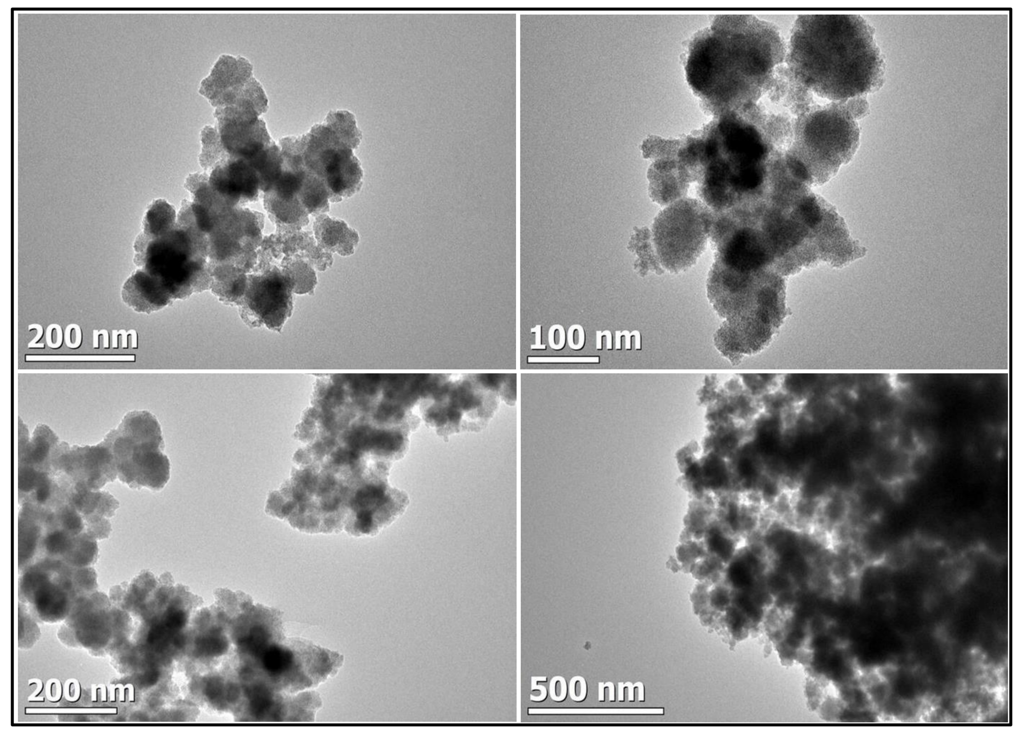


(Figure 4) – SEM images of Nb_2O_5 nanoparticles

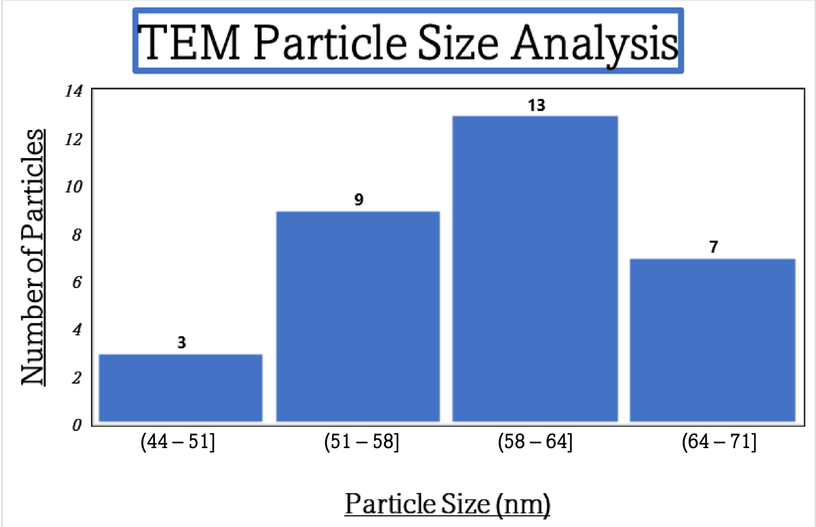
Important Characteristics:
– Surface topography; the secondary electron beam within the scanning electron microscopy instrument scans the surface of particles to gain an understanding of the shape and size of particles
– Scanned four different versatile areas of nanoparticles
– Circular shaped particles within massive clusters identified
– 8 – 10 approximate particles per cluster
– TEM was used to obtain more definitive insight on particle size

CHARACTERIZATION (CONT.)

Transmission Electron Microscopy



(Figure 5) – TEM images of four separate versatile clusters of Nb_2O_5 nanoparticles

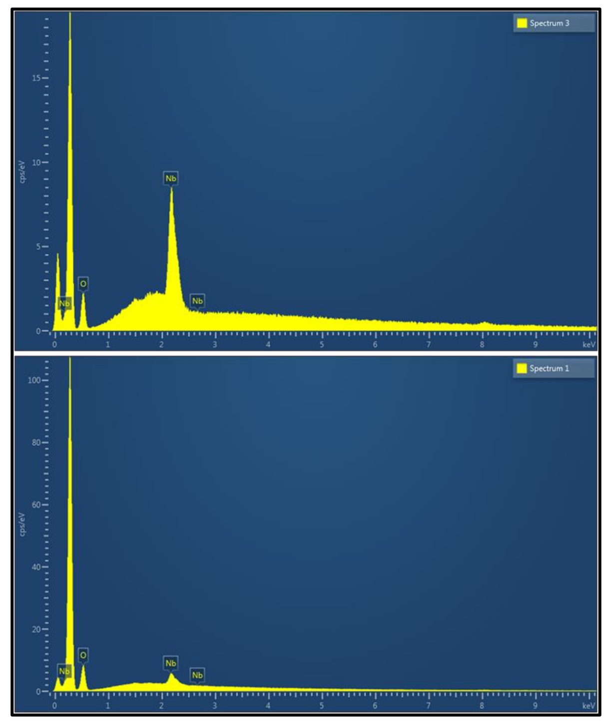


(Figure 6) – Histogram illustrates relationship between number of particles and particle size; effectively providing a visual for the standard deviation

Important Characteristics:
– Four different versatile nanoparticle clusters were selected and scanned using TEM instrumentation
– Particle size was then analyzed using ImageJ software
– 8 particle lengths / Cluster gathered
– AVG. Particle Size: (59.3 nm)
– Standard Deviation: (5.53 nm)
– Satisfies pre-existing standards

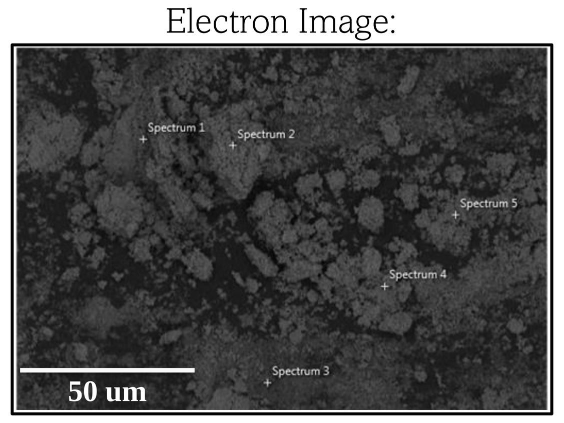
COMPOSITION

Scanning Electron Microscopy; Energy Dispersive X-Ray Spectroscopy



(Figure 7) – Graphical illustration of Nb_2O_5 nanoparticles obtained w/ SEM

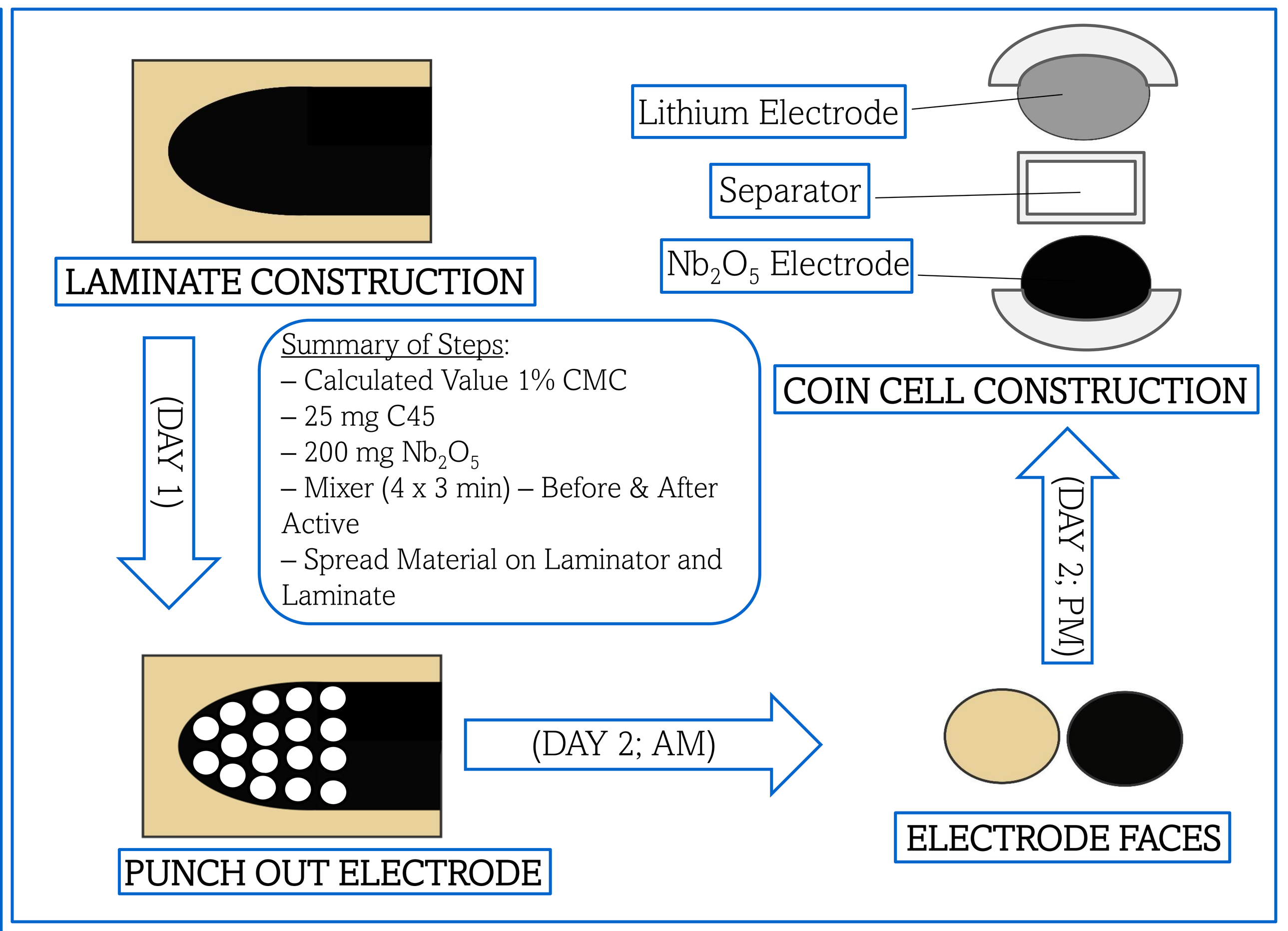
Electron Image:



(Figure 8) - Relevant SEM scanning region

Important Characteristics:
– Chose five versatile spectrums to scan – similar composition in each
– Huge carbon peak; used carbon tape
– Both necessary composition traits present (niobium & oxygen)
– Various oxidation state possibilities confirmed via XPS; still awaiting results

METHODS – (BATTERY CONSTRUCTION)



RESULTS

Table 1: (Experimentally Determined Values)

Synthesis Yield; Percent Yield (mL; %) (Pt. 1)	Synthesis Yield; Percent Yield (g; %) (Pt. 2)	Crystallinity	Particle Size; Standard Deviation (nm)	Composition	Electrode: (AVG. Weight) (g)	Electrode: (AVG. Thickness) (mm)	Coin Cell: (AVG. Voltage) (V)
6.76; 66.7	0.622; 51.4	Amorphous	59.3; 5.53	Niobium / Oxygen	0.011	0.032	2.64

(Table 1) – Collectively compiles the experimentally determined data from the study

Table 2: (Target Values)

Synthesis Yield; Percent Yield (mL; %) (Pt. 1)	Synthesis Yield; Percent Yield (g; %) (Pt. 2)	Crystallinity	Particle Size; Standard Deviation (nm)	Composition	Electrode: (AVG. Weight) (g)	Electrode: (AVG. Thickness) (mm)	Coin Cell: (AVG. Voltage) (V)
10.13; 100	1.21; 100	Amorphous	100; 10.0	Niobium / Oxygen	0.020	(0.04 – 0.05)	3.00

(Table 2) – Collectively compiles the target values in comparison to the data collected throughout this study

Future Directions:
– Testing! – (capacity, rate study, life cycle, and transformation capabilities)

REFERENCES

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(2) Kominami, Hiroshi, et al. "Novel Solvothermal Synthesis of Niobium (v) Oxide Powders and Their Photocatalytic Activity in Aqueous Suspensions." *Journal of Materials Chemistry*, 18 Dec. 2000, pubs.rsc.org/en/content/articlelanding/2001/JM/b008745i.

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