Research Performance Progress Report (Final)

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EXECUTIVE SUMMARY

For this Vehicle Technologies Incubator/Energy Storage R&D topic, Lambda Technologies teamed with Navitas Systems and proposed a new advanced drying process that promised a 5X reduction in electrode drying time and significant reduction in the cost of large format lithium batteries used in PEV's. The operating principle of the proposed process was to use penetrating radiant energy source Variable Frequency Microwaves (VFM), that are selectively absorbed by the polar water or solvent molecules instantly in the entire volume of the electrode. The solvent molecules are thus driven out of the electrode thickness making the process more efficient and much faster than convective drying method.

To evaluate the Advanced Drying Process (ADP) a hybrid prototype system utilizing VFM and hot air flow was designed and fabricated. While VFM drives the solvent out of the electrode thickness, the hot air flow exhausts the solvent vapors out of the chamber. The drying results from this prototype were very encouraging. For water based anodes there is a 5X drying advantage (time & length of oven) in using ADP over standard drying system and for the NMP based cathodes the reduction in drying time has 3X benefit. For energy savings the power consumption measurements were performed to ADP prototype and compared with the convection standard drying oven. The data collected demonstrated over 40% saving in power consumption with ADP as compared to the convection drying systems. The energy savings are one of the operational cost benefits possible with ADP.

To further speed up the drying process, the ADP prototype was explored as a booster module before the convection oven and for the electrode material being evaluated it was possible to increase the drying speed by a factor of 4, which could not be accomplished with the standard dryer without surface defects and cracks. The instantaneous penetration of microwave in the entire slurry thickness showed a major advantage in rapid drying of the electrode materials.

For the existing electrode materials, the material analysis and cell characterization data from ADP dried electrodes showed equivalent (or slightly better) performance. However, for high loading and thicker electrode materials (for high energy densities) the ADP advantages are more prominent. There was less binder migration, the resistance was lower hence the current capacities and retention of the battery cells were higher.

The success of the project has enabled credible communications with commercial end users as well as battery coating line integrators. Goal is to scale ADP up for high volume manufacturing of Li-ion battery electrodes.

The implementation of ADP in high volume manufacturing will reduce a high cost production step to bring the overall price of Li-ion batteries down. This will ultimately have a positive impact on the public by making electric and hybrid vehicles more affordable.

Project Goals and Actual Accomplishments:

A novel Advanced Drying Process (ADP) was proposed with a goal of 5X reduction in electrode slurry drying time and a 30-50% reduction in energy and hence operating costs. As a result one would anticipate a significant reduction in the overall cost of large format lithium batteries used in Plug-in Electric Vehicles.

A hybrid prototype system utilizing Variable Frequency Microwaves (VFM) and hot air flow was designed and fabricated and the results from this ADP equipment were compared to the standard drying method.

As described in the pages to follow, for water based anodes there is a 5X drying advantage in using ADP over standard drying system and for the NMP based cathodes the reduction in drying time has 3X benefit. Continuous coating at these conditions was employed to fabricate electrodes for lithium ion high energy cells (HEC), which were characterized to be as good as or better than the standard drying method.

The power consumption measurements were performed to ADP prototype and compared with the convection standard drying oven. The data collected (details below) demonstrates a > 40% saving in power consumption with ADP as compared to the convection drying systems. The energy savings are one of the operational cost benefits possible with ADP. Please note that the objective of the project was a 30-50% reduction in energy and cost.

The project activities for the entire funding period have been summarized in the following sections, which include the figures and characterization performed on the electrode materials as well as the fabricated battery cells.

Section I, Accomplishments & Milestones Update:

Milestone	Type	Status
Determine the appropriate Static/Dynamic Processing Parameters	Technical	Completed on January 30, 2015
Complete Microwave Choke Prototype Testing	Technical	Completed on April 15, 2015
Complete Design Review	Technical	Completed on February 24, 2015
Complete Factory System Acceptance evaluation	Go/No Go	Completed on July 22–23, 2015
Completed ADP Prototype was shipped and integrated at Navitas coating line	Technical	Installation on September 22–25, 2015
Electrode property evaluation and cell fabrication	Technical	Completed
Cycle life testing	Technical	Completed
Project ended	Technical	November 30, 2016

Static/dynamic processing parameters

To evaluate the static electrode processing parameter an apparatus with proposed features shown below had to be set up.

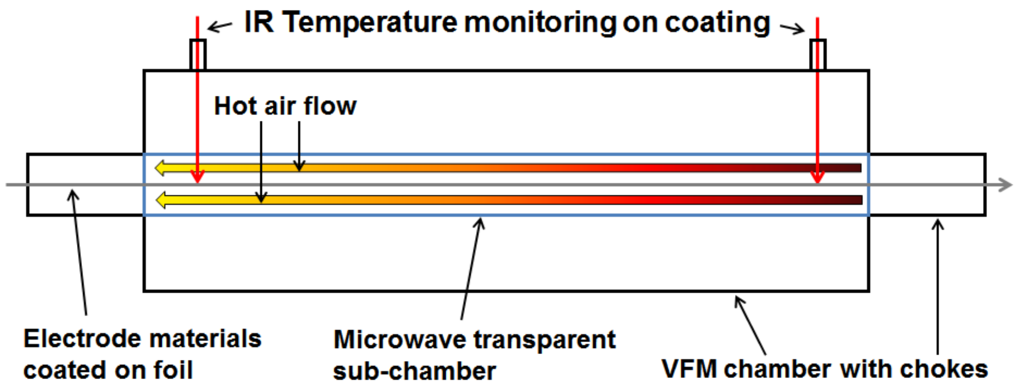


Figure 1: Schematic of the Advanced Drying Process (ADP) apparatus showing the VFM chamber, the microwave transparent sub-chamber, microwave chokes and the hot air flow.

Lambda used mostly in-house components to set up the apparatus shown below.

Figure 2: The digital image shows the tall MC2100 delivering VFM power in the chamber with chokes on both ends.

For drying testing of slurries in the lab prototype set up shown above, both the aqueous anode and NMP cathode, the slurries were mixed according to mixing instruction provided by Navitas personnel. The slurries were manually cast onto the metal foil using a doctor blade with blade height set to provide appropriate loading for either slurry. The aqueous anode slurry was cast on copper foil and the NMP cathode on aluminum foil.

The processing parameters for the samples prepared and provided to Navitas are shown in the following Table.

Table I

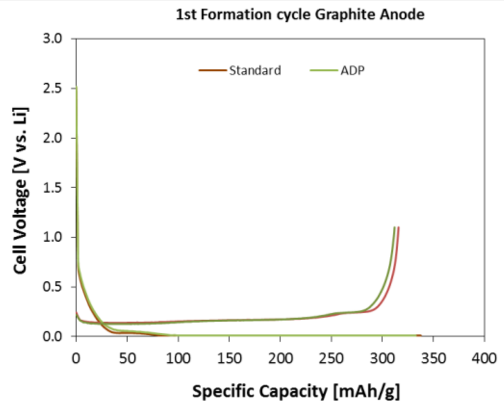
	Drying method	**Solvent**	**Drying time (min)**	**Loading (mg/cm^2)**
Anode	Standard	Water	7.0	10.4
	ADP	Water	1.5	10.6
Cathode	Standard	NMP	7.0	18.2
	ADP	NMP	3.5	18.9

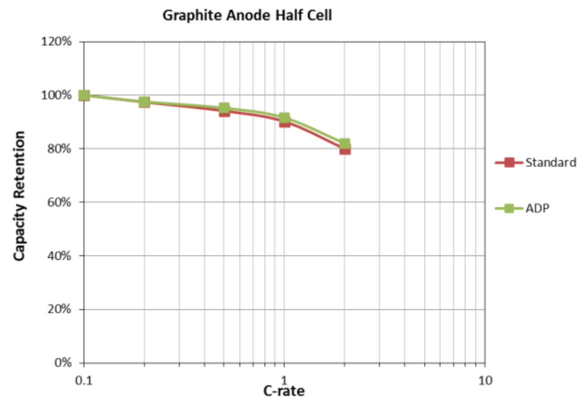
<u>Characterization</u>

The Electrode testing performed at Navitas includes solvent content, adhesion and binder distribution. The Electrochemical validation was performed using half coin cell testing.

- All the electrodes passed both wet and dry adhesion tests.
- Binder distribution ratio (electrode surface to near foil substrate) observed values were < 1.3 the target value.
- Electrodes fabricated by Lambda whether dried by hot air (standard) or by the advanced drying process (ADP) passed Navitas standard electrode qualifications.

Half Cell Testing: Anode



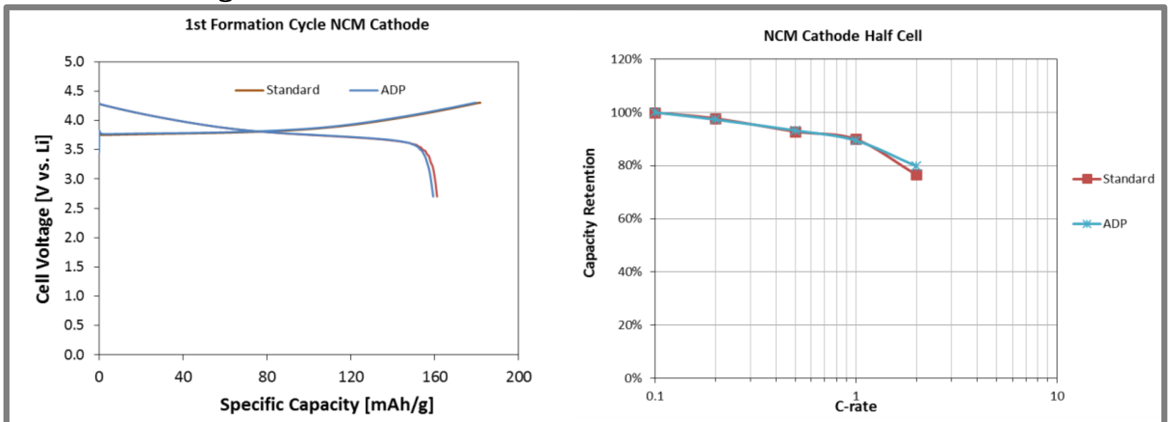


Electrode	Drying Method	Drying time (min)	Loading		Reversible Capacity (mAh/g)	ICL (%)
			(mg/cm^2)	(mAh/cm^2)		
Anode-water	Standard	7.0	10.4	3.4	**325.6**	**7**
	ADP	1.5	10.7	3.5	**322.9**	**7**

Figure 3: Half Cell testing showing Cell Voltage and Capacity Retention for Anode prepared using Standard and ADP method.

- Standard drying was used as baseline to compare electrochemical performance of microwave dried electrodes (anodes and cathodes).
- Anodes dried with standard method and ADP show no difference in performance.

Half Cell Testing: Cathode



Electrode	Drying Method	Drying time (min)	Loading		Reversible Capacity (mAh/g)	ICL (%)
			(mg/cm^2)	(mAh/cm^2)		
Cathode-NMP	Standard	7.0	18.2	2.9	**161.3**	**12**
	ADP	3.5	18.9	3.0	**159.4**	**11**

Figure 4: Half Cell testing showing Cell Voltage and Capacity Retention for Cathode prepared using Standard and ADP method.

- Standard drying was used as baseline to compare electrochemical performance of microwave dried electrodes (anodes and cathodes).
- Cathodes (NMP based binder) dried with standard method and ADP show no difference in performance.

Full single layer pouch (SLP) cells were fabricated at Navitas facilities and cycle life testing was performed.

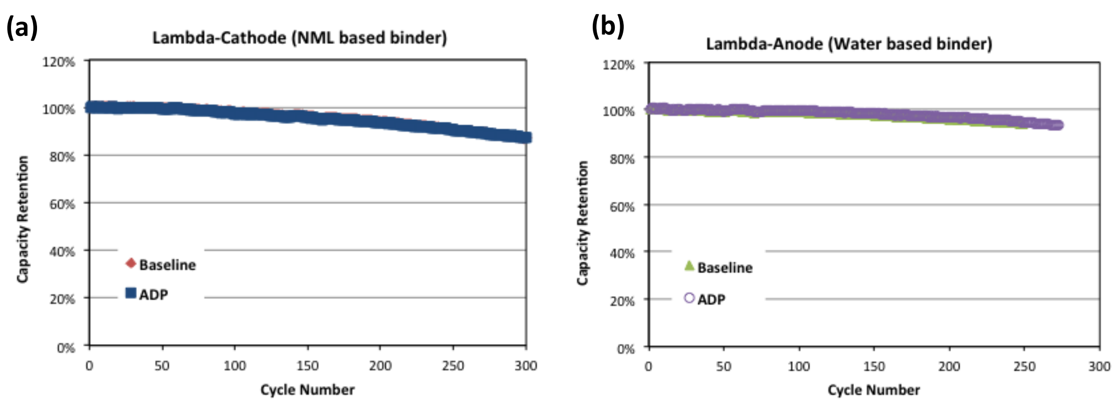


Figure 5: Cycle life testing for single layer pouch cells (a) cathode (NMP based binder) dried using ADP and Navitas standard graphite anode (NMP based binder); (b) Navitas cathode (NMP based binder) matched with ADP dried anode (water based binder). In all cases the standard cells were dried with conventional drying methods.

The data above demonstrated that samples produced even with a preliminary experimental setup, where VFM power is used together with a rudimentary hot air flow (ADP), the processing time can be substantially reduced especially for the aqueous (~5X drying speed) electrodes.

Microwave Choke Prototype Testing

The microwave chokes need to be designed to allow the slurry coated 175 mm wide metal foils to pass through the ADP/VFM processing chamber without leaking microwave energy into the manufacturing area. The international standard for microwave leakage to be considered safe for human exposure is $5mw/cm^2$.

To minimize the overall length of the system, the choke design integrated the hot air delivery and recovery manifold such that it also acts as a microwave choke. These components were designed, fabricated, assembled and tested for microwave leakage with full power of 1600W delivered into the chamber.

Preliminary microwave leakage with metal foil passing through $< 1mW/cm^2$

ADP/VFM System Design Review

A gating decision in designing the final system was to define the length of internal heating space required to allow comparison of ADP/VFM drying to convection oven drying. Using a 1 minute resident time for the aqueous slurry coated foil to be in the ADP chamber, the length of the ADP chamber was defined by the slurry casting speed. The casting speed of 0.5 meter per minute requires drying the slurries in more than 8 feet long convection/IR ovens. In contrast, with the above casting speed the ADP chamber needs to be only 0.5 meter (~ 20inches), plus hot air manifold and choke on each end giving a total length of ~ 40" or ~ 100cm.

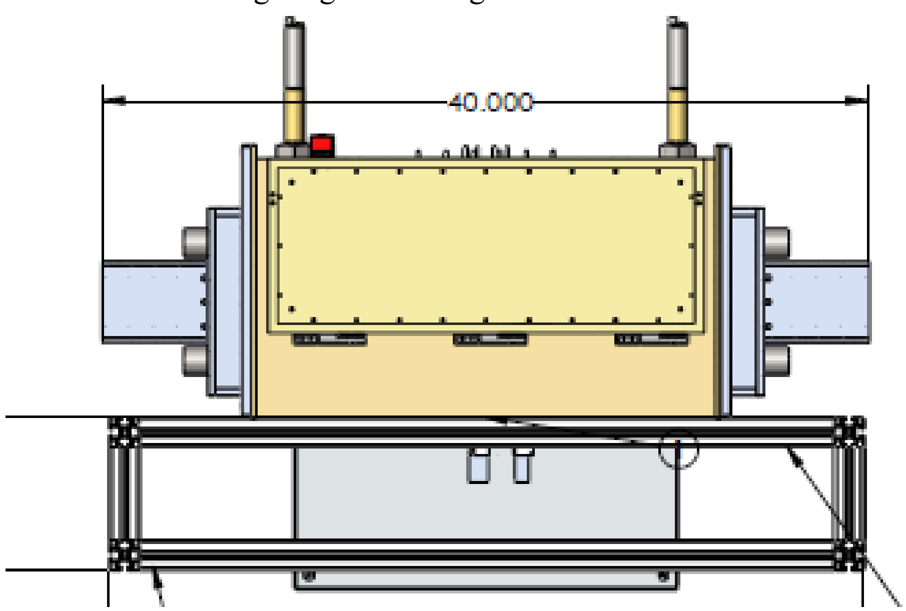


Figure 6: ADP/VFM Processing Chamber

Build of ADP/VFM Prototype System

Custom designed and fabricated components were received and the ADP prototype system was assembled. The following digital image shows the complete ADP system.

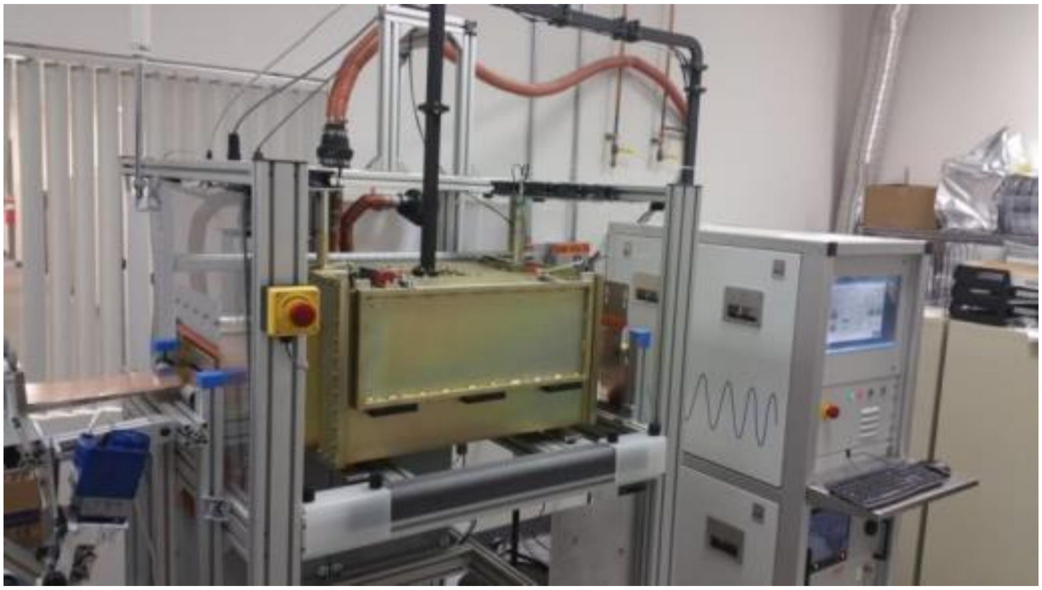


Figure 7: Complete ADP/VFM Processing System

Go/No Go Demonstration

A temporary web handling motor was installed on the system along with a pump to deliver slurry from a reservoir to the doctor blade casting slurry on the moving as shown below.

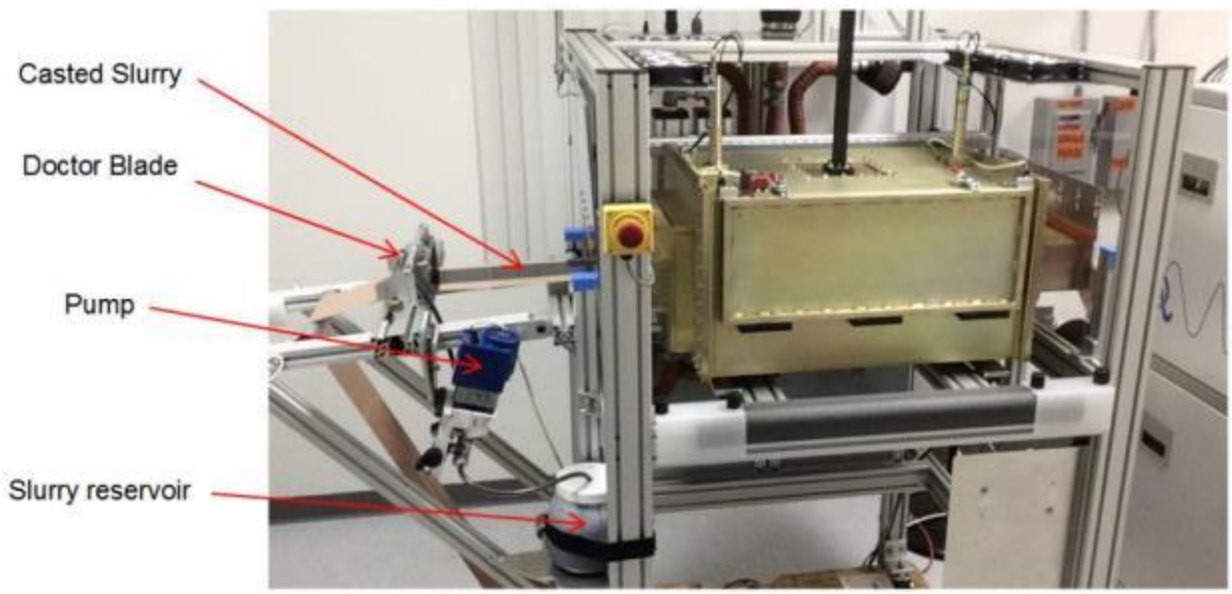


Figure 8 (a): Slurry casted on moving foil enters ADP chamber.

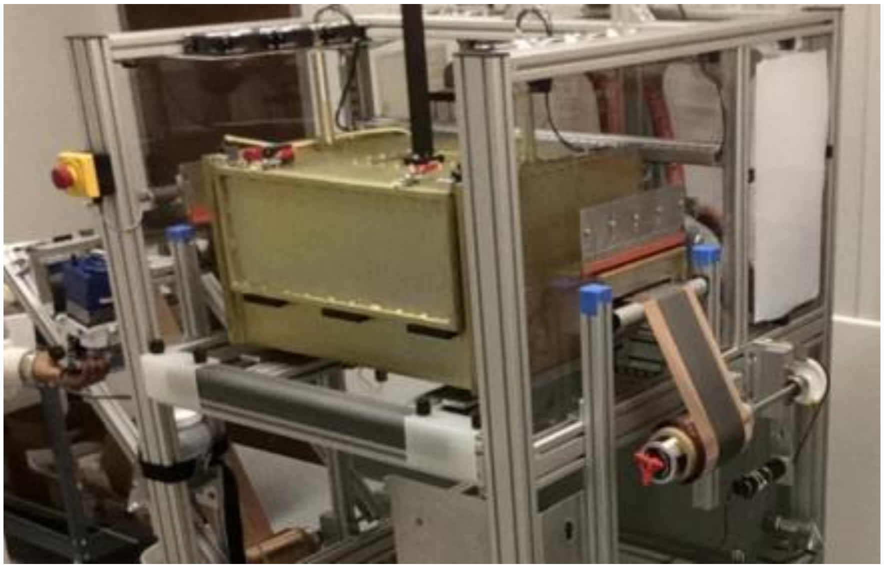
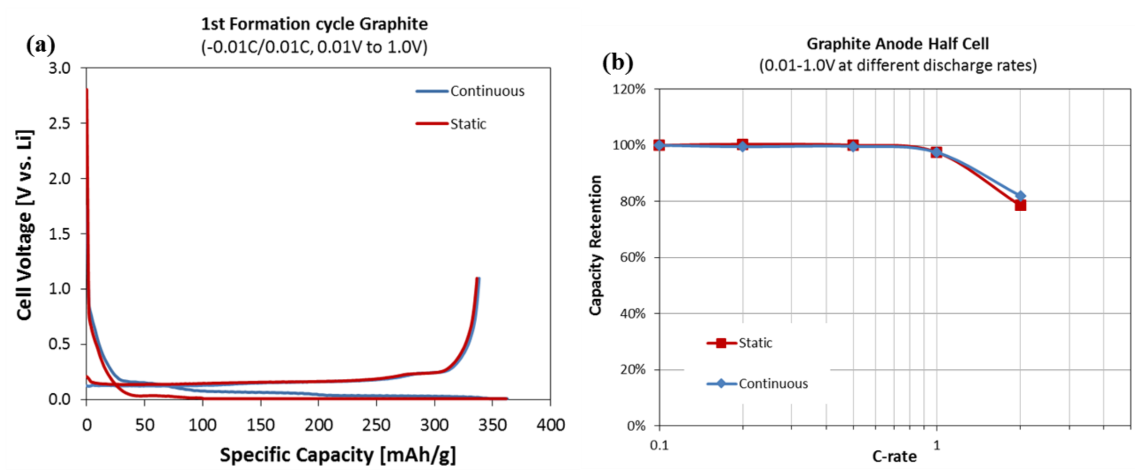


Figure 8 (b): Electrode continuously casted exits ADP process chamber completely dry.

<u>Characterization of Continuously Cast Electrode</u>

The analytic work was performed by Navitas on the continuous ADP drying during the Go/No Go demonstration and compared to the static drying performed earlier.

The anode formation half-cell testing data comparison is shown below in Figure 9.



Electrode	Drying conditions	Loading		Reversible Capacity (mAh/g)	ICL (%)
		(mg/cm^2)	(mAh/cm^2)		
Anode- water based binder	Static-batch	10.1	3.7	**343.2**	**7**
	Continuous	10.2	3.8	**345.1**	**7**

Figure 9: Half-cell electrochemical performance results for anode electrodes dried under static and continuous ADP (a) first formation at C/20 and (b) rate capability and half-cell formation parameters. Specific capacities and initial capacity loss (ICL) are similar for both sets of cells.

Additionally, life cycle data for single layer pouch (SLP) cell (3.0 mAh/cm^2, @ C/2,100% DoD) made with electrodes dried under static ADP is shown in Figure 10. The plots show comparable performance between the baseline and the cell fabricated with ADP dried electrodes.

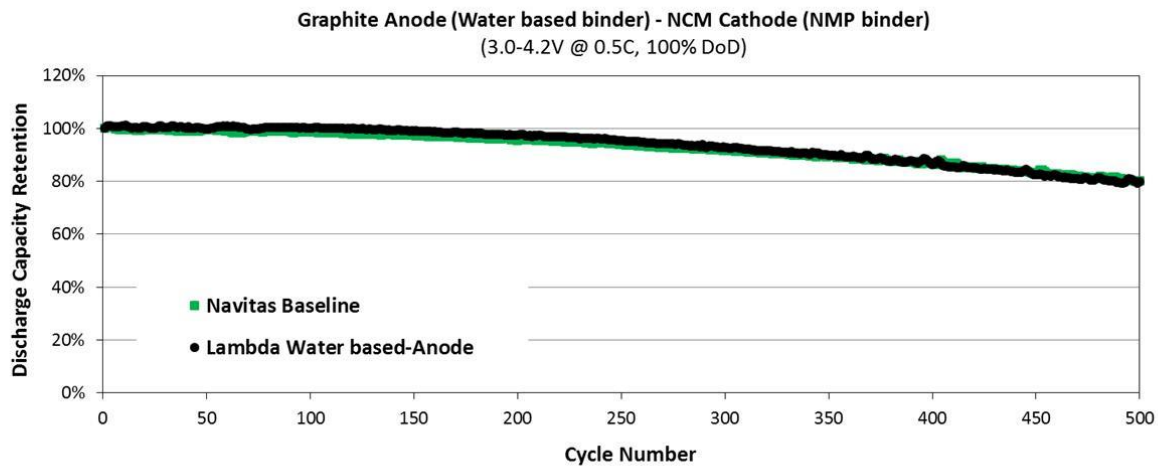


Figure 10: Life cycle experiments for SLPs. Discharge capacity retention for static ADP dried cells (performed earlier this year) in comparison to Navitas Baseline.

Figure 10 shows cycle life results for SLP with electrodes dried using static ADP. Since the continuous ADP drying results were equivalent to the static dried cells, it was concluded that the continuous ADP dried electrodes will behave identically in life cycle experiments.

Installation of ADP/VFM Prototype System at Navitas

On successful completion of the Go/No-Go demo, the temporary web handling setup, the doctor blade and pump were all dismounted and the ADP system and was packed and shipped to Navitas. Lambda personnel visited Navitas (Sep. 22 – 25, 2015) for installation of the ADP Prototype and training for appropriate use of the ADP tool. Drying tests of aqueous anode and NMP cathode slurries cast onto moving foil were performed during the installation visit.

Figure 11 (a) shows the Power Module placed behind the Process Module. VFM power is transmitted and delivered by the waveguide on the top connecting the two modules.

Figure11 (b) shows a clear view of the Process Module beside the convection oven.

Drying Comparison

Anode and cathode films have been routinely coated and dried using the ADP system and Navitas IR/convection drying line (baseline). The anode active materials include commercial synthetic graphite (97wt. %) combined with water based binder (CMC + SBR). The cathode slurries include NCM523 (93wt. %), conductive carbon and PVDF binder (NMP solvent). For the above mentioned anode, the coating and drying speed used was 500 mm/min for both conventional and ADP drying. For NCM cathode the coating/drying speeds were 350 and 225 mm/min for Navitas baseline and ADP system respectively. In all cases the ADP oven is a fraction of the POR oven size. Table 2 summarizes the advantage of using ADP over conventional the drying system. In the case of water based anodes there is a 5X drying advantage over IR convection drying; while for NMP based cathodes the reduction in drying time has a 3X benefit. Continuous coating at these conditions was employed to fabricate electrodes for lithium ion high energy cells (HEC) for the project deliverables.

Table 2. Comparison between conventional drying system and the advance drying process (ADP) system.

Electrode	Drying Method	Loading (mg/cm^2)	Drying speed (mm/min)	Drying length (m)
Anode (graphite, water based binder)	Standard (2 Zone Convection System)	10.4	500	2.5
	Advanced drying (VFM + hot air)	10.6	500	0.5
	ADP Advantage	**5X**		
Cathode (NCM523, NMP based binder)	Standard (2 Zone Convection System)	18.2	400	2.5
	Advanced drying (VFM + hot air)	18.9	225	0.5
	ADP Advantage	**3X**		

<u>High Energy (HEC) Prismatic Lithium Ion Testing</u>

Electrochemical performance tests (formation, rate capability, cycle life) were carried out earlier on 2.0Ah HE cells. Figure 12 shows cathode, anode, and final prismatic cell.

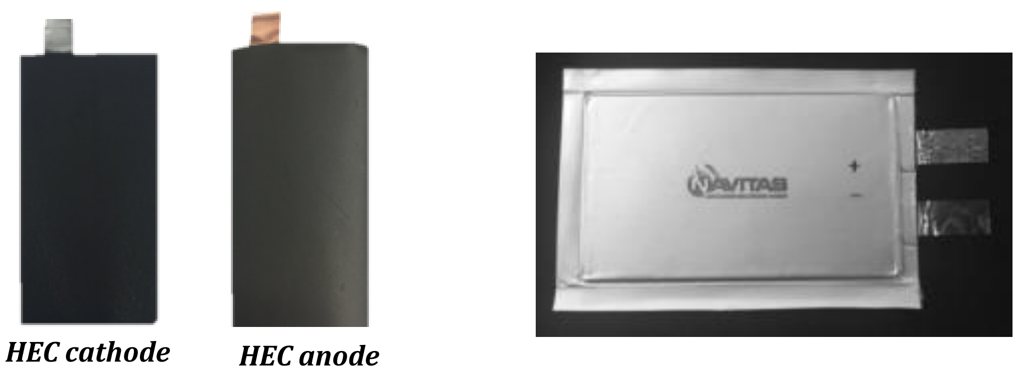


Figure 12. Fabricated electrodes (cathode and anode) as well as the SLP cells dried using Navitas conventional and ADP drying systems.

Adhesion & Binder Migration

Navitas standard adhesion tests (performed using industrial standards, Navitas trade secret) were carried out on sample electrodes (anode and cathode) dried using standard and Advanced Drying Processes. Binder distribution measurements were done using elemental mapping (EDX: Energy Disperse X-ray spectroscopy) on 4-6 sections of the (anode/cathode) film cross-section (from top to bottom). Binder distribution ratio, from electrode surface and near the metal foil substrate, should be under 1.3. Table 3 summarizes adhesion and binder distribution results.

Table 3. Summary of adhesion testing and binder migration data for standard and ADP drying of anode and cathode

Electrode	Drying Method	Binder Solvent	Mass Loading (mg/cm^2)	Adhesion	Binder Distribution
Anode	Standard	Water	10	Pass	1.19
	ADP	Water	10	Pass	1.07
Cathode	Standard	NMP	18	Pass	1.09
	ADP	NMP	18	Pass	1.03

Cathode Binder distribution

Cathode electrodes dried with and without ADP were analyzed under SEM (cross-sectional view) to study the binder distribution (fluorine) across the thickness of the electrode. Figure 13 shows the SEM and EDX micrographs and fluorine mapping (blue shades on EDX pictures).

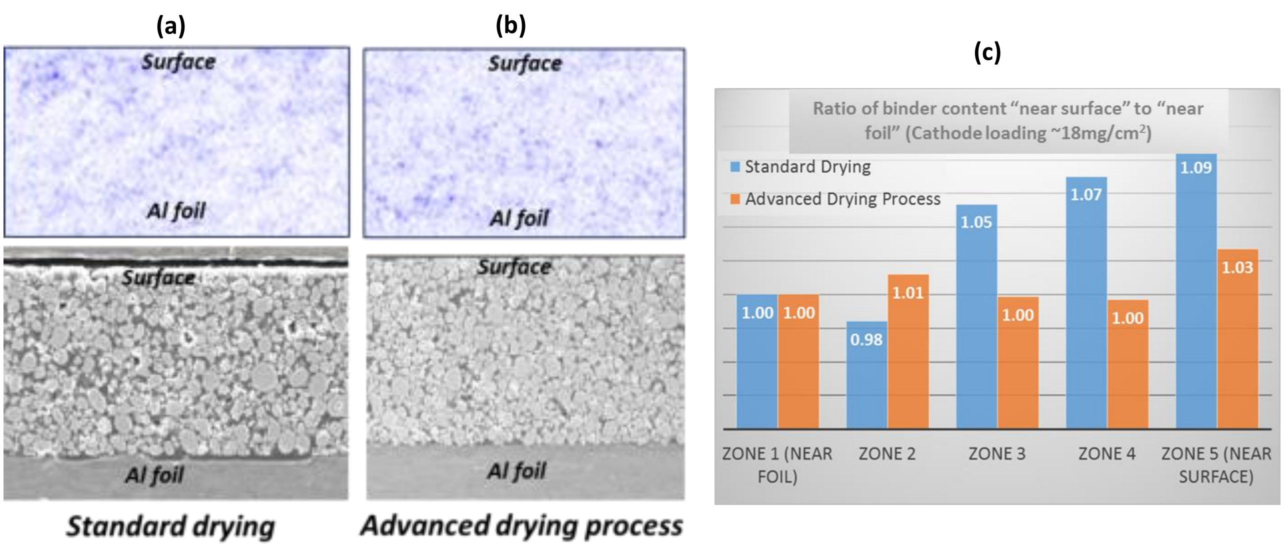


Figure 13. Binder distribution for cathode (NCM523) electrodes dried using (a) standard or baseline drying and (b) ADP, hot air plus variable frequency microwaves (VFM). (c) Binder ratios measured at various zones across the electrode (from near foil to the electrode surface).

The ratio of binder content (weight %) between surface and foil interfaces is 1.10 for the conventional dried electrode. Conversely, the electrode dried under ADP has a ratio of 1.03. The latter confirms earlier observations, indicating less (or no) binder migration to the electrode surface when ADP is used to dry the electrodes.

Anode Binder distribution

Anode electrodes dried with and without ADP were analyzed under SEM (cross-sectional view) to study the binder distribution. Samples were stained with osmium tetroxide prior to SEM studies. Backscattered electron (BSE) microscopy was used to map osmium through the anode cross sections. The metal shows as white bright spots on cross section micrographs. Figure 14 shows the BSE micrographs for both sets of samples. The ratio of binder (Figure14c), content between surface and foil interfaces, were 1.2 and 1.1 for conventional and ADP dry electrodes.

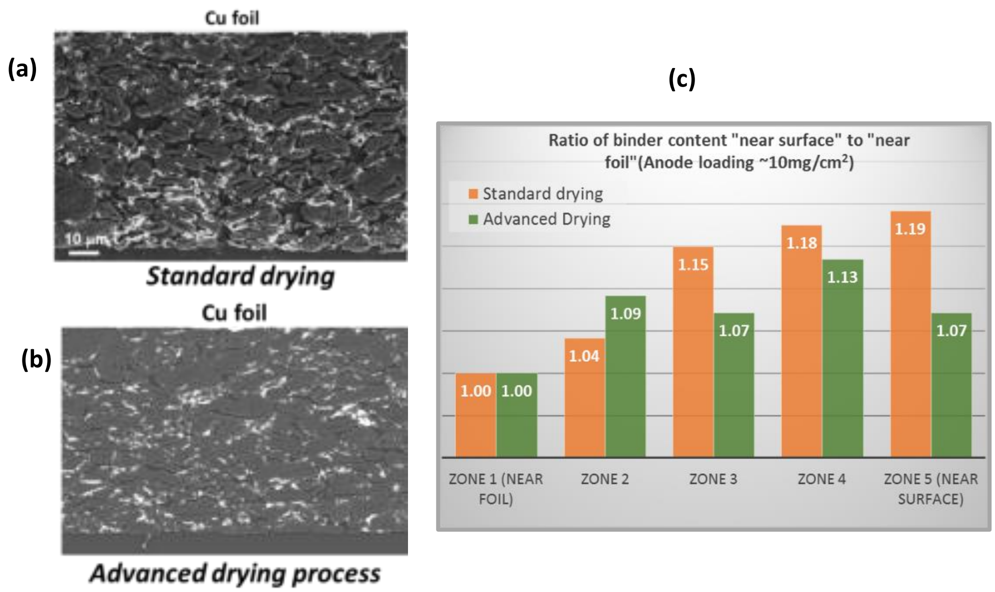


Figure 14. BSE micrographs showing binder distribution for anode (graphite) electrodes dried using (a) standard and (b) ADP). (c) Binder ratios measured at various zones across the electrode (from near foil to the electrode surface).

Electrochemical performance

Fifteen (6 baselines, and 9 with ADP) 2.2Ah cells were also fabricated. Formation cycles were taken in all cells at C/10 from 3.0 to 4.2V, showing average 2.2 Ah reversible capacity and 16% ICL for all cells. The cathode electrochemical loadings were set to 3.0mAh/cm^2. Table 4 summarizes numerical values for specific capacities and initial capacity loss (ICL). It is worth emphasizing that no difference has been observed during formation of all cells.

Table 4. Summary of 2.2 Ah prismatic lithium ion cells formation parameters

Drying method	Formation Capacities (Ah)		ICL (%)
	First Charge	Reversible	
Standard drying	2.61	2.19	16%
Advanced drying process	2.61	2.19	16%

Life cycle experiments for the prismatic cells (both standard and ADP dried electrodes) were carried out at C/3 current rate from 3.5 to 4.2V, at this voltage window the cell discharges to 80% (or 80% depth of discharge, DOD). Cycle numbers versus discharge capacity retention for both methods are shown in Figure 15. Both set of cells (with baseline and ADP dried electrodes) have completed 1000 cycles, with capacity retention of 80%. The plots show comparable performance between the baseline and the cell fabricated with ADP dried electrodes. Average numerical values are summarized in Table 5.

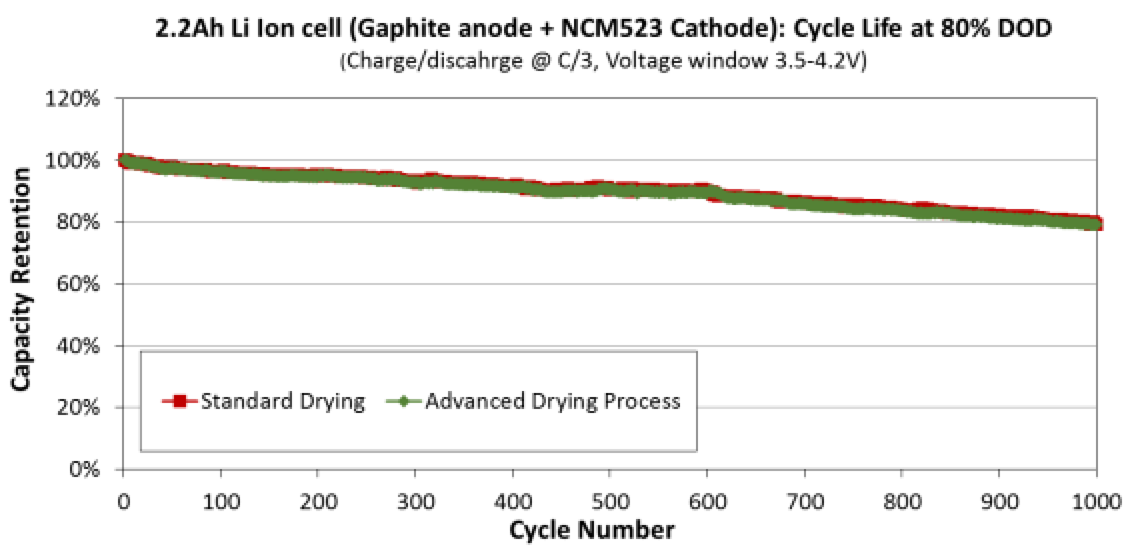


Figure 15. Cycle life testing for HEC prismatic cells comparing standard and ADP drying methods. Average values are presented in this plot. Experiments were carried out at C/3 current from 3.5 to 4.2V.

Table 5. Summary of 2.2 Ah prismatic lithium ion cells formation parameters. Standard drying refers to the Navitas IR convection system, and the advanced drying process (ADP) combines lower temperature hot air flow plus VFM.

Drying method	Capacities (Ah)		Capacity retention at 1000th cycle
	Reversible @ 0.33C, 100% DOD	1st cycle @ 0.33C, 80% DOD	
Standard drying	2.0	1.6	80%
Advanced drying process	2.0	1.6	80%

Additional Experiments

Advanced Drying Process System as a Booster to Conventional Drying Systems

The current ADP system was designed to be ~ 50cm in length and the slurry drying speeds were limited to 0.5m/min for graphite/water based binder and to 0.25m/min for NCM cathode/PVDF (Please refer to Table 2 above).

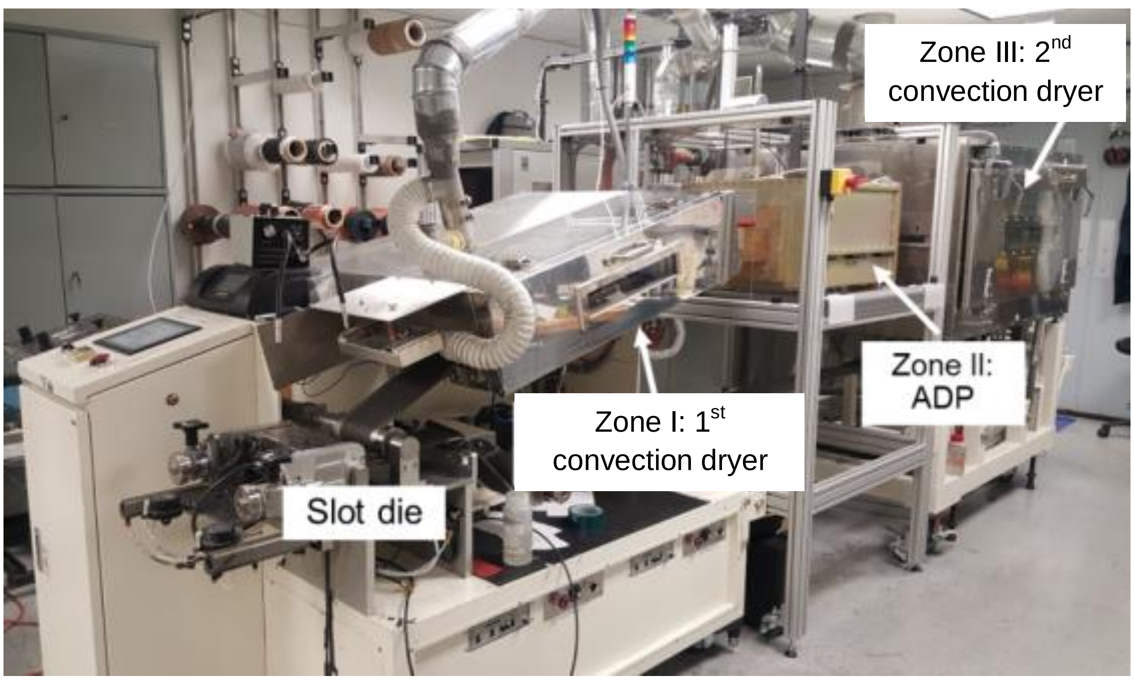


Figure 16. Navitas coating system with 3 drying zones. Zone I is 1st convection dryer, Zone II is ADP system & Zone III is the 2nd convection dryer.

However, since most of the bulk drying can be rapidly performed with ADP, the 0.5m ADP system was explored as a booster module to the POR convection drying system. After the bulk drying with ADP the surface drying can be completed with regular convection drying. Figure 16 shows the configuration of Navitas coating and drying system which includes the ADP chamber.

Zone I is 1.0m convection dryer while Zone II is the 0.5m ADP chamber and finally Zone III is the second 1.5m convection dryer.

NCM 523 cathode slurry with 55% solid content and NMP solvent was used to perform these experiments. As shown in Table 1 above the usual speed has been 225mm/min for this slurry. The combination of zone II (0.5m ADP as booster module) and III (1.5m convection) were used to dry the slurries. Films were coated with 3.0mAh/cm^2 electrochemical loading. Cathode films were coated and dried at 0.8m/min with this drying configuration; no defects were observed on the dried films. The power of zone III was reduced (which reduces the air temperature) when ADP systems was turned on.

To further test the advantage of using ADP in this configuration, even films with increased mass loading as high as 4.0mAh/cm^2 were also tested. Without VFM or ADP surface cracks were observed on these thicker coatings. However, when the VFM (in the ADP) was turned on, the films were completely dried without any surface defects. The power supplied to zone III was decreased by 20% when VFM was on.

Furthermore, the cathode slurry was coated and dried at 1.0m/min. Without ADP there were surface cracks and wet spots (Figure 4). The combination of ADP and convection drying made it possible to completely dry the films without any defects.

Convection Drying | VFM boosted Convection drying

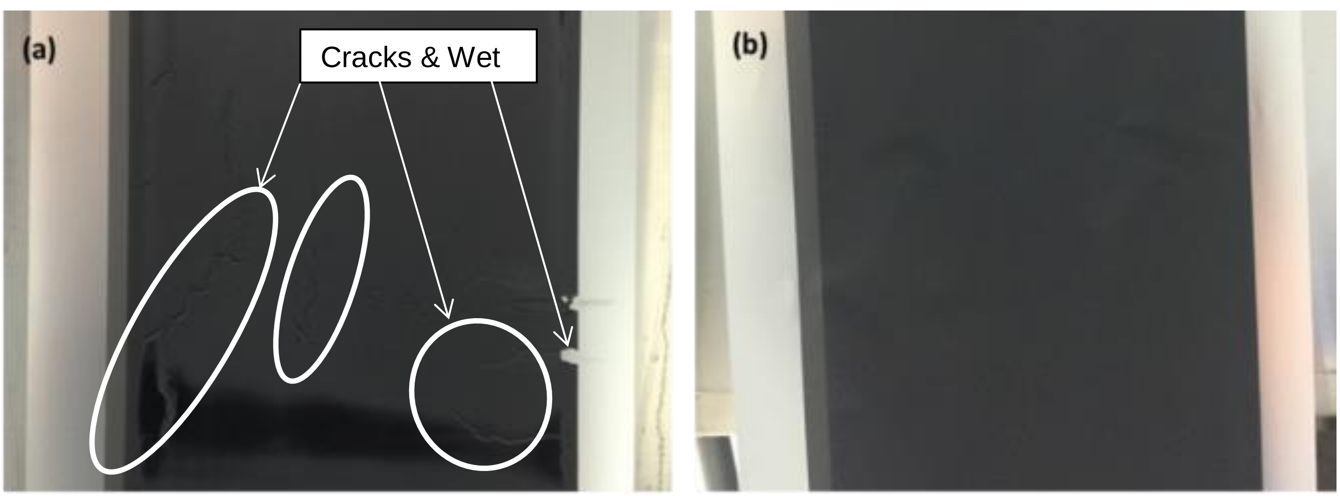


Figure 17. NCM cathode (24.5mg/cm^2) films dried using ADP as booster. Coating and drying speed was 1.0m/min (a) Without VFM, surface cracks and wet spots are observed (b) with VFM, film completely dried without defects.

Binder distribution measurements were performed using five zone elemental mapping with EDX: Energy Disperse X-ray spectroscopy on the electrode film cross-section. Binder distribution ratio, from electrode surface and near the metal foil substrate, was calculated based on the fluorine concentration. Binder ratios measured at various zones across the (NCM523) electrode (from near foil to the electrode surface) dried using combination of ADP as booster.

Table 6 summarizes binder distribution results. The binder distribution ratio was calculated by comparing fluorine (binder) content of the "surface" zone to that of the "foil interface" zone. A standard cathode (18 mg/cm^2) was dried at conventional drying method at 400 mm/min speed. The ratio was 1.12 (i.e., surface zone binder content is 12% higher than the foil interface zone). When the electrode was coated at higher loading (25mg/cm^2) and at faster speed (800 mm/min), the hot air dried sample showed a ratio of 1.87. The severe binder migration is due to aggressive drying conditions with forced hot air only. However, the electrode dried under ADP boost showed a ratio of 1.10, indicating a uniform binder distribution. Additionally, VFM enables even faster (1000 mm/min) coating with lower binder migration than conventional drying method. The above experiments have demonstrated the benefit of using the ADP system to boost drying process. The benefit of low binder migration is still obtained along with faster drying speeds.

Table 6. Summary of binder migration data gathered using the ADP (hot air + 95%VFM) system as a booster to dry NCM cathode

Loading (mg/cm^2)	Description		Coat/dry Speed (mm/min)	Binder migration (%)
	Drying method	Zone-III IR Temp		
18	Standard (Hot Air)	115°C	400	1.12
25	Standard (Hot Air)	125°C	800	1.87
25	ADP	105°C	800	1.10
25	ADP	110°C	1000	1.52

Energy Savings Evaluation

The power consumption measurements were performed to evaluate one of the objectives of the project on energy and cost savings. The data collected for the ADP process is compared with the IR drying oven process of record (POR). The POR oven is a Megtec Systems Flotation dryer, which uses 8 IR heaters in combination with convective airflow for the drying process. In addition, the POR process uses a separate IR preheat module.

For POR the power consumed by the IR preheat module as well as for Megtec Floatation dryer was measured.

(a) (b)

Figure 18. Images of control screens for IR preheat module (a) and 8 IR heaters on Megtec Flotation dryer (b).

The average power consumed during a 500mm/min drying run is shown in Figure 19 below.

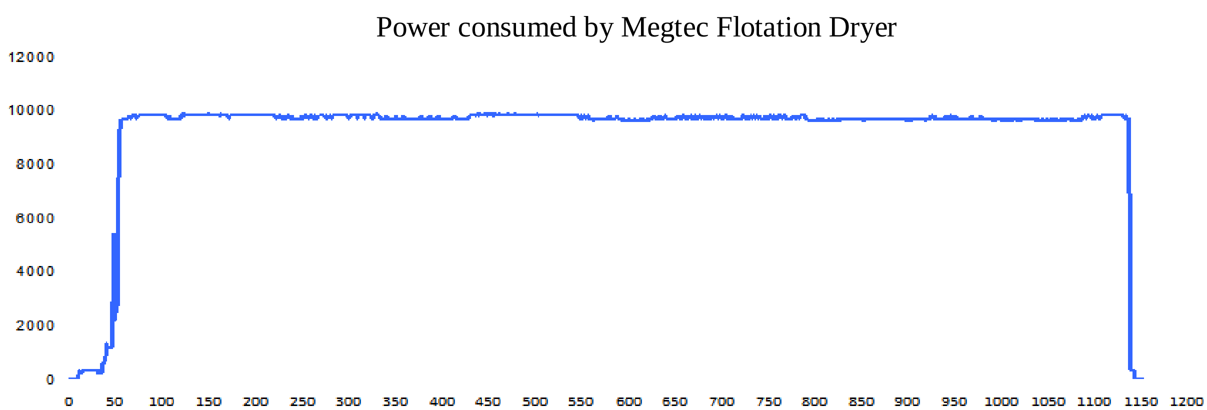


Figure 19. Average power consumed during a drying run = 9.727 kVA

Two meters were used to monitor power consumption for the ADP, one for power module and another for the hot air provided in the process module.

Figure 20 below shows the average power measurement for the ADP power and process module (hot air).

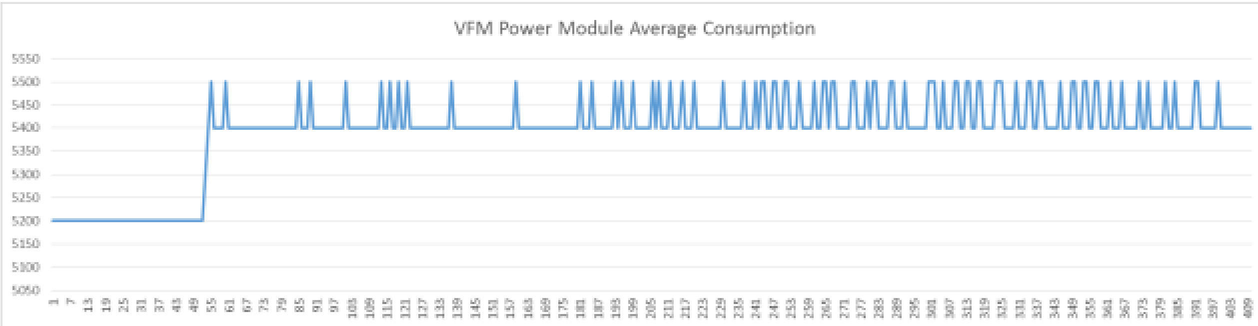


Figure 20(a). Average power consumed by the VFM power module during a drying a 500m/min drying run = 5.422 kVA

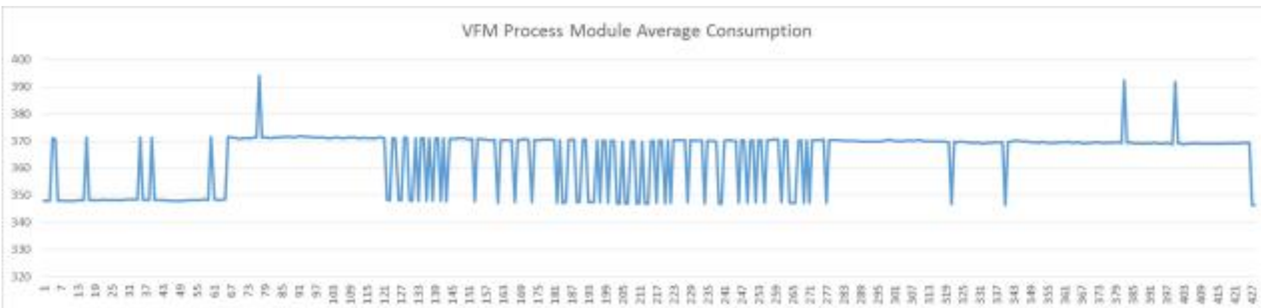


Figure 20(b). Average power consumed by the hot air in process module during a drying a 500m/min drying run = 367 VA

Total power consumed by the ADP dryer is 5.422 + 0.367 = 5.789kVA

ADP drying efficiency = 5.789/9.727 = 0.595 or 59.5% of average power used by POR ovens.

This evaluation demonstrates a > 40% saving in power consumption with ADP as compared to the convection/IR drying systems. Thus one can expect the same saving in the operational cost with ADP. Please note that the objective of the project was a 30-50% reduction in energy and cost.

__Thick Higher Loading Electrodes__

The following data is the most remarkable performance of penetrating VFM energy.

__Cathode with 4% binder and loading up to 50 mg/cm^2 (or 7.7 mAh/cm^2)__

The cathodes were coated and dried using the ADP system and hot air only (baseline). The slurry consisted NCM523 (93wt. %), conductive carbon and PVDF binder. The loadings ranged from 30 to 50 mg/cm^2.

Adhesion

The baseline coating starts to show surface cracks when the loading is higher than 35 mg/cm^2. However, the ADP dried coating shows no cracks with the loading even at 50 mg/cm^2.

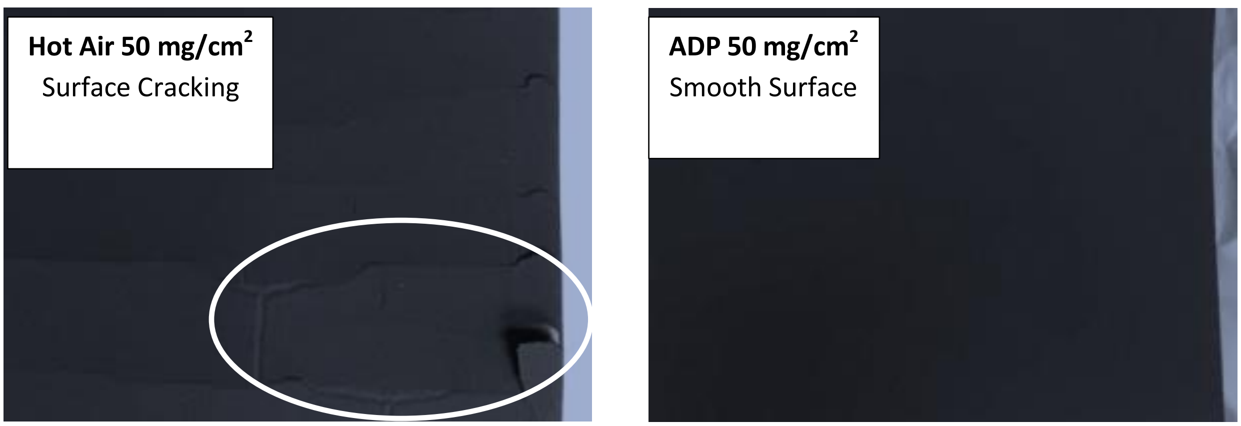


Figure 21. The surfaces of as-dried cathodes (without calendering). Hot air dried baseline coating shows surface cracks while the ADP dried coating shows no cracks.

Electrode Resistance (Conductivity)

The electrode through-plane resistance was measured by a two-probe conductivity meter. As shown in Figure 22, the ADP and baseline electrodes show the same resistance at loading of 30 mg/cm^2. However, the ADP dried electrode shows much lower resistance than the baseline at loading of $\geq$ 35 mg/cm^2. Furthermore, at loading of 30 mg/cm^2 as the applied pressure increases, there is a significant decrease in the resistance for both the baseline and ADP dried electrodes. However, at higher loading of 50 mg/cm^2, firstly the resistance is much lower than the baseline and secondly the resistance for ADP dried electrode does not decrease any further after 20psi. In contrast the baseline resistance is influenced by the pressure all the way till 70psi. This indicates that the ADP electrodes present better conductive bridging microstructure and/or lower coating-to-foil interface resistance at ultra-high loadings. This is believed to be a result of the internal heating with VFM.

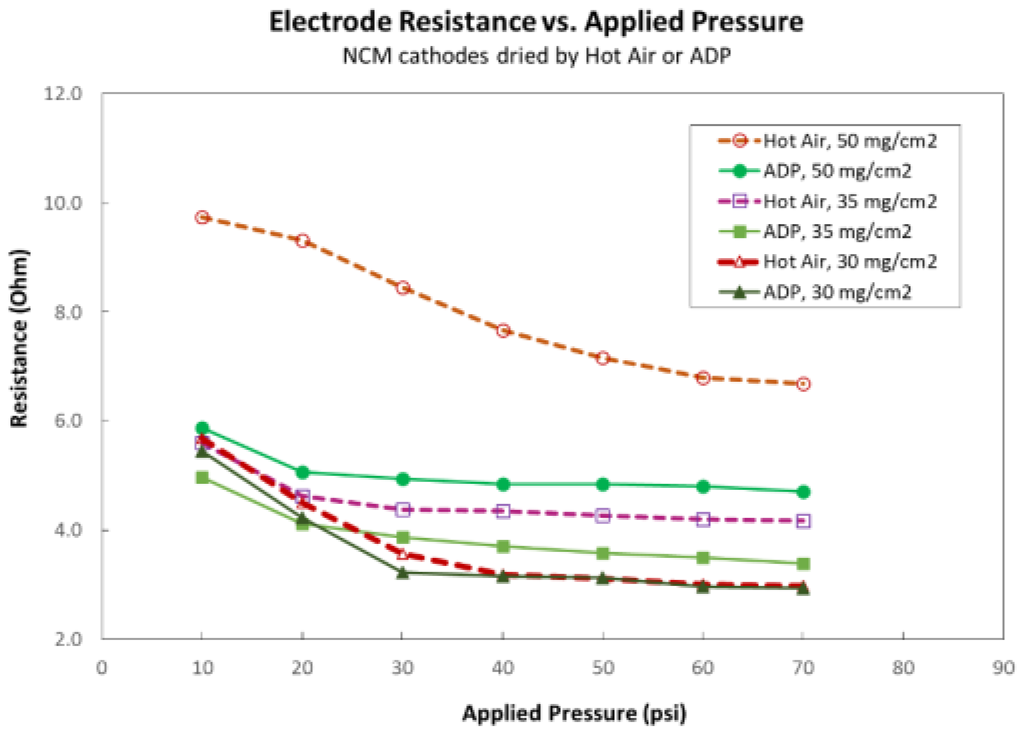


Figure 22. Comparison of through plane resistance of NCM cathodes dried by ADP and baseline hot air methods.

The improved microstructure was observed and presented earlier but is being reproduced here.

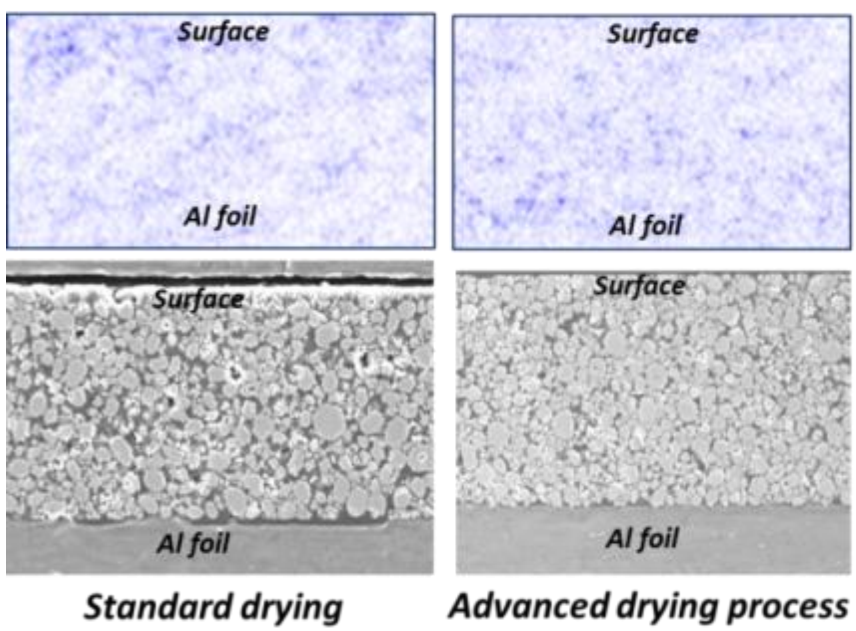


Figure 23. Comparison of microstructure of NCM cathodes dried with standard baseline hot air method and with Advanced Drying Process (ADP) using VFM.

It is apparent from Figure 23 that the active particles in the ADP dried electrodes are much more densely packed and will result in lower resistance as compared to the standard drying method. Even the electrode coating to Aluminum interface shows more contact and hence lower resistance. These improvements in conductivity (lower resistance) were anticipated but not claimed until the real resistance measurements (shown in Figure 22) were made.

Binder distribution for high loading electrodes

The cathode cross-section samples have been monitored by SEM/EDX. Fluorine mapping was performed to compare PVDF binder contents at different zones. For the electrodes at loadings of 50 mg/cm^2, the ADP dried cathode shows lower level of binder migration than the hot air dried baseline cathode (Figure 24).

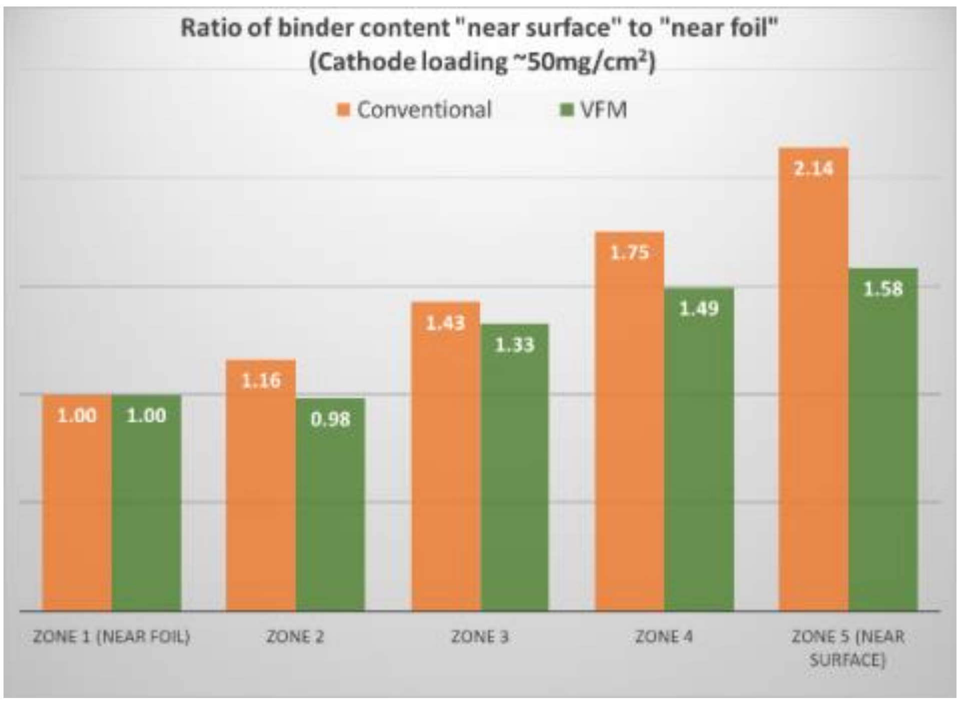


Figure 24. Binder contents at different zones of the cathodes. The cathode cross-section is divided to 5 zones from “near foil” to “near surface”. The binder contents have been normalized (based on the content in Zone 1 “near foil”).

Capacity and Rate Capability

The capacities for ADP and baseline cathodes and ICLs are shown in Table 7.

Table 7. Capacity and ICL of cathodes dried with ADP or hot air only.

Loading (mg/cm^2)	Drying condition	Specific Capacity (mAh/g), C/10		ICL (%)
		FCC	Reversible	
30	Hot air only	183.1	159.3	13.0
	ADP	182.7	157.8	13.5
35	Hot air only	183.2	157.9	13.8
	ADP	183.5	158.5	13.7
50	Hot air only	179.0	151.4	15.4
	ADP	183.2	157.7	13.9

Although the capacities for the two methods are similar at 30 & 35 mg/cm^2, but 50 mg/cm^2 ADP shows higher specific capacities as well as lower ICL.

The baseline and ADP cathodes with different loadings were tested for rate capability in half cells. The cells were charged at 0.2C (tapering to 0.05C) and then discharged at different C rates up to 1C from 2.7 to 4.2V. The discharge capacity retention is shown in Figure 25.

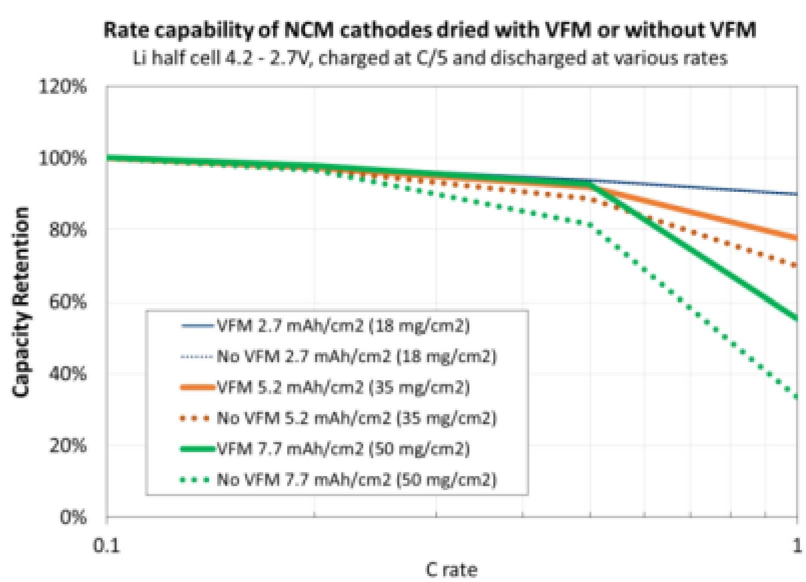


Figure 25. Rate capability of the cathodes with various loadings in half cells. The cells are charged at 0.2C to 4.2V (tapering to 0.05C) and discharged at 0.1, 0.2, 0.5, and 1.0C.

Key points to note:

- At loading of 18 mg/cm^2, the baseline cathode shows the same performance as the ADP dried cathode
- At loading of 35 mg/cm^2, ADP cathode shows slightly better rate capability than the baseline.
- At loading of 50 mg/cm^2, ADP cathode shows much better rate capability than the baseline. The ADP electrode at 50mg/cm^2 loading even shows the same rate capability as a conventional 18mg/cm^2 electrode at C/2 rate.

Graphite Anode with 4% PVDF binder and loading up to 20 mg/cm^2 (or 6.5 mAh/cm^2)

The graphite anodes with PVDF binder were coated and dried using the ADP system and hot air only (baseline). The slurry consisted a natural graphite (95wt. %), conductive carbon and PVDF binder. The loading is up to 20 mg/cm^2.

Adhesion

The baseline dried coating shows multiple surface cracks at the loading of 20 mg/cm^2. In contrast, the ADP dried electrode coating shows much lower level of cracks at the same loading. Pictures of the as-dried anodes are shown in Figure 26.

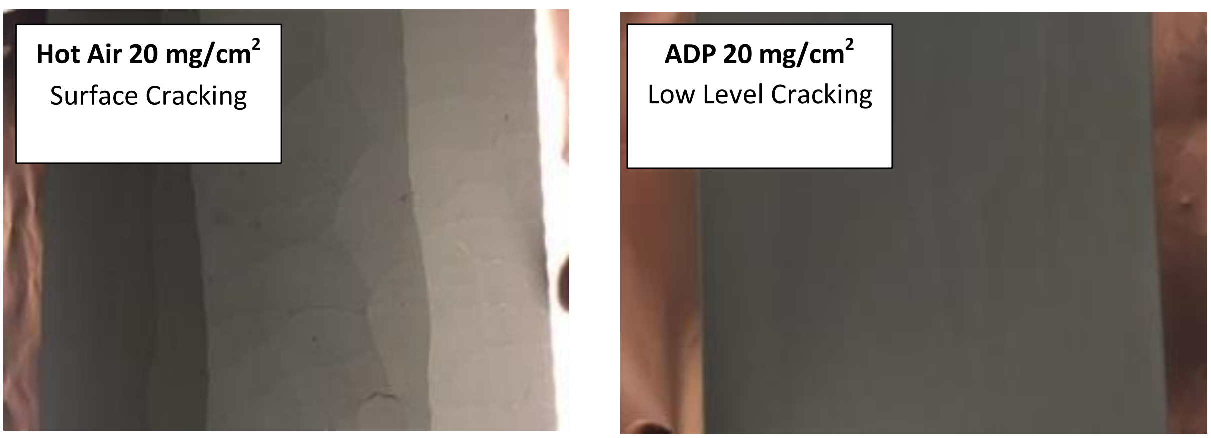


Figure 26. The surfaces of as-dried graphite anodes (without calendering). Hot air dried baseline coating shows deep surface cracks and the ADP dried coating shows lower level of cracks. Due to limited time no attempts were made to optimize the process to eliminate these cracks.

Binder Distribution

The anode cross-section samples have been monitored by SEM/EDX. Fluorine mapping was performed to compare PVDF binder contents at different zones. For the electrodes at loadings of 20 mg/cm^2, the ADP (VFM) dried anode shows lower level of binder migration than the hot air dried baseline (Figure 27).

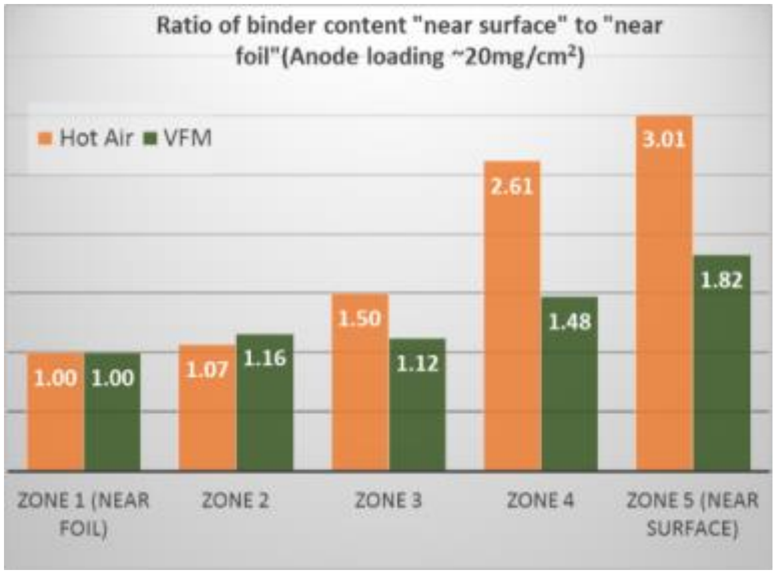


Figure 27. Binder contents at different zones of the electrodes. The electrode cross-section is divided to 5 zones from "near foil" to "near surface". The binder content is normalized (based on the content in Zone 1 "near foil").

Rate Capability

The baseline and ADP anodes at 20 mg/cm^2 were tested for rate capability in half cells (Figure 28). The cells were lithiated at 0.2C (tapering to 0.05C) and then delithiated at different C rates up to 1C from 1.2V to 0.01V. The ADP anode shows better performance than the baseline at 1C.

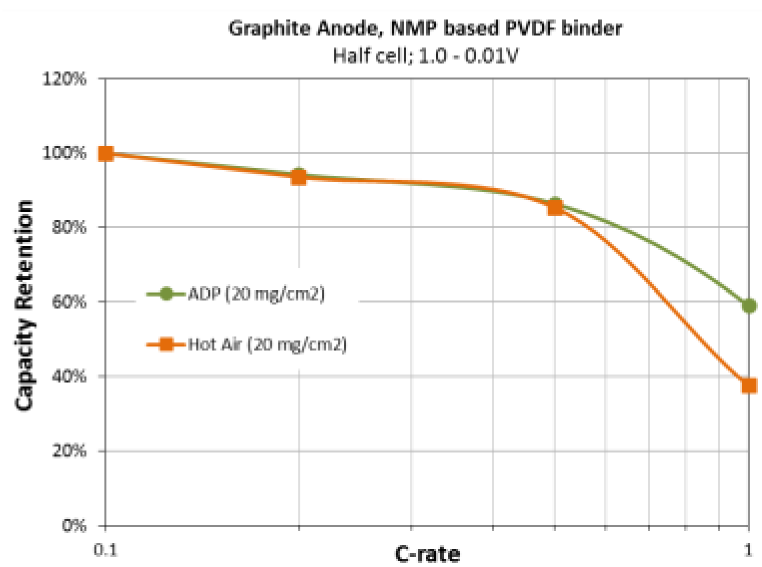


Figure 28. Delithiation rate capability of the PVDF anodes at 20 mg/cm2 in half cells. The cells are lithiated at 0.2C to 0.01V (tapering to 0.05C) and delithiated at various rates to 1.0V.

Graphite Anode with aqueous binder and loading up to 20 mg/cm^2 (or 6.5 mAh/cm^2)

The graphite anodes with aqueous binder were coated and dried using the ADP system and hot air only (baseline). The slurry consisted of natural graphite and CMC/SBR binder. The loading was up to 20 mg/cm^2.

Adhesion

The baseline coating shows surface cracking / delamination at the loading of 20 mg/cm^2. Meanwhile the ADP dried coating shows much lower level of cracks at the same loading. Pictures of the as-dried anodes are shown in Figure 29.

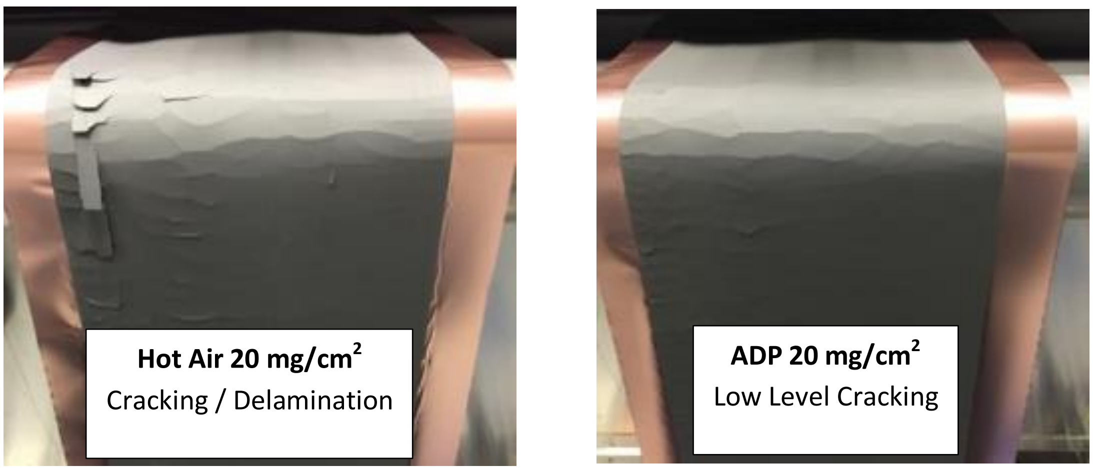


Figure 29. The surfaces of as-dried graphite anodes (without calendering). Hot air dried baseline coating shows surface cracks/delamination while the ADP dried coating shows lower level of cracks.

Binder Distribution

The anode cross-section samples have been monitored by SEM/EDX. The anodes were Osmium (Os)-stained to observe the SBR distribution. Os mapping was performed to compare the binder contents at different zones. The ADP (VFM) dried anode shows lower level of binder migration than the hot air dried baseline (Figure 30).

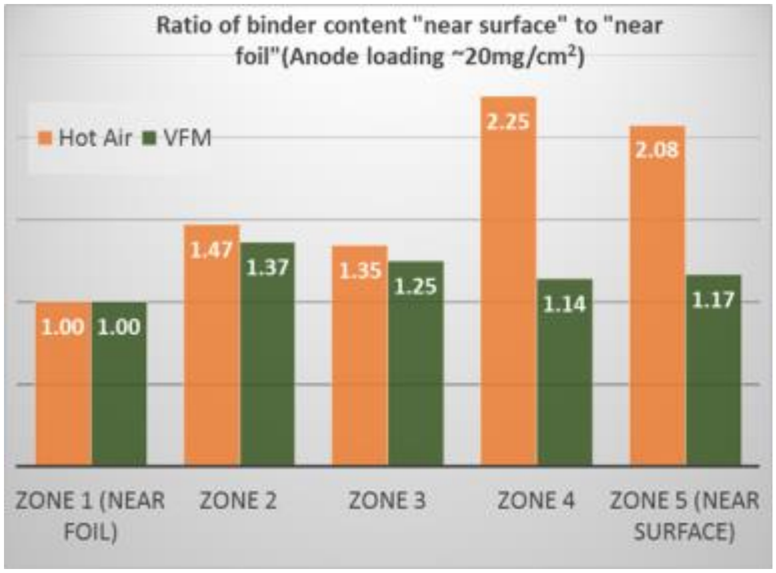


Figure 30. Binder contents at different zones of the electrodes. The electrode cross-section is divided to 5 zones from "near foil" to "near surface". The binder content is normalized (based on the content in Zone 1 "near foil").

Rate Capability

The baseline and ADP anodes at 20 mg/cm^2 were tested for rate capability in half cells (Figure 31). The cells were lithiated at 0.2C (tapering to 0.05C) and then delithiated at different C rates up to 1C from 1.2V to 0.01V. The ADP anode shows better performance than the baseline at 1C.

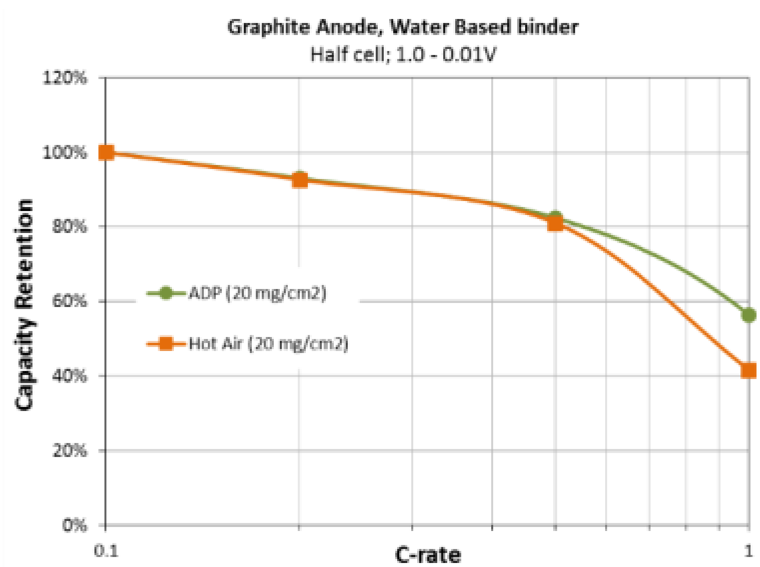


Figure 31. Delithiation rate capability of the anodes (with aqueous binder) at 20 mg/cm2 in half cells. The cells are lithiated at 0.2C to 0.01V (tapering to 0.05C) and delithiated at various rates to 1.0V.

Cycle life testing for high loading half cell cathode

Life cycle experiments for half cells (both standard and ADP dried electrodes) with various loadings were performed at C/2 current rate and 3.0 – 4.2V. It should be noted that high loading electrodes typically show low cycle life in half cells due to the Li counter electrode impedance growth. The cycle life testing of the cathodes is shown in Figure 16. The baseline cathode (at 50 mg/cm^2) failed the cycle life test (repeat was planned but could not be performed because the project period ended). For low loading the cycle life testing were equivalent for hot air and ADP as presented in previous reports. However, as evident from the data in Figure 32, all the high loading ADP cathodes show better cycle life than the respective hot air dried cathodes.

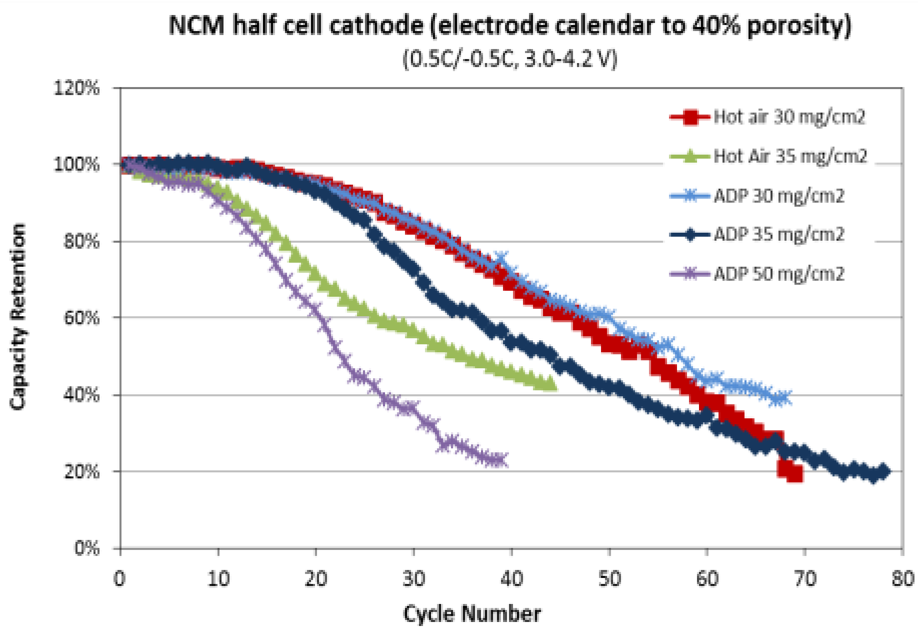


Figure 32. Cycle life testing for half cells comparing standard and ADP dried cathodes (3.5 to 4.2V; 0.5C/-0.5C; room temperature)

Summary

- Preliminary experiments in using ADP as a drying speed booster, has shown very promising results increasing the drying speed of NMP based cathode from 0.25m/min to 1m/min within the same Navitas drying line footprint. ADP as a booster module creates a 4x throughput improvement with only 33% additional dryer footprint required.
- Power measurements demonstrated a 40% reduction in ADP power over convection oven.
- Binder migration studies have been performed on electrodes dried using ADP as a booster. The results show the advantage of using ADP as a booster to conventional drying system – faster drying speeds with minimal increase in binder migration.
- High loading electrodes were fabricated and tested. Not only the binder migration is lower for ADP, but also initial capacities are slightly higher than that for conventionally dried electrodes.

- ADP dried high loading electrodes have shown improved rate capability compared to baseline. This is due to lower binder migration (anode and cathode) and the improved particle cohesion, which reduces electrode impedance.
- VFM/ADP enables drying and fabricating high loading battery electrodes.

Section II, Issues, Risks, and Mitigation:
No issues or delays and actions taken to resolve them.

Section III, Changes in Approach:
The technical milestones were achieved as planned. All ADP low loading samples were as good as the baseline results. A no cost extension was obtained until November 30, 2016. Increased anode and cathode loading has been carried out during the no cost extension. The ADP advantages are actually enhanced for high loading electrodes. Binder migration differences increased. Capacities were higher. Life cycles were better with ADP. Successful drying of other electrode slurries has also been performed. VFM has been explored as a booster module to further increase the drying speed.

Section IV, Key Personnel:

No changes in key personnel or teaming arrangements.

Section V, Project Output:

A. Publications *N/A*
B. Technologies/Techniques: *N/A*
C. Status Reports: *Final report being sent*
D. Media Reports: "*Lambda Technologies Develops Rapid Inline drier for Electrode" Posted on August 13, 2015 in The Battery Show Newsletter;*
Rapid, Low-Cost Battery Electrode Dryer, 2015 US DRIVE Highlights.
E. Invention Disclosures: Two Provisional patents were submitted from this work
   1. *A Provisional application on Apparatus & Method submitted in September 2015*
   2. *A Provisional application on Product by Process Submitted in 2016*
F. Patent Applications: One utility patent has been filed based on this work
   1. *The above Apparatus & Method was filed as Utility Patent in September 2016*
G. Licensed Technologies: *N/A*
H. Networks Fostered: *In Progress*
   - Lambda is in communication with end users as well as battery coating line integrators. Megtec, Kroenert, Manz, Stramma, Tesla and A123Systems are talking with us about how to potentially work together to scale VFM systems for high volume manufacturing.

- Lambda exhibited at the Battery Show September 2015 & 2016 held in Novi, MI to promote VFM to the larger battery community.

I. Websites Featuring Project: *The Battery Show Newsletter*
2015 US DRIVE Highlights

J. Other Products: *N/A*

K. Awards, Prizes & Recognition: *N/A*

Section VI, Follow-On Funding: *N/A*