



# THE PARTICLE TECHNOLOGY FORUM (PTF) NEWSLETTER

An American Institute of Chemical Engineers (AIChE) Forum

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## Message from the Chair

### Greetings!

I hope everyone reading this newsletter took some time this summer to R&R&R - Relax, Refresh, and Reset. It is now time to register for the AIChE Annual Meeting



in Phoenix, AZ! We will be having our first PTF Awards Dinner in 3 years (details to come), social interactions during our poster session and planning meetings, and invited talks by some of our awards winners. A hearty congratulations to all of our award winners! You can read about them in this issue and hear about their research at the conference in person. I will have much more to say about this year's winners at our PTF Awards Dinner.

My own summer was busy moving into a brand new building on Lehigh University's campus, a 190,000 sqft. interdisciplinary facility with shared labs that have dual glass walls facing both student workspaces and South Bethlehem. We are thrilled that after 18 years we are now located in the heart of Lehigh's campus. You can see the building featured on my website, <https://gilchristlab.info> or please come and visit in person! Our research on magnetic Janus particles as responsive microrollers has kept us quite busy in this new space and I'm thrilled to be once again working on granular

## Editorial

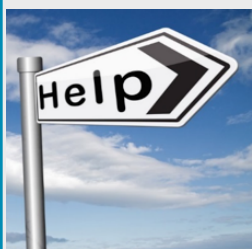
Dear Fellow PTF Members,

I hope you and your families are safe and healthy, and had a wonderful summer!

As a reminder, the face-to-face World Congress on Particle Technology (WCPT9) will be held in Madrid, Spain, from September 18-22, 2022. This newsletter provides key information on the conference.



You will also find in this newsletter technical contributions from the 2021 AICHe PTF SABIC Young Professional Award recipient and Professor Al Weimer.



Please feel free to reach out to me with your idea if you would like

to contribute to the 2022 Fall or Spring 2023 newsletter.

Stay safe!! Stay healthy!!

Regards,

**Mayank Kashyap, SABIC**  
**Editor - PTF Newsletter**

media – even if it is perhaps one that falls far from traditional studies, at least it has forced me to catch up with the literature. Likewise, we hope to be launching our experimental apparatus up to the International Space Station sometime this fall, aiming to study thermophoresis in complex fluids in microgravity to design viral separation platforms. On the personal side, I was happy to take the opportunity to explore Philadelphia (1 hour away) nearby New York City (1.5 hours away), and Washington, D.C. (3 hours away) now that the threat of COVID-19 has somewhat subsided. I also very much enjoyed studying the “single particle dynamics” of tennis balls with my children, both now in high school.

We are entering the period where we nominate and elect new leadership for the Particle Technology Forum Executive Committee. My 4 years of leadership and 10+ years of PTF service now seems to have gone by quickly and has been extremely rewarding, both professionally and personally. If you have interest or questions, please contact me or any of the EC members. Nominations are welcome - I cannot say enough good things about the volunteers we have on our EC, especially my sincere gratitude to Dr. Mayank Kashyap who prepared this newsletter and Dr. Reddy Karri for organizing awards and planning our awards dinner.

Regards,

**James Gilchrist**

**Ruth H. and Sam Madrid Professor, Lehigh University**  
**Chair, The Particle Technology Forum of AICHe**

[Email](#), [Website](#), Twitter: @Gilchrist\_Lab, [LinkedIn](#)



## Our Commitment on Diversity and Inclusion

*Approved at 2019 AIChE Annual Meeting*

The AIChE Particle Technology Forum is committed to maintaining a diverse and inclusive community of highly skilled chemical engineering professionals within the environment of the Institute and profession in which all members, regardless of characteristics such as gender identity and expression, race, religion, age, physical condition, disability, sexual orientation, educational level, socioeconomic class, nationality or ethnicity, are valued and respected.”



As a global scientific and engineering society, we affirm the international principles that the responsible practice of science, free from discrimination in all of its forms, is fundamental to scientific advancement and human wellbeing, as outlined by the International Council for Science’s (ICSU) Statute 51. We also affirm our commitment to an engineering and scientific environment that facilitates the planning, execution, review and communication of engineering and scientific work with integrity, fairness, and transparency at all organizational levels. This extends to our general scientific endeavors—including our professional interactions and engagement with other engineers, scientists, students, trainees, and the general public. We recognize that harm to our profession, our scientific credibility, individual wellbeing, and society at large is caused by not doing so.

To this end, the PTF will implement the principles of diversity, inclusivity, and equity within PTF leadership and membership to build a community across the chemical enterprise. We are committed to quantifying and monitoring our diversity at least annually at the Executive Committee and reported at the general business meeting.

## 2021 AIChE Particle Technology Forum Award

### SABIC Young Professional Award

#### Particulate Process and Product Design: Single Drop Granulation

**Heather Emady**

*Associate Professor*

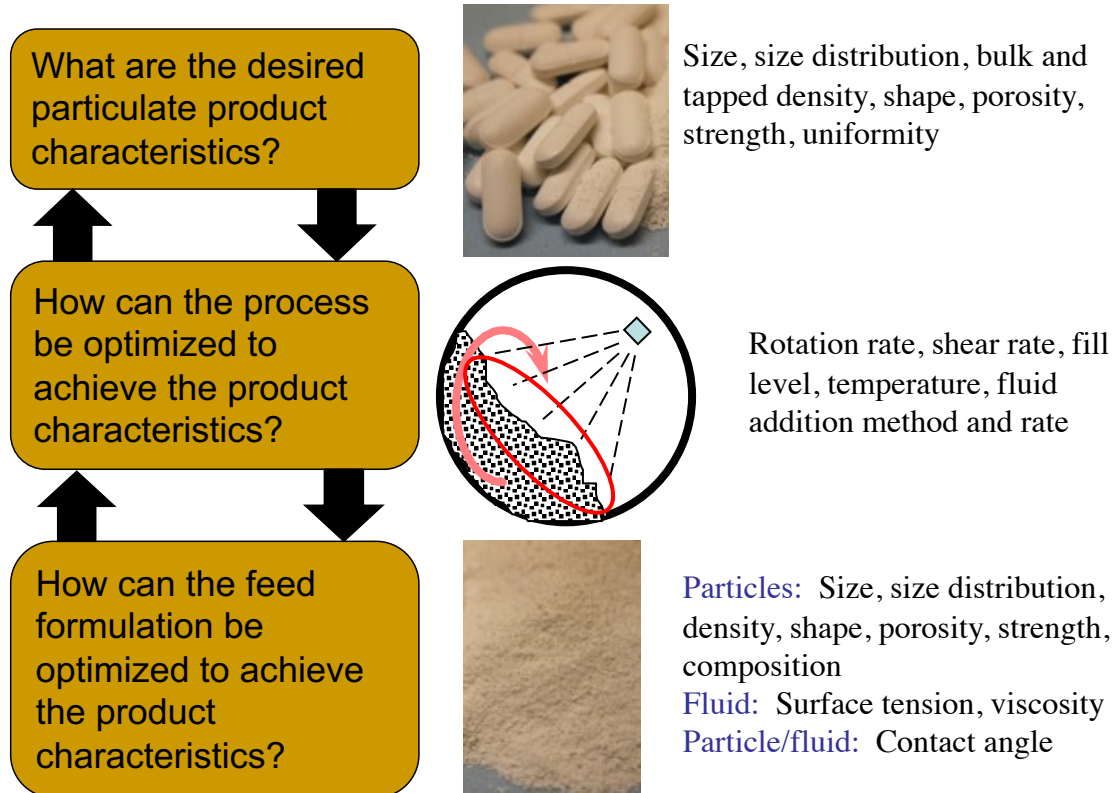
*School for Engineering of Matter,  
Transport and Energy*

*Arizona State University*



Many industries, including pharmaceuticals, food, agricultural chemicals, detergents, catalysts, and consumer products, manufacture a variety of products from particulate materials. Product or intermediate specifications may include size, size distribution, shape, strength, porosity, and content uniformity of particles or of particulate-based delivery forms. Our research

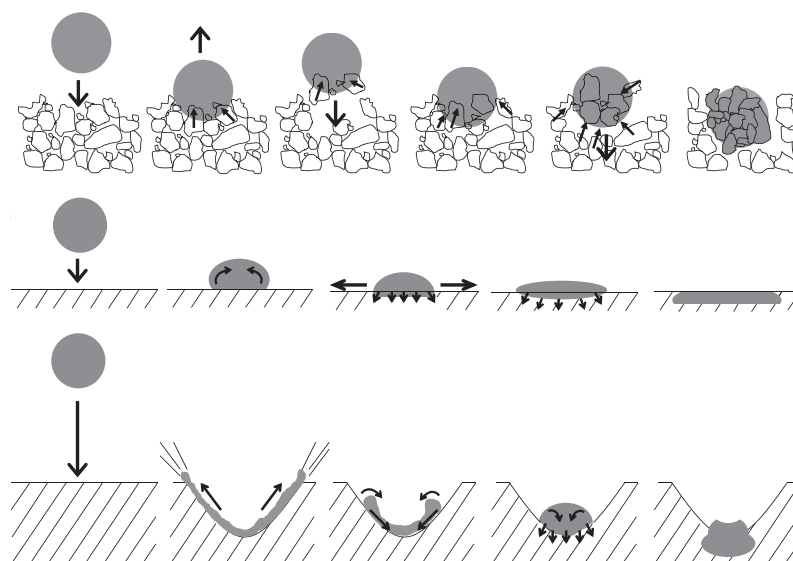
group designs particulate products and processes through the study of fundamental phenomena involved in particle-particle, particle-fluid, and particle-equipment interactions to produce required product specifications. By working backwards from the desired end product attributes, this multiscale design approach covers the entire sequence of raw feed formulation handling through equipment processing to the final granular product (see Fig. 1). Here, we will highlight single drop granulation as a form of particulate process and product design.



**Fig 1.** Particulate process and product design framework: quantitative relationships between granule properties and formulation and process parameters are needed

Our group is known for experimental and theoretical work on granule formation mechanisms (i.e., the processes by which a liquid drop interacts with a granular bed to form a larger granule composed of smaller primary particles). In experiments utilizing high-speed video imaging, three different mechanisms that can occur for granule formation over a wide range of formulation and process parameters were identified: Tunneling, Spreading, and Crater Formation.<sup>1</sup> Tunneling occurred in loose, cohesive powder beds, with dry powder aggregates being sucked into the drop which then tunneled into the bed, producing round granules (see Fig. 2, top). With coarser powder beds, Spreading occurred at low impact velocities, forming flat disks (see Fig. 2, middle). Using the same coarser beds at high impact velocities led to Crater Formation, with the drop forming a crater and elastically deforming within the crater, coating the drop in a layer of powder before penetrating into the bed by capillary action (see Fig. 2, bottom). Granules formed

via Crater Formation were of varying shapes, but generally rounder than those produced from Spreading.



**Fig 2.** Schematics of the three granule formation mechanisms, where a droplet impacts and penetrates into a powder bed to form a granule: Tunneling (top), Spreading (middle), and Crater Formation (bottom).<sup>2</sup>

To quantify the conditions under which each mechanism will occur, dimensional analysis was performed and a new regime map was created that plots the powder bed porosity,  $\epsilon$ , against the modified granular Bond number,  $Bo_g^*$ , which is a ratio of the capillary force acting on a particle to the gravitational force acting on the particle:<sup>2</sup>  $Bo_g^* = \frac{\gamma \cos \theta}{d_{32}^2 \rho_p g}$  and  $\epsilon = 1 - \frac{\rho_{bed}}{\rho_p}$ , where  $\gamma$  is the liquid surface tension,  $\theta$  is the solid-liquid contact angle,  $d_{32}$  is the surface mean particle size,  $\rho_p$  is the particle density, and  $\rho_{bed}$  is the powder bed density. Thus, this regime map takes into account primary particle properties, liquid properties, and the packing state of the powder. Each regime is associated with a range of granule shapes; thus, calculating the dimensionless groups allows *a priori* knowledge of the expected product granule shape. Without wasteful and time-consuming lab-scale and pilot-scale testing, the feed and process properties can be adjusted (altering the dimensionless groups) to operate in a different regime that will result in the desired granule shape.

### Case Study: Binary Mixture of APAP and MCC<sup>3,4</sup>

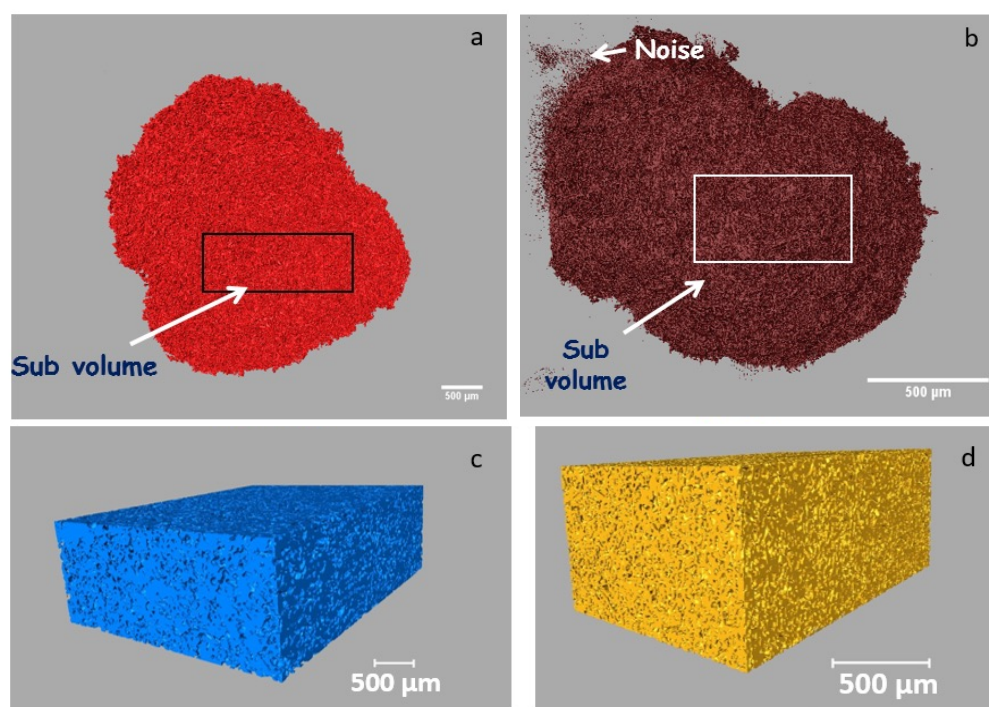
To test the regime map beyond single-component inorganic powders, the two-component active pharmaceutical ingredient (API)-excipient mixture of acetaminophen (APAP) and microcrystalline cellulose (MCC) was studied. The hydrophobicity and primary particle size distribution were varied to determine their effects on the drop penetration time, granule formation mechanism, granule morphology, granule and powder bed internal structure, and granule content uniformity.

For drop penetration time, the penetration time increased with an increase in APAP proportion, and drastically increased in mixtures with fine components. For granule formation mechanisms, Spreading occurred in coarse mixtures, Tunneling occurred in fine mixtures, and Spreading



transitioned to Tunneling with decreasing particle size in mixtures with both coarse and fine components. For granule morphology, coarser mixtures (Spreading) formed plate-like granules, while fine mixtures (Tunneling) formed rounder granules. These results confirm that the mixtures follow the same trends observed for single component materials, where particle size dominates.

Using X-ray micro-CT, the internal structures of the resulting granules were examined (see Fig. 3). The Spreading-formed granules had evenly distributed pore networks and higher overall porosities than the Tunneling-formed granules, which had smaller pores with large isolated voids. Here, the granule internal structure aligns with the formation mechanism. However, granules formed from a transitional Spreading/Tunneling mechanism had non-uniform pore distributions, with both dense and loose regions. To better understand the formation of this irregular pore distribution observed with the transitional mechanism, the internal structure of the powder bed prior to granule formation was studied, and it was found that the heterogeneous powder packing existed before granulation. Also, it was confirmed that the granulation densified the internal structure, with the granule porosity being much larger than the bed porosity.



**Fig 3.** X-ray microtomography images of granules of a) microcrystalline cellulose (MCC), and b) micronized acetaminophen (APAP). From these 3D granule reconstructions, sub volumes were selected to measure porosities of c) MCC: porosity  $68.7\% \pm 0.6\%$ , and d) APAP: porosity  $60.8\% \pm 0.4\%$ .

Using UV-vis spectrometry, the API content uniformities of the powder bed surface and the resulting granules were quantified. The API content was close to the theoretical values for mixtures with less than 50% APAP; above this threshold, the actual amount was generally less than expected, and thus the formed granules were sub-potent. Above 50% APAP, in mixtures of different particle sizes, there

were generally discrepancies between the bed surface and the formed granules, and thus preferential wetting<sup>5</sup> occurred.

### Summary and Future Directions

Single-drop formed granules have narrow size distributions, but the shape depends on the formation mechanism. Tunneling, Spreading, and Crater Formation are the different granule formation mechanisms, which can be predicted by a regime map for single components.

APAP/MCC mixtures displayed expected formation mechanism and morphology behavior, based on particle size. The granule internal structure corresponds to the formation mechanism. The granule content was compromised for all mixtures for APAP amounts greater than 50%. The next step in this work is to test the regime map for these results, or develop a new regime map that incorporates the complex properties of mixtures.

This study leads us closer to our big-picture goal of creating a link between the formulation properties, the granule formation mechanism, the granule morphology and internal structure, and granule performance metrics.

### Acknowledgements

I would like to acknowledge financial support from Science Foundation Arizona's Bisgrove Scholar Award, as well as the NSF CAREER Award. I would like to thank my PhD advisor, Prof. Jim Litster, for first introducing me to the wonders of high-speed video and single drop granulation; Prof. Rohit Ramachandran and Prof. František Štěpánek, for providing the idea for the pharmaceutical mixtures based upon their interesting high-shear granulation results; Prof. Nik Chawla, for use of his X-ray micro-CT; and Dr. Tianxiang Gao, who was my first PhD student, and performed the newer work with the pharmaceutical mixtures. I am also very grateful to the PTF for this prestigious award, and to my nominators and the award selection committee, in particular.

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# How Semi-continuous Al<sub>2</sub>O<sub>3</sub> ALD Films on Li-ion Cathode Particles Improve Battery Performance

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(This article is adapted in part from the author's research group's published open access article found at link: <https://pubs.acs.org/doi/pdf/10.1021/acsnm.9b01490>)

It took about 15 years from discovery of the platform technology using Particle Atomic Layer Deposition (ALD) to coat primary particles with a film<sup>1</sup> before industry finally became interested. The process is low-cost, scalable to thousands of mt/day, and is unequalled in terms of film quality for various applications. It was found that only several ALD cycles (2 to 4) were required to provide for optimal improvement of Li-ion cell performance, the cells fabricated from alumina ALD coated cathode powders<sup>2</sup>. This was an intriguing result, since 2 to 4 ALD cycles would not be enough to place a continuous film on a substrate. So, why were these exceptional results obtained? How could the surface chemistry be analyzed since the film thickness was typically sub-nm?

It was commonly assumed, prior to the work described here, that the ALD films on the cathode particles were of uniform thickness and were optimally thin enough to facilitate lithium diffusion while blocking side reactions of the electrolyte with the cathode substrate. The key to understanding the actual reason required elucidating the nature of thin alumina films deposited with only several ALD cycles on the lithium nickel manganese cobalt oxide cathode (NMC) precursor particles. Most of the prior studies used x-ray photoelectron spectroscopy (XPS) to analyze the ALD coated particles, but XPS has a resolution of 5 nm (it really cannot effectively analyze sub-nm films).

So, XPS was ruled out for this investigation in favor of Low Energy Ion scattering (LEIS), resolution of outer the atomic surface, and Secondary Ion Mass Spectroscopy (SIMS), resolution of 1 nm, used in tandem. This is believed to be the first use of this dual analysis for investigating powder coatings.

Lithium nickel manganese cobalt oxide particles obtained from Sigma Aldrich with the composition LiNi<sub>0.333</sub>Mn<sub>0.333</sub>Co<sub>0.333</sub>O<sub>2</sub> (NMC111) were coated in a vibrating fluidized bed reactor. Additional separate experiments deposited alumina ALD onto LiOH (Sigma Aldrich, reagent grade > 98%, 3.2 m<sup>2</sup>/g), and Li<sub>2</sub>CO<sub>3</sub> (Sigma Aldrich, ACS reagent grade > 99%, 0.7 m<sup>2</sup>/g) powders. The reactor system consisted of a reactor tube heated in a vertical clamshell furnace, a vibration generation system, a data acquisition system (DAQ) interfaced with LabView™, and in-situ mass spectrometry.

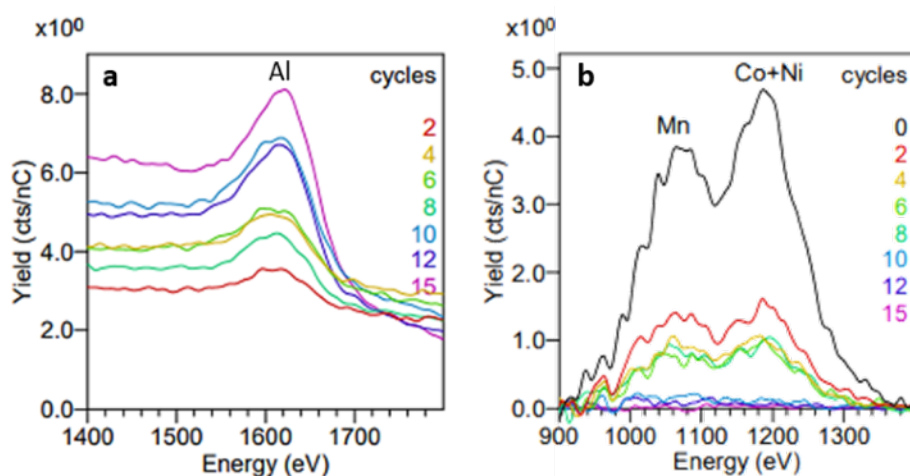
Alumina ALD was performed using the two-step TMA/water reaction. Particle ALD was carried out for 2, 4, 6, 8, 10, 12, and 15 cycles. Energy dispersive X-ray spectroscopy (EDS) mapping of NMC111 particles coated with 2 ALD cycles shows uniform distribution of aluminum across a population of particles indicating that there was consistent coating within the batch.



Electrochemical coin cell cycling studies using coated powders indicated that 4 cycles of  $\text{Al}_2\text{O}_3$  ALD exhibited an optimal stability without sacrificing too much capacity. Therefore, our surface characterization investigation focused on the optimal 4 cycles of  $\text{Al}_2\text{O}_3$  coated NMC particles.

The full range of ALD coated NMC111 samples was analyzed with LEIS to determine the composition of the outermost atomic layer with increasing alumina deposition. This measurement directly relates to the coverage of alumina on the surface of the powders as the elemental composition is measured for the outermost atomic layer only. LEIS cannot detect Li because it is too light for analysis with 3 keV  $^4\text{He}^+$ , and therefore the LEIS results only indicate the presence of Al, Ni, Mn, and Co in the outermost atomic layer.

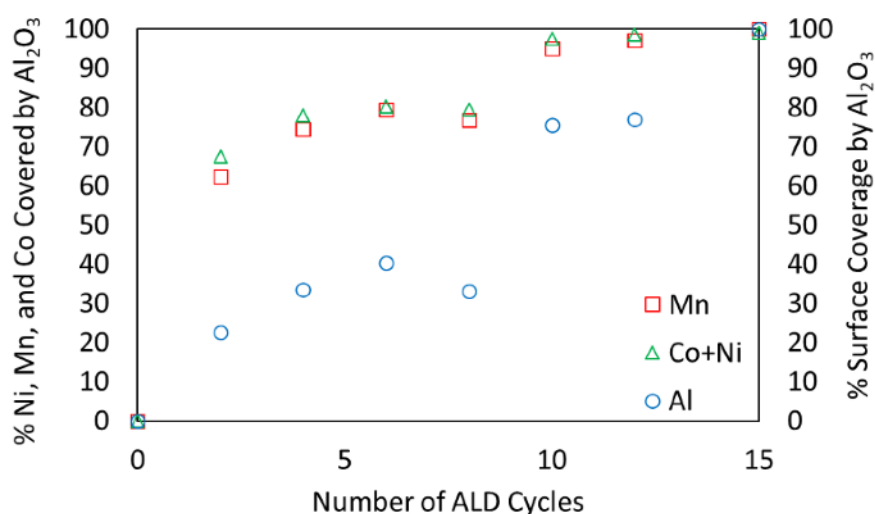
The LEIS results demonstrate that with increasing cycle number, the Mn, Co, and Ni signals decrease, indicating that the amount of Mn, Co, and Ni in the outermost atomic layer decreases with increasing ALD cycle numbers. At the same time, the background Mn, Co, and Ni signals shift to lower energies, indicating that the Mn, Co, and Ni reside further from the sample surface because they are now covered by aluminum oxide. Additionally, the Al peak intensity increases over the course of the deposition because the amount of Al in the outermost atomic layer increases with ALD cycle number. Figure 1a shows separate spectra where only the region around the Al peak was recorded. Because the detection of Mn, Ni, and Co by LEIS is best achieved using 5 keV<sup>20</sup>  $\text{Ne}^+$  ions, the quantitative analysis for these elements was performed using that spectra (see Figure 1b).



**Fig. 1.** LEIS spectra of the (a) Al and (b) Mn and Co+Ni characteristic peaks. As the cycle number increases the Al signal increases with a corresponding decrease in both the Mn and Co+Ni signals, indicating that the film is coating on the substrate surface. The Mn and Co+Ni peaks are completely suppressed by 10 cycles of  $\text{Al}_2\text{O}_3$  ALD.

The Al, Mn, and Co+Ni peaks were integrated to allow direct comparison of the two separate LEIS spectra. The Mn, and Co+Ni data were normalized to the Mn and Ni+Co signals of the uncoated sample to determine the fraction of the original Mn, Co, and Ni surface atoms that were covered over the course of deposition. In this way, the fraction covered references that fraction of the original Mn, Co, and Ni present on the surface and not the fractional coverage of the overall surface. The Al data were normalized to the sample coated with 15 cycles, because this sample represents the maximum amount of  $\text{Al}_2\text{O}_3$  deposited within this study. Due to the first atomic layer

sensitivity of LEIS, the aluminum signal would be expected to plateau after a uniform film is deposited over the entire surface. Continued increase of Al concentration within the first atomic layer indicates that the film has not yet uniformly covered the surface of the particles. The results are summarized in Figure 2, where Mn, Ni, and Co in the first atomic layer are nearly completely covered by 10-12 cycles of ALD. The aluminum signal continues to increase for the three samples with the thickest coatings, indicating that a component of the original surface persisted and the surface was not completely covered by the ALD layer. This phenomenon alone does not unequivocally support the hypothesis of incomplete coverage. Due to LEIS's inability to directly measure Li, we interpret this observation as an indication that the uncoated component was Li surface species; however, this cannot be confirmed without the use of additional analytical methods that are capable of directly detecting Li. To probe this conclusion, time-of-flight secondary ion mass spectroscopy (TOF-SIMS) was employed to directly measure the presence of Li near the surface.



**Fig 2.** Integrated LEIS data representing the fractional coverage of the surface as the number of ALD cycles increases. Mn, Co, and Ni are completely covered by 10 TMA/H<sub>2</sub>O ALD cycles. However, the aluminum at the surface is not yet saturated as it would be for a complete film, indicating that the ALD preferentially deposits on the Mn, Co, and Ni sites while leaving Li uncovered until a continuous film is formed.

TOF-SIMS (Physical Electronics TRIFT III) was used as an additional spectroscopic method to study the surfaces of these samples. Lithium can be measured with TOF-SIMS, which provides direct detection of lithium that LEIS cannot provide. Additionally, the resolution of TOF-SIMS is  $\sim 1$  nm. TOF-SIMS analysis was completed for 3 of the 8 samples from which LEIS data were collected. Samples composed of uncoated NMC111, and NMC111 with 4 and 15 ALD cycles of alumina were analyzed with TOF-SIMS to directly measure the lithium concentration within the first nanometer of the surface.

The ion images presented in Figure 3 show the integrated intensities of mass selected ions as a function of pixel position. In these images, brighter colors correspond to higher concentrations of the selected ion in the sample relative to the concentration of that element in the other samples. The ion images and the spectra collected across the range of samples show that the initial concentrations of Ni, Mn, and Co are much lower than that of Li on the surface. Consistent with our LEIS measurements, as additional layers of alumina are deposited by ALD, the amount of Ni, Mn,

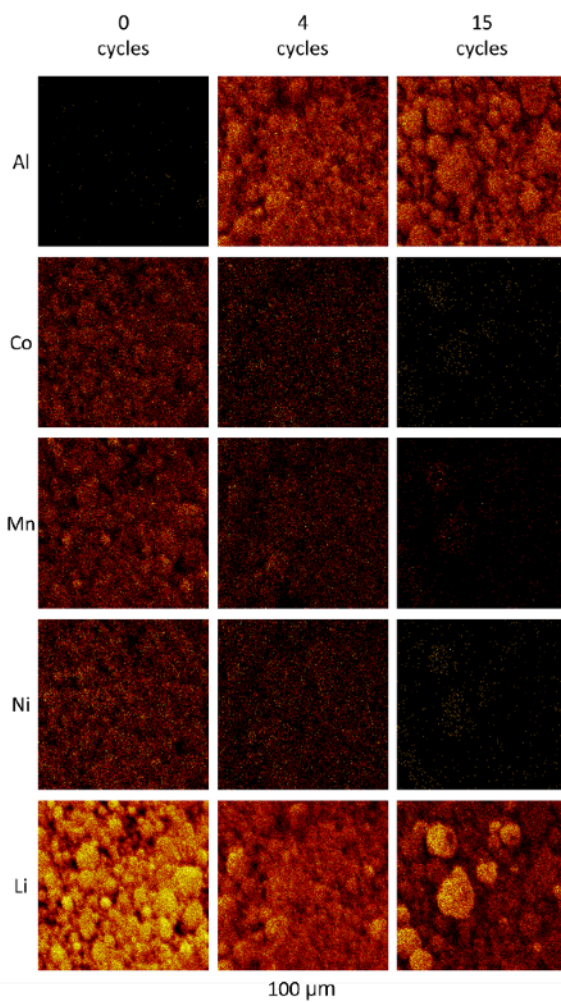
and Co on the surface is nearly completely suppressed while the presence of Li is still clearly observed. A comparison of the residual amount of each signal as ALD proceeds (Figure 4) shows that after 4 cycles of alumina ALD, the Ni, Mn, and Co are all 70-80% covered by alumina, while almost 50% of the original Li remains exposed. After 15 cycles of ALD, the Ni, Mn, and Co signals have been completely suppressed but over 25% of the original Li signal is still observed, again indicating that the alumina ALD preferentially coats the transition metal bound sites leaving Li exposed on the surface. With increasing cycle number, the signal for the Ni, Mn, and Co metal centers decreases to a larger degree than the Li signal. This effect could be caused by increased ejection of Li, compared to transition metal atoms due to the relative atomic weights. To explore this potential, the TOF-SIMS signal count for  $^6\text{Li}$ , which is significantly lighter than  $^7\text{Li}$ , was compared to that for  $^7\text{Li}$  (Table 1 in SI). The signal counts for both  $^7\text{Li}$  and  $^6\text{Li}$  appear to follow the same trend, indicating that the differences seen in the overall TOF-SIMS analysis are not entirely driven by atomic weight.

LEIS and TOF-SIMS indicate that the outer surface of the NMC111 particles following ALD is comprised mainly of oxides of Li and Al. The ratio of Al to Li present at the surface of the particle increases as additional ALD cycles are carried out (Figures 3 and 4).  $\text{Li}_2\text{CO}_3$  and  $\text{LiOH}$  are typically present on the surface of NMC materials<sup>3-7</sup> and were confirmed with XPS analysis. FACTSage free energy minimization indicates that  $\text{LiAlO}_2$  is the thermodynamically favorable product of a reaction between  $\text{LiOH}$  and  $\text{Al}_2\text{O}_3$ . Hence, it is likely that  $\text{LiOH}$  on the NMC111 surface reacts quickly with  $\text{TMA}/\text{H}_2\text{O}$  to produce an amorphous  $\text{Li}_2\text{O} - \text{Al}_2\text{O}_3$  type film that supports rapid Li diffusion.<sup>8-10</sup> Continued ALD cycling results in  $\text{Al}_2\text{O}_3$  deposition and an eventual insulating film that inhibits battery charge-discharge cycling.

In order to assess the formation of a  $\text{Li}_2\text{O}-\text{Al}_2\text{O}_3$  hybrid film and to quantify the rate at which ALD alumina deposits on the known Li compounds present at the surface ( $\text{LiOH}$  and  $\text{Li}_2\text{CO}_3$ ), the same alumina ALD chemistry used on the NMC111 materials ( $120^\circ\text{C}$ , 2 Torr) was replicated on  $\text{LiOH}$  and  $\text{Li}_2\text{CO}_3$  powders. Following ALD, the materials were analyzed using BET to determine their surface area and ICP-MS to determine their elemental Al content. The concentration of Al on the  $\text{LiOH}$ ,  $\text{Li}_2\text{CO}_3$ , and NMC111 powders is compared in Figure 5. The results are expressed as Al wt% normalized by the BET surface area of the uncoated substrates.

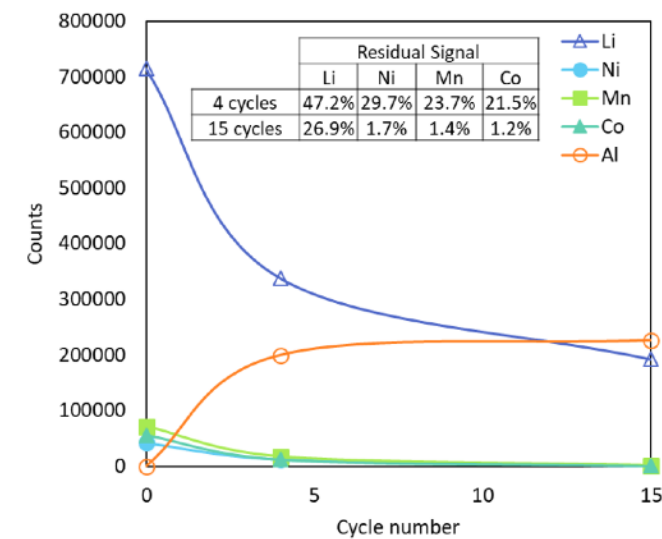
The higher concentrations of Al present in the  $\text{LiOH}$  samples relative to the  $\text{Li}_2\text{CO}_3$  indicate that the ALD growth rate is slightly higher on  $\text{LiOH}$  than on  $\text{Li}_2\text{CO}_3$  powders. The ALD growth on  $\text{Li}_2\text{CO}_3$  appears to be self-limiting  $\text{Al}_2\text{O}_3$  ALD-type growth, where each half reaction deposits a consistent amount of material controlled via the concentration of active sites present on the surface. This is observed by the linear growth that intercepts the y-axis at zero (i.e., with 0 ALD cycle, there is a negligible amount of Al measured with ICP). However, the film growth on  $\text{LiOH}$  appears to develop in a different manner. The y-intercept of the growth curve is elevated above zero, indicating that the consistent linear deposition is observed between 2 and 12 cycles is not the same deposition that occurs between 0 and 2 cycles. The initial reaction does not appear to be self-limiting ALD and is likely the formation of a hybrid Li-Al oxide reaction product. The growth of Al observed on the NMC111 particles is offset from zero at zero cycle, indicating that potentially the initial  $\text{TMA}/\text{H}_2\text{O}$  dose resulted in a stoichiometric reaction producing an Li-Al oxide product. There then appears to

be a rapid growth of Al on the surface until an inflection of the growth is observed at ~9 cycles, which may be due to achieving complete or nearly complete coverage of the NMC111 surface by the film at this point.

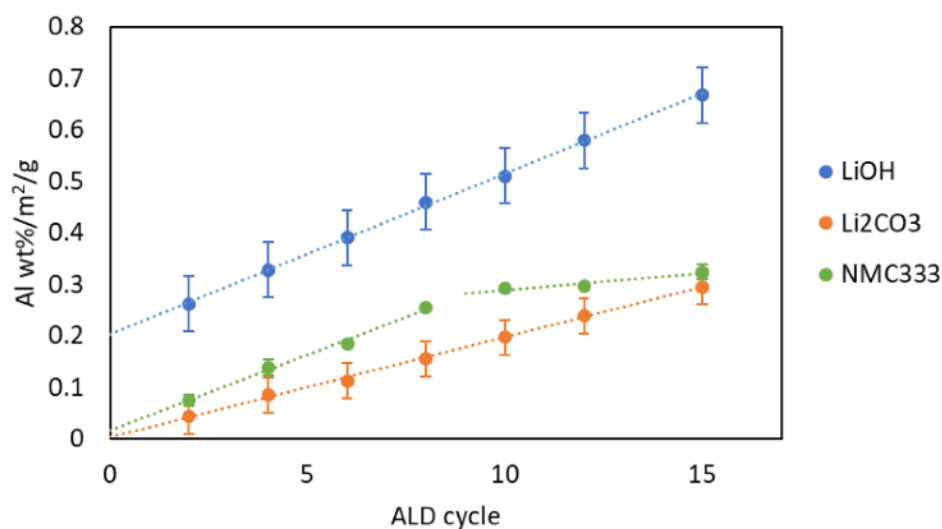


**Fig 3.** Ion images from TOF-SIMS analysis. Images of Li, Ni, Mn, Co, and Al signals are presented top-to-bottom with the number of alumina cycles shown left-to-right. The images show the Li concentration decreasing as alumina is deposited on the sample. However, the Li signal is not completely suppressed as compared to the signals representing Ni, Mn, and Co. The suppression of the signals for Ni, Mn, and Co indicate that they are nearly completely covered. The intensity of the color in the TOF-SIMS images is correlated to the elemental concentrations within the measurement depth. Intensity of color can be quite subjective, so the results of this study were also presented in a numerical form via the signal counts presented in Figure 4. The variation in intensity visible in the 15 cycle lithium image can be attributed to large variation in particle size/curvature and z-height.

The surface area normalized deposition rates on the LiOH and Li<sub>2</sub>CO<sub>3</sub> powders are 0.031 wt% Al/m<sup>2</sup>/g per cycle and 0.019 wt% Al/m<sup>2</sup>/g per cycle, respectively. While we cannot discount the potential for lithium to diffuse through the substrate and film as lithium has a high mobility, these results indicate that the bonding characteristics of the lithium present at the surface will significantly impact the rate at which these sites are coated by the film.



**Fig 4.** Absolute signal counts of Li, Al, Ni, Mn, and Co from TOF-SIMS maps. Residual signal percentage (relative to the uncoated sample) for the main components of the NMC cathode (Li, Ni, Mn, and Co) after 4 cycles and 15 cycles of alumina ALD are presented in the inset table. The values clearly indicate that ALD preferentially deposits alumina on transition metal surface sites and full coverage of surface lithium with greater than 1 nm alumina is not achieved within the 15 ALD cycles presented here.



**Fig 5.** Comparative area-normalized wt% Al for LiOH, Li<sub>2</sub>CO<sub>3</sub>, and NMC111 for TMA/H<sub>2</sub>O ALD cycling. Al wt% data from ICP-MS were normalized by the BET surface area of each uncoated substrate powder. The higher growth rate on LiOH vs. Li<sub>2</sub>CO<sub>3</sub> indicates that these surfaces behave differently from one another during the Al<sub>2</sub>O<sub>3</sub> ALD process, which has implications for the observed Al<sub>2</sub>O<sub>3</sub> growth on NMC substrates. It appears that some non-ALD reaction is occurring during the first ~ 9 ALD cycles, possibly a reaction forming a Li-Al oxide product, until a typical Al<sub>2</sub>O<sub>3</sub> ALD film deposits from 10 to 15 cycles.

Surface analysis showed that low-cycle number ALD films were not uniform nor uniformly thick over the surface of the cathode particles and that alumina ALD preferentially deposited on transition



metal bound sites on the cathode particle surface and coated Li on the surface to a lesser extent. Lithium was found to still be present on the cathode powder surface, even after 10 ALD cycles. Contrary to current supposition, low-cycle ALD appeared to improve the cycling stability of battery cathode active materials through this preferential growth that stabilized the transition metal oxides in the presence of electrolyte without blocking lithium intercalation pathways. This was the first study to determine that Li remains exposed on the as-synthesized surface of ALD coated cathode particles and that the ALD film is non-uniform and non-uniformly thick when less than 10 ALD cycles are used. This process can be scaled-up using a turn-key commercial spatial Particle ALD CIRCE coating system with production rates up to 4 mt/hr (<http://www.forgenano.com/products/circe/>).

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# 2022 AIChE Particle Technology Forum Awards

## Elsevier PTF Lifetime Achievement Award



### Professor Shankaran Sundaresan's

research at Princeton University has led to a better understanding of the instability mechanisms through which inhomogeneous structures emerge in granular and fluid-particle flows and filtered models for momentum, mass, and energy transport in gas-particle flows, which account for the consequences of mesoscale structures that would not be resolved in coarse simulations of industrial scale devices. His research has also led to refined models for the rheology of granular materials that bridge across inertial, frictional, and cohesive flow regimes.



Over the past several decades, Sundaresan and his collaborators

have applied Electrical Capacitance Tomography to elucidate flow regimes in pneumatic conveying and loop instability in circulating fluidized beds, probed flow and separation efficiency in cyclones through detailed simulations, analyzed the interplay between triboelectric charging and fluid-particle flows, performed particle-resolved simulations to develop improved constitutive relations for fluid-particle interaction force, and investigated the fluidization and deagglomeration of particles in dry powder inhalers. His contributions constitute an impressive body of work that spans the spectrum of gas-solid flows and is remarkable for its unique combination of formal elegance and practical utility. He has also served the particle technology forum community through mentorship of students and postdoctoral fellows, associate editorship of the AIChE Journal, service on editorial boards of particle technology-related journals, and several review and perspective articles.

## Thomas Baron Award in Fluid-Particle Systems



### Professor Ah-Hyung (Alissa) Park is the

Lenfest Earth Institute Professor of Climate Change in the Departments of Earth and Environmental Engineering & Chemical Engineering at Columbia University. She is also the Director of the Lenfest Center for Sustainable Energy. Her research focuses on sustainable energy and materials conversion pathways with emphasis on integrated Carbon Capture, Utilization and Storage (CCUS) technologies based on particle technology fundamentals. Park received a number of professional awards and honors including



ACS ENFL Mid-Career Researcher Award (2022), U.S. C3E Research Award (2018), PSRI Lectureship Award at AIChE (2018), and NSF CAREER Award (2009). Park also led a number of global and national discussions on CCUS including the Mission Innovation Workshop in 2017. Park is a Fellow of AIChE, ACS, RSC and AAAS.

### PSRI Fluidization and Fluid Particle Systems Award



**Dr. Naoko Ellis** is a professor in the University of British Columbia (UBC) Department of Chemical and Biological Engineering with a profound



desire to help create a low carbon future. Her expertise in multiphase systems, especially in fluidization is applied through biomass utilization (bio-oil upgrading, biochar potential, and hydrogen production), and CO<sub>2</sub> capture (chemical looping systems). She is curious about how learning with and from "others" - crossing the disciplinary boundaries - can inform and frame the complex societal problems we face. Her current projects includes Transdisciplinary Collaborative PhD Program for Climate Emergency development, Carbon

Capture Utilization and Sequestration road mapping, and renewable natural gas production among others.

### SABIC Young Professional Award



**Professor Edison Huixiang Ang** is currently an Assistant Professor at the National Institute of Education, an institute of Nanyang Technological University. He received his PhD degree from Nanyang Technological University in



2017, then he continued his postdoctoral research at National University of Singapore and Nanyang Technological University. He is also currently an Early Editorial Board Member of Chemical Engineering Journal and Young Editorial Board Member of the Journal of Leather Science and Engineering. He received the Outstanding ASIAN Science Diplomat Award and the Vebleo Fellow Award in 2021, as well as the Young Scientist Medal Award and

Journal of Materials Chemistry A Emerging Investigators in 2022, in recognition of his outstanding research work and leadership in the fields of science, engineering, and technology. His current research interests combine nanotechnology and materials science, additive manufacturing approaches to develop functional nanostructures and smart materials for advanced energy storage, membrane technology, catalysis and sensing applications.



## George Klinzing Best PhD Award



**Dr. Tony Feric** completed his undergraduate studies at the University of Maryland College Park in 2017, where he majored in Chemical Engineering and minored in Technology Entrepreneurship through the Hinman CEOs program. Tony received his M.S. and Ph.D. in Chemical Engineering at Columbia University in 2022 under the supervision of Ah-Hyung Alissa Park. While at Columbia, Tony developed nanoscale hybrid electrolytes for combined CO<sub>2</sub> capture and conversion applications. The focus of his thesis was understanding the thermal, structural and transport behaviors of Nanoparticle Organic Hybrid Materials (NOHMs) in their application as electrolyte additives. Tony also participated in the



University of  
Pittsburgh

Columbia Technology Ventures Fellows program where he assisted Technology Licensing Officers in making decisions about the patentability of various technologies developed by Columbia faculty members. Additionally, Tony served as one of the co-chairs for the 2022 Gordon Research Seminar on Carbon Capture Utilization and Storage (CCUS). Currently, Tony is an ORISE Science, Technology and Policy Post-Doctoral Fellow at the Department of Energy's Office of Fossil Energy and Carbon Management, and hopes to pursue a career pathway at the intersection of CCUS enabling science, technologies and policies.





## WORLD CONGRESS ON PARTICLE TECHNOLOGY (WCPT9)



The 9<sup>th</sup> World Congress on Particle Technology (WCPT9) will be held in September 18-22, 2022, in Madrid (Spain) as a face-to-face event. WCPT9 is the world's most influential event for the particle and bulk technology community under the auspices of the World Assembly of Particle Technology, representing main societies and federations around the world. It's where world-leading researchers and companies share the latest thought leadership about the progression and future of particle technology. And it's the best place for networking opportunities with your colleagues to share mutual professional goals.

It happens every time. You arrive in WCPT and think "What a great atmosphere!" there's something in the air, an energy that invigorates you and fills you with positive emotions sharing your knowledge with your colleagues. Together, face-to-face, we will shape the future of particle technology. It's time to reconnect, meet again, enjoy networking with your colleagues in a safe environment discovering a unique way of living in Madrid.

The scientific program outlined covers the most relevant topics nowadays for industry and our society. WCPT9 main topics are:

- Particulate solids handling
- Particle and particulate systems characterization
- Particle processing
- Particle-fluid systems: fluidization and multi-phase flow
- Particle formation and design
- Particle separation
- Aerosol particles
- Nanoparticles: production, characterization, and applications
- Modelling and simulation
- Science, Technology, Engineering and Design in particle-based materials and products.

WCPT9



EXPLORING  
BEYOND  
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Crystals Food powders  
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Aggregates Atomized particles  
Fly ash Metal powders Pellets Grains  
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Granules Dust Healing particles  
Powders Emulsion drops Nanoparticles  
Recycled particles Aerosols Microplastics  
Colloidal nanoparticles Microgel particles  
Particle emissions Biomass Microcapsules  
Biomolecule-based particles Quantum dots  
Micro-particles Fluidized bed particles  
Particle-based materials and products  
Pharmaceutical tablets ...

Beyond these main topics, Joint Events (JE) will be also organized in engineering, scientific or technically related fields, where particle technology is present. Three WCPT9-JE have been already confirmed:

- Challenges of microplastics: analysis and control.
- III ANQUE-DECHEMA Leading edge conference “Particle Technology. Shaping the future”.
- Multidimensional particle properties: characterization, separation, and application.
- Thermomechanical behaviour of granular materials (MATHEGRAM).

Two memorial sessions are being also organized in honor of Prof. Rolf K. Eckhoff and Prof. Ugur Tuzun to remember them and celebrate their work.

WCPT9 has received over 570 contributions from all around the world until now, counts with the confirmation of 8 plenary speakers, around 40 keynotes, over 450 oral and flash presentations, and over 80 poster communications covering a wide range of areas related to particle technology. Under the theme “**Exploring beyond limits**”, WCPT9 aims to engage the whole community on particle technology in the different continents and their countries through an outreach and promotion program specifically designed for this purpose.

The deadline for abstract submission was 2 May 2022 but it will be possible to send us your research work as a poster (5 min orally presentation) until **18 September 2022, do not miss the opportunity to participate!** You will find all the details to prepare your abstract and the information related to the evaluation in the following link:

[CALL FOR ABSTRACTS](#)

**Poster presentations are very welcome until 18 September 2022!!.**

We **welcome sponsors and exhibitors** to be part of this great event with world-wide impact that continues regularly being celebrated every four years for over a quarter of a century. A **guide with different opportunities for sponsorship and exhibitors** can be found [here](#).

The **website** will be updated constantly with information provided from organizers and participants, so please be aware of the notifications regarding WCPT9 at: <https://wcpt9.org/>

If life were a city, it would be Madrid. If your interest is particle technology WCPT9 is the conference to participate in, **what else?**

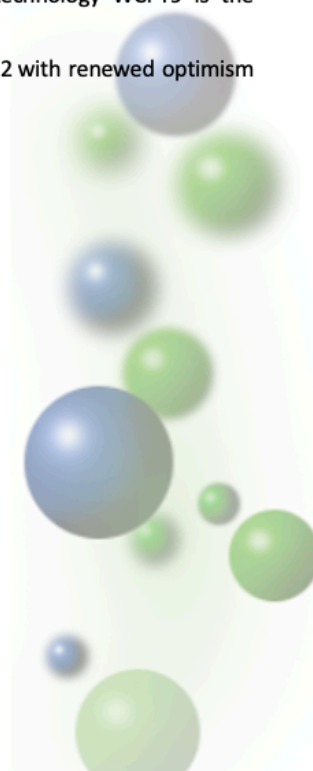
The organizers of WCPT9 look forward to welcoming you in Madrid 2022 with renewed optimism and ambition, in September 18-22, 2022. **JOIN US!!**

Carlos Negro, *Congress Chair*

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Álvaro Ramírez-Gómez, *Scientific Committee Chair*

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# Job Postings

## Engineer at Particulate Solid Research, Inc. (PSRI), Chicago, Illinois



PSRI is looking for an independent engineer / researcher who can carry out experiments and / or simulations to understand processes involving particulates. Please apply here, if you are interested: <https://psri.org/about/careers>

- **Key responsibilities:**
  - Design and perform experiments and/or simulations to qualify/quantify various fluidization phenomena
  - Design custom test rigs to support experimental work.
  - Collaborate with and communicate results to clients
  - Share research outputs in the form of presentations and reports
  - Contribute to the technology advancement and expansion
- **Competencies and Qualifications:**
  - Bachelor's or Ph.D. in Chemical or Mechanical Engineering or related
  - Background in particle technology and multiphase-flow preferred
  - Meticulous with data
  - Self-motivated and takes initiative
  - Independent and can work well with a team
  - Organized and detail oriented

PSRI is an international consortium of companies focused on advancing technology in multiphase flows with granular and granular-fluid systems. PSRI focuses on large-scale experiments, mathematical models, and the design and optimization of granular-fluid unit operations such as cyclones, pneumatic conveying, fluidized beds, ebullated beds, slurry beds, and circulating fluidized beds. We are the conduit from using new technology developed in the lab to applications in the field for many companies, including Ascend Performance Materials, BASF, BP, Chevron, Chevron Phillips, Dow Chemical, ExxonMobil, Flour, SABIC, IFPEN, Phillips 66, Siemens, Technip Energies, UOP Honeywell, etc. Our company has amassed a prolific amount of design data, technology, know-how, design criteria and models on all aspects of slurries, liquid injection, fluidization, entrainment, pneumatic conveying, attrition, erosion, distributors, standpipes, solids transfer, and circulating fluidized beds. In short, we are globally recognized as being the premiere leader in the research and development of granular fluid operations.

If you have any questions, please contact **Dr. Jia Chew** ([jia.chew@psri.org](mailto:jia.chew@psri.org))



**PEARL** Process Engineering  
Advanced Research Lab

**CRCT**  
Centre for Research in Computational Thermochemistry

## Post-doctoral position (Quebec, Canada)

### Process Engineering Advanced Research Lab (PEARL) and Centre for Research in Computational Thermochemistry (CRCT), Chemical Engineering Department, Polytechnique Montreal

**Keywords: Microwave and Conventional Heating Assisted Reactors, Modeling and simulation, CFD-DEM, Catalytic reaction, Powder technology, Fluidization**

#### Description of the position:

One post-doc position is immediately available for eighteen (18) months to work on pilot scale development of a catalytic reactor, in collaboration with PEARL and CRCT. The main goal is to employ modeling/simulation studies and answer uncertainties for commercialization of a new technology, which cannot/can hardly be answered by pilot scale experimental activities. The tasks may include:

- To develop a computational fluid dynamics (CFD)-discrete element method (DEM) model with species and energy transport equations to simulate a conventionally heated gas-solid fluidized bed reactor for catalytic dry reforming of methane (CDRM).
- To extend the developed CFD-DEM model to integrate Maxwell's equations of electromagnetic waves to simulate a microwave (MW) heated gas-solid fluidized bed reactor for CDRM.
- To evaluate performance of a commercial scale fluidized bed reactor for CDRM under conventional and MW heating conditions.

#### Desired Qualifications:

- PhD in chemical or mechanical engineering.
- Expert in reaction and/or fluidization engineering, and reactor modeling and/or reactor design.
- Experience working with CFD/CFD-DEM software, e.g., OpenFOAM, COMSOL Multiphysics, Ansys Fluent, MFiX, and/or Barracuda Virtual Reactor.
- Advanced degree involving model development and simulation.

- Experience or familiarity with different programming languages, e.g., Fortran, C, C++, Python.
- Background in application of non-conventional, e.g., microwave, heating method in development of chemical processes.
- Familiarity with detailed and basic design of pilot and industrial scale fluidized bed reactors.

The ideal candidate must be strongly motivated and be capable of working independently. Only candidates, who completed their PhD in the last five years are eligible to apply for this position.

**Application:**

Interested applicants should send:

- I. A cover letter of one page maximum.
- II. A complete curriculum vitae, including the list of journal articles and conference presentations/proceedings, as well as two contacts that could provide a reference.

Applications should be sent by email to [jaber.shabaniyan@polymtl.ca](mailto:jaber.shabaniyan@polymtl.ca), [jamal.chaouki@polymtl.ca](mailto:jamal.chaouki@polymtl.ca), and/or [jean-philippe.harvey@polymtl.ca](mailto:jean-philippe.harvey@polymtl.ca). Incomplete or non-conform applications and those sent on LinkedIn will not be evaluated. The applications will be evaluated as they arrive. Good candidates will be contacted to be virtually interviewed.

**About PEARL:**

<https://pearl.polymtl.ca/>

Process Engineering Advanced Research Lab (PEARL) reflects professor Jamal Chaouki's research group at Chemical Engineering Department of Polytechnique Montreal in Montreal, Quebec, Canada. PEARL is one of the strongest and advanced process engineering research labs in North America. Fluidization, multi-phase reactors, hydrodynamics, chemical reaction engineering (kinetics and catalysis), and innovative measurement techniques form PEARL's research mainstream, where cutting-edge projects for a wide range of industrial topics related to oil, gas, petrochemical, renewable energy resources, power plants, minerals, and batteries are carried out. PEARL's research projects are conducted in lab and pilot scales, where innovation is a cornerstone. Novel reactors for high temperature and pressure reactions, as well as novel measurement and analytical techniques have been developed at PEARL. Experimental work, modeling, and simulation are intensely practiced in PEARL.

**About CRCT:**

<https://crct.polymtl.ca/>

Centre for Research in Computational Thermochemistry (CRCT), co-directed by Prof. Jean-Philippe Harvey, at Polytechnique Montreal is among the world leaders in thermodynamic calculations and DFT simulations for alloys. CRCT includes expert researchers in alloy catalyst screening, alloy fabrication, multi-scale thermodynamics (including atomistic simulations and constrained Gibbs free energy minimization), process simulations, and material characterization. In addition to training engineers and scientists, the mandate of the CRCT is the research and/or development of



thermodynamic models and data for different types of phases and solutions techniques for estimating phase diagrams of multicomponent systems methods for representing and calculating complex chemical equilibria.

### About Polytechnique:

<https://www.polymtl.ca/>

Polytechnique Montreal promotes excellence, creativity, collaboration, integrity, respect, and openness. Recognized as one of Montreal's best employees, it offers excellent working conditions, including work-life balance and the well-being of its employees. At Polytechnique Montreal, we prepare a safe environment for all members of the university regardless of their gender, sexual orientation, race or religion. We are always welcome to hear about the feedback and to have open discussions.



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To continue receiving the PTF newsletters (3 issues per year) and stay current with particle technology events and news, please make sure to renew/ start your membership by either:

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