25.09.2024

Highlights

Dynamic Image Analysis

Interferometric Nanoparticle Tracking Analysis

Illegitimi non-Carborundum

Newsletter 5 (PCIG N5)

Welcome to the fifth edition of our newsletter! Join Us In Advancing Scientific Research

RSC INTEREST GROUP
PARTICLE CHARACTERISATION

Contents

Welcome to the fifth edition of our newsletter!

PREFACE

This newsletter aims to serve as a means of internal communication of useful information and strengthen the engagement among the group members. This quarter's newsletter (April - September 2024) consists of three main sections:

A. Research highlights, which presents emerging technologies in particle characterisation.

B. People focus, which reveals the motivation and sharing from different researcher members.

C. Update corner, which summarises new events, collaboration, and other opportunities.

Our current edition team includes Mel Disher, Merel Bout, Phil Jackson, Stefanos Mourdikoudis, Sayantan Das, Leon Xydias and Tien Quach. The graphic design has been performed by Leon Xydias. We would like to express great appreciation to the PCIG Committee for encouraging and advising us to issue the first edition of PCIG Newsletter. Many thanks for the contribution from the people who are willing to cooperate with us. We look forward to your collaboration in the next editions!

Welcome to the PCIG Newsletter,

where we network and collaborate for better particle technologies.

A. RESEARCH HIGHLIGHTS

A1: Dynamic Image Analysis

-PHIL JACKSON

Dynamic Image Analysis measures predominantly dry powders by taking 2 -D silhouette images and calculating equivalent spherical diameters. It also quantifies shape in terms of how closely particles resemble a define shape (e.g. a sphere or a needle).

Dynamic Image Analysis (DIA)

Dynamic Image Analysis (DIA) is gaining recognition as an analytical technique that can quantify particle shape as well as particle size. Much has been written about how particle size and particle size distribution can impact powder handling properties such as unwanted segregation of different powder chemistries in a mixed powder blend, or powder flow into a die for tablet pressing. But powder shape (e.g. sphericity or aspect ratio) can have an important role too.

Essentially, DIA typically involves tumbling dry powder particles over an edge and then capturing 2-D images as they cascade downwards (Figure 1). Imaging of powders in liquid suspension is also possible but won't be covered in this short article. The use of falling particles is critical as it means their orientation at the point of image collection is random. Static Image Analysis relates to imaging particles when they are resting against a surface. A drawback with static imaging is that certain particle shapes lead to a preferred orientation at rest giving a bias in terms of average dimensions.

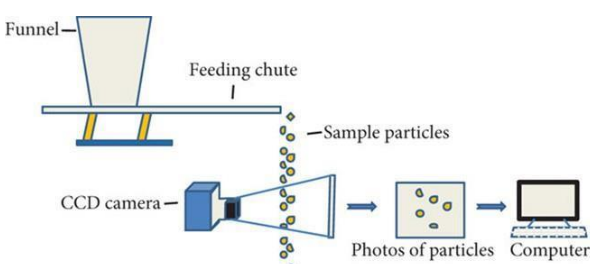

Figure 1: Set up of the dynamic image analyzer [1].

[1] Zhang Z, Lan X, Wen G, Long Q, Yang X. An Experimental Study on the Particle Size and Shape Distribution of Coal Drill Cuttings by Dynamic Image Analysis. Geofluids (2021); Article ID 5588248. https://doi.org/10.1155/2021/5588248

There are two important aspects to DIA:

1) Sample introduction

A good DIA system needs to employ compressed air and vibration to overcome mild agglomeration and so properly disperse particles prior to measurement.

Excessive use of compressed air however can lead to unwanted breakdown of (for example) granulates. Optimisation is therefore often required depending on the sample being analysed. There is also a need to control the feed rate for particles falling into the measurement zone. Fast feed rates are likely to lead to the imaging of overlapping particles as they tumble, generating data that suggest an excessively high particle size as well as providing false shape information.

2) Image capture

DIA systems typically feature use of a pulsed light source (with illumination times as low as nano-seconds) and a high speed camera (~500 frames per second). This leads to the capture of sharp 2-D images for each particle, despite their downward movement under gravity. To generate statistically robust particle size and shape distributions, tens of thousands (or ideally 1 million+) particle images need to be collected and analysed.

Once a particle image has been captured, pixels are used to define the particle shape and size (see figure 2 below, where square pixels are imposed on a particle shaded in blue). A decision is made by the software as to whether the majority of a given pixel covers the silhouette image of the particle or not. Whilst reliable particle size data can be gained down to ~1 micron, sensible shape data is typically realised at 20-30+ microns. This is because as particle size decreases, the size of the particle starts to approximate to the size of the pixel, resulting in a very crude definition of the 2D image (see again, the right-hand image in figure 2 below).

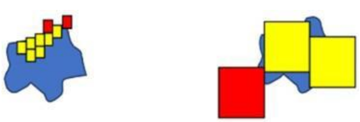

Figure 2: Impact of "pixel to particle image size" ratio on quality of defined shape. Yellow squares represent pixels selected to define a shape; red squares are pixels that are de-selected.

Having defined a 2D shape, software can calculate a sphere-equivalent diameter to provide particle size distribution information. Equally, the software can “score” a particle on a scale of 0 to 1 (1 being the best) for how closely it resembles a perfect sphere, square (1:1 aspect ratio) or cylinder etc. In figure 3 below, the sphericity of two powders is compared to provide the reader with a real-life application. The powders are both mixtures of fine non-oxide ceramics that have been subjected to Eirich mixer agitation to create a homogeneous spherical granulate. The red line in figure 3 relates to a sample granulated with a mixture of a solvent and a dissolved polymer. The blue line relates to granulation with a low MW polymer only.

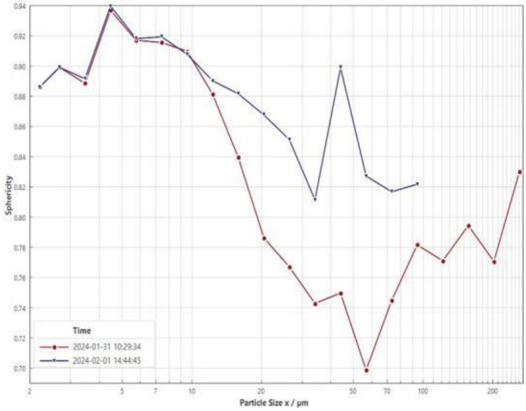

Figure 3: “Sphericity” graphs (1.0 = perfect sphericity) generated by DIA for two mixed ceramic powder blends. Red line = “wet” granulation; blue line = “Dry” Granulation

A. RESEARCH HIGHLIGHTS

A2: Interferometric Nanoparticle Tracking Analysis as a method to measure nanoparticle concentration - Review of a relevant research work

-STEFANOS MOURDIKOUDIS

Summary: Accurate ways to determine nanoparticle concentrations in liquid suspensions are in high demand, for example, in medical and food industries. Conventional methods remain unsatisfactory, especially for polydisperse samples with overlapping size ranges. Sandoghdar and co-workers used interferometric nanoparticle tracking analysis (iNTA) to determine the concentration of subpopulations in a polydisperse mixture in a quantitative manner and without the need of a calibration sample [1]. This was achieved by counting the number of trajectories that cross the focal plane. The authors evaluated their method on both monodisperse samples and mixtures of known concentrations. Furthermore, they assessed the concentration of SARS-CoV-2 in supernatant samples obtained from infected cells.

Background-Objectives: Measurements of nanoparticle concentrations are significant in several domains because they provide information on the dosage and purity of a given specimen.

Interferometric Nanoparticle Tracking Analysis (iNTA)

Such data are crucial in drug characterization and administration, potential toxicity in the food industry, or in environmental domains, where concentrations of undesired entities such as nanoplastics in water need to be identified. Commonly used techniques for nanoparticle characterization are electron microscopy (EM) and atomic force microscopy (AFM).

While these methods achieve excellent size determination, they can generally only yield relative and not absolute concentration values unless the volume of the liquid is controlled to a high extent. Other broadly used techniques for measuring nanoparticle concentration include dynamic light scattering (DLS), nanoparticle tracking analysis (NTA), tunable resistive pulse sensing (TRPS), and nanoparticle flow cytometry (nFCM).

However, these techniques experience some limitations: DLS evaluates particle size by analyzing temporal correlations in the light scattered by diffusing particles, whereby the concentration should be sufficiently low to prevent multiple scatterings. Nanoparticle concentrations between 10^8 and 10^{12} particles/ mL can be measured in DLS, but the exact range depends on particle size and material. Since the concentration is extracted from the total amount of the scattered light, reference samples are needed for accurate concentration measurements. In NTA, particles in the field of view (FOV) or their trajectories are counted, and this number is converted to a concentration value by using a predetermined factor. The measured values range between 10^7 and 10^9 particles/ mL.

The concentration needs to be quite low to avoid swarming, where numerous nanoparticles enter the beam at the same time, but it has to be sufficiently high to prevent the background counts from dominating the measurement. None of the above-mentioned techniques offers an accurate concentration estimate for populations in polydisperse mixtures, especially when the refractive index is unknown or the size ranges overlap. The authors have already introduced interferometric nanoparticle tracking analysis (iNTA), which performs NTA using interferometric scattering (iSCAT) microscopy [2-4]. The method features superior performance for determining size and refractive index of nanoparticles and is able to resolve different subpopulations. In the present highlighted work, the authors demonstrated a strategy for performing absolute concentration measurements without the need for a calibration sample. They discuss the theoretical and practical limits of concentration measurements in iNTA and apply the method to supernatants of infected cells, where they specify the concentration change of SARS-CoV-2 virions over time.

Measurement Strategy: A straightforward approach to the measurement of particle concentrations is to count particles or their trajectories within the FOV of a given volume, depicted schematically as a box in Figure 1a. In practice, however, the volume that contributes to the optical signal is not as clearly defined. For example, in the case of a FOV defined by a focused Gaussian beam, the extent of the boundaries is not sharp. As a result, whether a particle is counted or not depends strongly on its position and scattering cross section as well as the sensitivity of the setup. This is illustrated by the highlighted section in Figure 1b. These subtleties call for a particularly careful calibration of the setup on well-characterised samples.

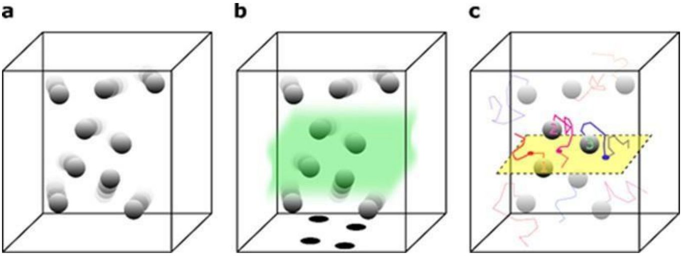

Figure 1. (a) A volume of liquid containing a certain concentration of nanoparticles. (b) The highlighted region suggests an effective volume that contributes to the optical signal. (c) The yellow plane depicts the focal plane. When particles cross the focal plane, their contrast reverses.

In NTA, which uses a dark-field microscope arrangement, the extent of the detection volume associated with the z-axis (e.g. see Figure 1c) is poorly defined and varies by a large degree with the particle scattering cross section, i.e., a larger particle remains visible further away from the focal plane compared to a smaller particle. This leads to an overestimation of the concentration for larger particles. To tackle this problem, the authors proposed to deduce the concentration by counting the trajectories of particles that cross the focal plane. As shown below, this number depends only weakly on the z-extent of the detection volume (Figure 1c). Implementing this strategy in NTA is cumbersome because the slow change of the point spread function (PSF) along the axial direction makes it hard to determine the focal plane of a dark-field microscope with big precision. In iSCAT microscopy, the central lobe of the PSF almost reaches the diffraction limit and the signal undergoes a contrast inversion between maximally bright and maximally dark when the particle crosses the focal plane. This feature permits to perform a robust measurement of the nanoparticle concentration. For technical-experimental details and protocols, please refer to [1]. Simulations: In order to relate the number of detected trajectories to an absolute particle concentration, the authors simulated a single particle with a given diffusion constant in a $10 \times 10 \times 10\ \mu m^3$ box, corresponding to a particle concentration of 109 particles/mL. Particle diffusion was simulated for 0.1–100 s with a random starting position at a frame rate of 10 kHz. Simulations were repeated 30,000 times. The average number of trajectories longer than a given threshold per video was extracted to be related to particle concentration.

Benchmarking-Results: To benchmark their methodology, the authors studied different dilutions of monodisperse particles that were characterized by the manufacturer. Particles with a density much larger than water (e.g., gold or silica nanoparticles) may bias concentration measurements due to sedimentation. To avoid this systematic issue in their benchmarking measurements, they chose polystyrene (density of 1.05 g/cm^3). The researchers measured 13 dilutions of NIST-certified 40, 60, and 100 nm polystyrene spheres (PS) for 5 min twice (300 videos each time). In every case, they extracted the number of detected trajectories that contained more than 100 localizations. Following this, the authors measured the concentrations of populations in a mixture of particles with overlapping sizes. They used 100 nm PS and silica beads whereby the latter had a broad size distribution. Finally, the authors evaluated the concentration of SARS-CoV-2 in supernatant samples obtained from infected cells. For more details and results, please see article under discussion, Ref 1 below.

Main findings-Conclusions: It was demonstrated that iNTA is a useful tool to characterise particle size and refractive index but also for the quantitative assessment of particle concentration. Concentrations of different populations in polydisperse samples were reliably determined, even when sizes overlapped. More specifically, the authors showed an approach to measure concentration without requiring tedious calibration steps. They found that the method under discussion performed well for concentrations up to 10^{11}particles/mL.

The lower limit was around 10^8particles/mL for a 5 min measurement, but it could be ameliorated by prolonging the measurement time. The authors showcased the power of their technique by determining the concentration of virus particles and residual extracellular vesicles obtained from cells infected with SARS-CoV-2.

References

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[4] K. Lindfors, T. Kalkbrenner, P. Stoller, V. Sandoghar. Detection and spectroscopy of gold nanoparticles using supercontinuum white light confocal microscopy. Phys. Rev. Lett., 2024, 93, 037401.

B. PEOPLE FOCUS

B1. Get to know

We can understand the research interest and career pathways from our PCIG members. We will start with an overview of two of the Committee members please contact us to share your background and experience in future new

Han Wu

"Dr. Han Wu has won several awards including UCL Technical Professional of the year in research 2024 and Papin prize finalist"

Han is the Research Lab Manager at UCL, managing key research facilities including SAXS, DLS, BET, TGA and IR etc. Han joined UCL as a post-doctoral research associate (PDRA) in 2010 to work on polymorphic crystallisation. Her recent SAXS study on amorphous ice was published in Science. Han has served as a guest editor for Current Organic Chemistry, the treasurer of PCIG, and an active member of the BSI and ISO committees, contributing to standards in SAXS and porosity. Han has won several awards including UCL Technical Professional of the year in research 2024 and Papin prize finalist.

Mel Disher

"I work in a consultative sales role where we advise both academia and industry on techniques that would best support their research."

My degree was obtained from the University of Lincoln in bio-veterinary science, and I went straight into a role working as a production scientist in regenerative medicine at a GMP facility culturing Chondrocytes and Mesenchymal stem cells for autologous implantation. From this role I moved into a contract research laboratory (Quotient Bioresearch) where I worked on environmental fate metabolism studies using radio-labelled compounds.

These studies were regulatory studies, completed according to OECD guidelines. During my time working in environmental fate metabolism, I characterised metabolites of parent compounds using Radio-HPLC. I remained at this company for over a decade, during this time Quotient Bioresearch became Pharmaron UK, and as the company changed names I also changed departments and spent the next 6 years working on In Vitro ADME DPMK studies. This role took me back to my biology background where I conducted the full range industry standard DMPK studies and I also set up a laboratory for xenograft studies.

I changed careers to work as a Product Specialist for Analytik Ltd in 2022, I specialised in the particle characterisation department with a portfolio of instruments ranging from nanoparticle tracking instruments to differential centrifugal sedimentation. I work in a consultative sales role where we advise both academia and industry on techniques that would best support their research. This role allows me to travel throughout the UK and Ireland conducting educational demo tours, particle characterisation webinars and attending networking events. I joined the RSC in 2022 and shortly after joined the Particle characterisation group and subsequently the PCIG committee. On the committee I work primarily with the events team and the newsletter team.

B2. "Conversation with our inspirers"

WHY JOIN US?

- We love to understand your technical and social experiences, especiall stories throughout the learning and working journey.
- We would like to motivate more students - researchers to follow their passio in particle science.
- We believe a single effort and contribution to help make our world bet recognised and spread out.

HOW?

If you are interested in participating, please contact us for more details!

Our inspirer ... Sayantan Das

Interviewed by Tien Thuy Quach

Could you please introduce yourself in general and in the scientific community?

Hello, I am Sayantan Das, currently an Assistant Professor and one of the Department's Program Coordinators at Texas A&M University-San Antonio. My research focuses on synthesising nanoparticles and their composites, emphasising using bottom-up self-assembly to create hierarchical structures on surfaces. These structures aim to enhance or replace traditional, costly manufacturing techniques. Specifically, I investigate the following areas: renewable and rapid nanocomposite synthesis, tuning and retaining nanoparticle physical properties in composites, and developing processes for hierarchical structuring of materials across multiple length scales on surfaces.

What discoveries have led up to your current work?

My Ph.D. work focused on the evolving mechanisms of self-assembly in mixed nanoparticles and nanocomposites on various surfaces. This research was motivated by the need to replace expensive top-down lithographic processes with novel structures created through bottom-up self-assembly. I aimed to answer the question: “How can self-assembly patterns be directed to create hierarchical structures by manipulating processing conditions and nanoparticle properties?” This research enables the development of industrially scalable processes, such as convective self-assembly and Sonoluminescence/ Sonochemistry, to create novel hierarchical structures on surfaces. Examples include spontaneous deposition of size-segregated particles (Fig. 1 i) for multi-wavelength anti-reflective solar cells, defect reduction in thin films (Fig. 1 ii) for high-quality surfaces, and the synthesis of nanocomposites for rapid humidity sensors, water sensors, and electrochemical sensors (Fig. 1 iii).

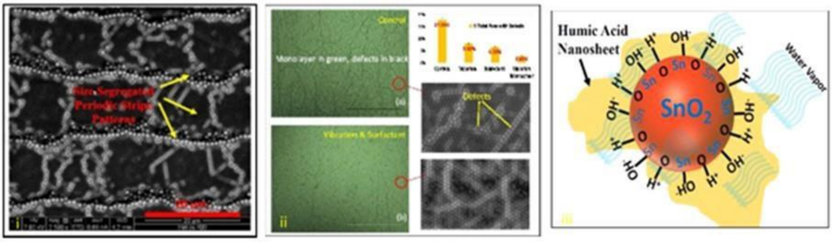

Figure 1i: Spontaneous and controlled stripe pattern formation using bottom up self-assembly of mixed nanoparticles. Figure 1ii: 86% reduction of defects in thin films by design of process during spontaneous deposition of nanoparticles. Figure 1iii: Schematic of Synthetic Humic acid and Tin oxide nanocomposite for electrochemical sensing. Reprinting from Das et al.’s publication(s).

What are the possible real-world applications? Why is your research necessary?

Two of my most significant research interests from my graduate work are: (1) responsive functional nano-based materials and (2) their interaction with the processing conditions of bottom-up self-assembly techniques. I am particularly interested in functional materials that respond chemically to thermal, mechanical, or specific chemical stimuli and actuate in response to electronic stimulation. Advancements in these areas are crucial for solar cells, water purification, and chemical detection. Further development of these materials using nanocomposites could expand their potential applications in agriculture, renewable energy optimization, and semiconductor manufacturing.

A vital aspect of this research is identifying new material options, developing affordable manufacturing techniques, and scaling these processes. Bottom-up techniques, which typically require fewer resources than lithography or chemical vapor deposition, are central to this work.

What kind of response have you received regarding your research/ findings?

My discoveries have demonstrated new spontaneous surface patterns that can be further controlled through processing conditions. Specifically, the spontaneous segregated surface patterns from mixtures of nanoparticles using a bottom-up approach (Fig. 1i) were unprecedented in the literature, earning a Key Article Award from Advances in Engineering. This finding has significant implications: current top-down lithographic processing methods used in industry are costly and time-consuming, hindering growth. The ability to scale these patterns using a bottom-up process offers a more efficient alternative, potentially benefiting the industrial sector. As a post-doctoral researcher, I developed new nanocomposite-based electrochemical sensors for rapid detection of humidity and hydrogen sulfide in solution and polymer-clay composite based gas barrier films, both of which have been published. The work on gas barrier films led to the creation of a startup company, Nabaco, which uses a similar but more scalable process to coat fruit surfaces, extending shelf life and reducing food waste.

Recently, I secured a collaborative grant with five other universities in Texas to improve the response of shear-thickening materials for injury prevention.

What is the coolest thing about your work/ research?

For me, the coolest aspect is the ability to create a wide range of things using simple methods like bottom-up self-assembly or sonochemical processes. For example, I can engineer an in-situ cooling device within an ultrasonic bath and use basic chemicals to conduct various thermal, mechanical, or chemical experiments. The same applies to bottom-up convective assembly processes, which involve a simple setup with a linear motor and a blade dragged at an angle. While the experimentation is challenging, I find it rewarding.

B3. Inspiring stories

Do not hesitate to share your stories to motivate other researchers and students. You can write about the people, the events that motivated you throughout your learning, working and research (either the good or the bad things happened). We look forward to hearing from you.

Where are our torches?

-Tien Thuy Quach

Louis Pasteur said, "Science knows no country, because knowledge belongs to humanity, and is the torch which illuminates the world", and it was my pleasure to learn a range of lessons about how "science is the torch which illuminates the world" when participating in the IUPAC-CHAINS 2023 conference. I believe you may have or already be on the way to find your own "science torches" that could enhance your current research and future career aspirations.

I am happy to share my small experience from Vietnam and UK when becoming one of the Young Observers. It's wonderful to know dissimilar researchers and students at the World Forum and we could explore the Hague, Netherlands together. Initially, the 52nd IUPAC General Assembly and 49th IUPAC World Chemistry Congress combined with the 11th edition of CHAINS (18-25th August 2023) can help strengthen my knowledge and skills. I really admired the works from Professor Molly Stevens who shared about "Healing the body and detecting diseases earlier through new materials" at the Opening Ceremony. I was very impressed by other award sessions and motivated talks, and big congratulations to all prize winners and speakers! It was fantastic to know more about many on-going projects and connect with great people from different IUPAC Meetings and the RSC Reception Event. For example, I could exchange ideas with different inspirational members from the IUPAC - Polymer Division and Analytical Chemistry Division. Our recent launch of the "Polymer Video Competition" has potential to enhance our shared passion for polymer science and provides an opportunity to showcase your student's collective creativity and knowledge. Another on-going project "A review of current status of analytical chemistry education" also received lots of positive feedback and discussion from international candidates.

Besides, I could join the series of young programmes that was included in this Congress for the first time to elevate the early-career researchers, postgraduates, and undergraduates. Particularly, I received a range of advice not only in conducting excellent research, pursuing dream jobs but also to having work-life balance from *"Road to Success I"* sessions presented by Dr. Rehana Sidat – GSK, Francesca Novara – Wiley-VCH, Laura McConnell – Bayer, and Lene Hviid – Shell. Moreover, I was happy to meet diverse National Delegates and Young Observers via the International Younger Chemists Network (IYCN), European Young Chemists' Network (EYCN), and Jong KNCV | Young Royal Netherlands Chemical Society.

It was amazing to attend informative scientific talks and IUPAC meetings in person. Along with joining different parallel and focus sessions, I was happy to share my knowledge and experience with diverse group and/or individual participants. For example, I had great discussions with a range of researchers and students when presenting my scientific poster *"Surface and interface study of 3D-printed biopharmaceutical products"* at the booth Health 78, and my community poster "*Making the most of my second-year PhD project during the Covid-19 pandemic (2020-2021)"* at the Young Programme Exhibition.

Additionally, I could join several social activities during and after the conference sessions. It would help me to explore more about the cultures and people in the Netherlands which is certainly different from the Southeast Asia area where my hometown is based. For instance, it was a perfect time to use one free afternoon for swimming at the beach as well as trying traditional cuisines and my Vietnamese noodles in the city centre could be one of good options.

Furthermore, I was glad to not only visit the *Organisation for the Prohibition of Chemical Weapons (OPCW)* – recipient of the 2013 Nobel Peace Prize for its extensive efforts to rid the world of chemical weapons, but also attended the presentation "Women in chemical security and safety". My happiness multiplied when I could understand different perspectives about women's roles in the Chemical Engineering areas because I have put great effort into enhancing Equality, Diversity, and Inclusion in my previous and current workplaces, particularly my focus on empowering women scientists in their professional and personal development.

I believe in-person discussion and networking will open great opportunities to build up new collaborations and enhance practical knowledge exchange activities. I could learn new things during various meetings, workshops, and exhibitions, and I also had fun exploring several beautiful landscapes in this welcoming city. I am very thankful for all motivators and facilitators to fulfil my role as a Young Observer and Presenter here, and I hope each student - researcher could stay in touch so we could learn, work, and grow together in the future. All the best for your research, work, and study, and please feel free to share with me and other colleagues your memorable events!

As it came to an end, I would like to summarise my top seven-lessons learned from the IUPAC|CHAINS2023:

1. Taking certain time and effort for fruitful collaboration.
2. Being open for unexpected things to happen.
3. Speaking out and finding routes to work together despite many differences or preferences among us.
4. Never ever stop learning and checking our happy states throughout the journey.
5. Remembering that talents spread out and talents with great kindness are highly recommended.
6. Gaining more great suggestions and ideas throughout official and unofficial discussions.
7. Continuing to work smart and fight hard for the things you believe in, give and receive.

I would like to thank the Royal Microscopical Society, the Royal Society of Chemistry, and the University of Nottingham for providing supports and funding which facilitated my participation at this event. Finally, I love to share the next IUPAC General Assembly will take place in Malaysia, July 2025 - more details at https://iupac.org/event/iupac-world-chemistry-congress-2025/

Illegitimi non-Carborundum

-Sayantan Das

In this conversation piece, Gary W. Beall [Google Scholar] is one of the top 2% of researchers chosen by Stanford in 2023. He shares his life motto of **Illegitimi non-Carborundum**: "Don't let the B**ards grind you down." Through his mentionable journey from 1970 to the present (2024) and his impact in the field of nanoparticles and nanocomposites before "nano" became popular, we delve into his journey of nanoparticles and nanocomposites research: notable projects, characterization tools he utilized, and the challenges he conquered during. Finally, he shares his post- retirement work on eliminating food waste using nanocomposite-based coatings.

I encourage our readers to share which aspect of Gary's career you want to know more about in the future newsletter via the PCIG social media/ Comment Section. We appreciate your feedback!

Biography: One of the factors that is quite different for me is that I'm a first-generation college graduate in my family. In undergraduate school, I had to work my way through it. So, I worked full time and went to school full time. I probably averaged 4.5 hours of sleep a night for four years. **But when I got to graduate school, that was such an advantage because now I had a research stipend**. I didn't have to work. I could concentrate on school. So, for me, graduate school was a lark. I had a great time, and I was very productive. I finished a master's and PhD in 2.5 years. The torture of four years of working through school paid off when I went to graduate school.

Graduate School Experience: My experience differed significantly from that of many graduate school students. I also had a professor who was, I guess, reasonably irascible, not a very kind gentleman, adamant. And he would always give his students about four projects to work on. And I didn't know this till later, but **one** of them he knew would pay off, and you could write up for your dissertation. The **second** one was a little harder. The **third** one is a little harder, and he considers the fourth one impossible. Of course, he never told us, you know, what classification or any of those things. And fortunately, I lucked into **solving the impossible problem**. And that's one of the reasons I got through so quickly.

More on the impossible Project and Characterization:

Keep in mind that this was before they were called nanoparticles; this was in 1972. Back then, they were referred to as colloidal materials. But they're nanoparticles. That was one of the problems in all the projects my advisor gave his students, which involved poorly crystalline nanoparticles and were very difficult to characterize. We also had several electron microscopes in our department. So, we did a lot of our work with electron microscopes. We did a lot of X-ray diffraction. And the project he gave me that was impossible was a project on transition metal cyanides. Those materials are very insoluble and make it challenging to get single crystals. The only data we had was from electron diffraction patterns or powder X-ray diffraction, and there was a significant dispute between several groups about the fundamental structure of these materials. Well, I figured out a way to grow single crystals. **And it was** so simple that it's almost ridiculous. I took the two reactants and put them in test tubes. Then, I put a water bridge across them in an oven for about three months. And let the reactants diffuse very slowly up that Water bridge. After two to three months, I went in there and saw these sparkly crystals growing where the front of reactants was meeting. I harvested those crystals and could do a single crystal structure of those compounds and solve that dispute that had been going on for 30 years between these various groups. Unfortunately, my advisor was wrong. The other group was closer to the correct structure, but we came up with a model that was even different from theirs. But that, you know, was a project that I saw was impossible but so simple.

Inspiration from Nature: I came up with the idea of a water bridge because of the simple concept that diffusion of reactants without any convective transport is very slow- so my thought was to grow crystals of insoluble materials, we need a prolonged process of diffusion, and part of it was looking back at Nature, if you look at caves where you see all these humongous crystal have grown, over millions of years because of this very dilute solution that is slowly dripping to grow- so it was kind of analogous to what Nature does that made me think simple!

Interesting Project Beyond Academia and its Artifacts: When I was at Oakridge, I was assigned the problem of understanding how the actinides from the weapons program and nuclear fuel will behave in the environment. Thus, we started looking at the aqueous behaviour of these actinides in solutions that would usually occur in Nature. It is very different than what you would do in a pure water solution. We first found that these things did not behave in the traditional pure water system way. And they interacted with minerals in entirely different ways than what would have been predicted. The surface interactions of those actinides were things like phosphate on the surface of a mineral or sulfide on the surface of the mineral absorbed much more strongly, which was very different than what we would have predicted from a typical aqueous solution. And it was an uphill battle because of the work done in the Manhattan Project. They thought they had this down; they understood the aqueous chemistry of these things. Suddenly, they started exposing themselves to environmental conditions and behaved entirely differently. So, we were tasked to develop new theories to explain these things. And we had a lot of fun doing that -it was complicated because all of these things are radioactive. But that made it enjoyable. I guess the one thing that we learned was that the Colloidal transport of things like plutonium in the environment is an essential mechanism. Usually, plutonium would stick to everything that comes along. But if you have a colloid in this environment, they can be carried along much further than expected- that was a big surprise and the thing that ties them together is colloids or nanoparticles.

The Rise of the Nanoparticle Era: Around 1992, I worked as technical director at American Colloid in Chicago. And I got a call from DuPont Research. And the guy said, do you know about this nanocomposite work that Toyota has done? I said this is not something I'm familiar with. Could you send me the paper? And I'll read it, and then we can talk later. Well, I did that. What I learned was neat because they've taken nanoparticles and put them in the polymer, increasing properties tremendously. Ken Sharp was the guy at DuPont, and I called, and he said, Well, what's the problem? He says, Well, we can't reproduce it; we took this K10 Clay, and it won't do what Toyota says. I said, Well, there's the problem. K10 is a dehydrated dehydroxylated clay. It's not going to disperse like the clay they use. Then, we started this collaboration. So, that launched me into the whole nano composite business.

Consequently, we wrote the first book in 2001. On polymer clay nanocomposites And that has been a very successful book. But that call from Dupont coloured my career from 1992. Until now, I've always been dealing with nanocomposites of some type. But it all came because DuPont researchers, their central resource, called up a clay expert to determine what was happening. As you know that almost happenstance turned out to direct my work for more than 20 years. Because of my interest, I also came up with this theory about constrained polymer as the reason for many current properties. And when I first presented that, it was resisted heavily. But now I think most people would agree that is the operative theory to explain a lot of stuff that goes on in Nanocomposites, like replacing the simple tortuous path model, but at first, there's tremendous resistance. Despite the resistance, there was also recognition that this was a new way to look at things.

Current Endeavors: Nabaco results from our research on self-assembling nanocomposites. So, we discovered that if you pick the right clay and the suitable polymer, they will self-assemble into this highly structured coating. And so, the whole company is now based on that technology, which again is a nanocomposite; it is just self-assembled naturally like abalone nacre.

Instead of me concluding for you(readers) I will really love to know: do you think Gary Beall's story resonates with his Life's motto of "**Illegitimi non-Carborundum**".
Please share your thoughts via emailing sdas@tamusa.edu.

C1. Report Committee Meeting 24/6

-Mel Disher

On the 24th June the Particle Characterisation Interest Group committee held our first in-person committee meeting of the year. While our meeting was conducted in a hybrid format we had an impressive in-person turn out, and met some new members of the committee in person for the first time.

We were hosted by Malvern Panalytical at their site on the edge of the picturesque Malvern Hills. We arrived with plenty of time to enjoy lunch together before being treated to a site tour by our host and committee member Stephen Ward-Smith. During the tour, we were kindly shown some of the instruments in the particle characterisation portfolio Malvern Panalytical has to offer. Our tour included introductions to the the Mastersizer, Morphologi, Omnisec and Zetasizer from Jenny Burt, Ben Lynch, Jess Watts, Nellie Chourmouziadi Laleni, who took time out of their schedules to talk us through the instrument's capabilities.

Our committee meeting took place after our site tour where we had lots of upcoming events and opportunities to discuss including our training meeting for Microstructure analysis using BET surface area, pore size, chemisorption, gas/vapor sorption and associated techniques, hosted at the home of the Royal Society of Chemistry at Burlington House in London on 2nd July. Other topics for discussion were the FORGE 2024 event that our committee members are organising on the 12 – 13th of November at the University of Leeds, and the Particle Size Analysis book which our committee members are currently writing. With a jam-packed agenda, the time flew by and the group either departed for their homeward travel or stayed locally for the evening to enjoy a meal and evening entertainment.

Our committee meetings are always an enjoyable event but more so when we can meet up in person, we look forward to the next opportunity for this at FORGE 2024 in November.

C2. Other upcoming events of interest

-Merel Bout

- **UK-based events**

TheFORGE2024, event organised by the PCIG 12 – 13 November 2024 Leeds

More info at: https://www.rsc.org/events/detail/79646/the-forge-2024

- **International events:**

Particle-based materials symposium 2024

23–24 October 2024 Universität Bremen, Germany

More info at: https://www.uni-bremen.de/mapex/pbm2024

Solid State Chemistry (SSC2024)

08–13 October 2024 Ústí and Labem, Czech Republic

More info at: https://www.ssc-conference.com/

Polymers 2024 (XIII Slovak-Czech Conference)

21 – 25 October 2024 Stara Lesna, Slovakia

More info at: https://polymer.sav.sk/polymery2024/index.php/polymers-2024/

Materials Science & Nanotechnology (5th International Conference)

21 – 23 October 2024 & online: 24 - 25 October 2024 Athens, Greece

More info at: https://materialsconference.yuktan.com/

Nanocon (16th International Conference on Nanomaterials)

16 – 18 October 2024 Brno, Czech Republic

More info at: https://www.nanocon.eu/en/

POLYMAT-2024

20 – 25 October 2024

Autonomous National University of Mexico (UNAM) More info at: https://www.iim.unam.mx/polymat/

The PCIG are always happy to hear about up-and-coming events that our members are interested in. If you have any suggestions for events to be included in our newsletters, please contact us and we will include these in our next edition.

CONTACT US

Visit our website for further information: https://www.rsc.org/membership-and-community/connect-with-others/through-interests/interest-groups/particlecharacterisation/

Do you have any questions, feedback or are you willing to contribute as a collaborative writer? Please email the RSC-PCIG Particle Newsletter Team via: Particlenewsletter@gmail.com and we will get back to you.