

Interview with Jawwad A. Darr of University College London, a speaker at the NanoMaterials2010 conference

Q: What is the remit of the Clean Materials Group at UCL?

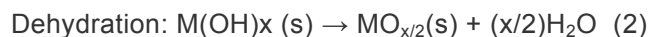
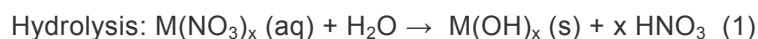
This can be summed up by our mission statement which reads "We aim to exploit the advantages afforded by clean technologies to give either novel materials, better control of processes or control of materials properties." In 2000, the Clean Materials Technology Group was set up to encourage multidisciplinary research in emerging technologies such as 'Supercritical Fluids' (mainly carbon dioxide and water) for the clean and controlled manufacture of biomedical related materials and nanoceramics. Since its inception, the research has expanded to other areas from manufacture of advanced composites to nanoparticle electronic / optical materials.

In 2007, the group moved to UCL Chemistry dept where the emphasis was centred more on the use of supercritical water flow processes for controlled nanoparticle synthesis. Specifically, the groups has been leading the development of such process and evaluating nanoceramics made via these processes.

Q: Could you tell us about the UCL hydrothermal nanoparticles formation process?

The process at UCL is known as CHFS (continuous hydrothermal flow synthesis) and I was the first researcher in the UK to develop such a process over 10 years ago. Currently, there are less than 10 active research groups in the world who are working on a similar technology. Continuous hydrothermal flow synthesis (chfs) reactors work as follows; water is heated (in flow) to above 400 C (at very high pressure) and this hot flow is then mixed with a metal salt source (normally a solution) in a special engineered mixer which causes nanoparticles to precipitate. The reaction and formation of nanoparticles happens in less than a second and by varying the process parameters in CHFS, it is possible to alter size and shape of nanoparticles to a degree. Differing oxidation states and even metastable inorganic oxides can be readily obtained.

The formation of nanoparticles occurs in less than a second via a two-step mechanism; a hydrolysis followed by rapid dehydration of the metal salt.



More recently, the my group reported a significant development of the chfs reactor known as a high-throughput continuous hydrothermal (HiTCH) flow synthesis reactor which can produce many compositionally unique metal oxide nanomaterials in a matter of hours. Using a manual approach the HiTCH reactor was able to produce an entire 66 sample library of $\text{Ce}_x\text{Zr}_y\text{Y}_z\text{O}_2$ in 12 hours. This productivity for nanomaterials is unknown in the chemical literature and is of use for industry who are keen to screen areas of compositional space in nanoceramic oxides. A fully autoamted HitCH system has also been developed known as RAMSI (rapid automated materials synthesis instrument). RAMSI not only makes samples, but it also cleans and prints them via a fully automated system.

More recently, a pilot plant (x20 scale up) has also been developed via EPSRC funding and this is being evaluated for commercial development. The idea here is to develop materials on the lab-scale and then for the most important materials they can be scaled up and evaluated for application.



Importantly, a x20 scale up process is not x20 bigger (as the case for flow), because it's a flow reactor and so it is ca. x5 bigger in terms of footprint and the flow rates are considerably higher.

Thus a pilot plant for hydrothermal flow contains relatively small volumes at any time so it offers a safety advantage. The other advantages of flow reactors are that the product can be monitored in real time, i.e. as it exits the reactors.

Q: In what ways is this different from current techniques?

The basic CHFS method is a flow system. The vast majority of current industry and academics use "hydrothermal batch" reactors in which the reagents are cooked under pressure for long periods in superheated water and this can be done on a large scale up to tonne scale. However, the major problems with these batch hydrothermal processes is that (i) they do not offer a great deal of control over nanoparticles properties as they are slow (reactions take hours), (ii) scale up is quite dangerous as large volumes are involved, (iii) temperatures for reactions are restricted to under 300C which means certain materials may not be accessible and (iv) the particle in CHFS are made in water and so no nasty organic solvents are used and the nanoparticles are always safely contained in a liquid and not airborne (important safety feature). CHFS is essentially doing similar reactions to batch in a flowing rather than static system.

Other techniques such as flame and plasma processes have been scaled up for the production of nanoparticles, however, such processes have considerable problems. Plasma processes are excellent for the production for carbides and metal nanoparticles, however, the extremely high temperatures inside a plasma means that the nanoparticles are generally highly fused together and not made in a well defined manner. The high temperatures tend to lead to low surface areas and poor control over particle size. Similarly, flame processes are relatively high temperatures and both processes make nanoparticles in the gas phase which is a real concern in terms of product collection and exposure for operators of such processes. Airborne nanoparticles offer unknown hazards to health and such processes would be better suited if the nanoparticles can remain dispersed in a fluid where they can be safely contained. In contrast the CHFS process at UCL makes nanoparticles in water and they remain in water until they are needed. CHFS made nanoparticles can readily thus be formulated into printable inks and will remain in a safe liquid form throughout.

Q: What advantages does this bring and what are your meters for a successful pilot?

As discussed above, the scale up process allows at least 5kg of nanoparticles to be made in water (a green solvent). The important thing that our research has shown is that nanoparticles made on the laboratory scale (100g a day) are almost identical in quality to that obtained on the x20 scale up pilot plant.

Prior to developing the pilot plant, the following objectives were set as measures of success

- (i) to be able to model the particle production in the pilot plant process
- (ii) to develop new IP pertaining to the mixer that is used for the point where nanoparticles are formed (this is the most vital part of the process)
- (iii) to design and develop a pilot plant and work with industry and other collaborators in evaluating the performance of materials. Compare these materials to those made via more conventional methods
- (iv) compare the nanomaterials made on the smaller lab scale and the pilot plant via measurement and evaluation from end users
- (v) develop new inks and products which could be sold commercially

Q: Who are the potential end users?

- Industry or academia who are developing devices or products which could be improved by being nanoceramics or well dispersed in water or having metastable compositions
- These include any nanomaterials which are obtained as oxides and require a high surface area or size and shape control, or to be well dispersed in a liquid medium such as an ink.

Q: What stage of development is your project at? What are the next stages of development?

The combinatorial facilities for making nanoceramics and the pilot plant are fully operational and we are seeking to scale up key nanomaterials which have been developed on the lab scale. We have a number of nanoceramics which are currently being formulated by end users and depending on these trials; we anticipate commercial orders to follow. These nanoproductions are essentially high value and relatively low volume products (high value manufacturing) for which the pilot plant could readily handle in terms of production capacity if it's needed.

As we move forward, if the case can be made for full scale hydrothermal flow reactors (100 kg a day or so), then our research and UCL's IP will put us in a strong position to undertake such a venture. Indeed, the pilot plant was developed as a scaled down version of a full industrial size reactor so we envisage this will be straightforward. This will be a future stage of development which will only be done if there is a strong commercial case.

The next stage of development is really about getting acceptance from industry and end users in evaluating the pros and cons of this technology compared to other methods (we believe it offers significant advantages over more well established methods). Fortunately as we have a reasonable size research group, UCL have been able to publish a number of papers on applications including papers in photoelectrochemical water splitting, catalysis, solid oxide fuel cell nanomaterials, automotive catalysts, magnetic materials, microwave dielectrics, biomaterials, phosphors, etc. The recent research has fed directly into the pilot plant

development and so we have a number of exciting nanomaterials under development which we have been requested by our industrial partners.

Q: Why did you decide to speak at the NanoMaterials conference and what are your aims from attending?

- Attract potential end users for high throughput facilities or for scale up production of nanoceramics and nanoparticles inks.
- Find investors to commercialise the technology, particularly the pilot plant
- Find licensing opportunities for the IP we have developed in nanoceramics production
- Find collaborators, customers and end users who wish to access our nanoceramics for research and applications.
- Apply for joint TSB and FP7 funding with industry partners towards the development of nanoparticles and nano-products.

