Nanomaterials plant gives students huge responsibility

Nottingham placement students given input over design, construction and commissioning in SHYMAN project

EDWARD LESTER, TECHNICAL DIRECTOR OF PROMETHEAN PARTICLES
SPEAKS TO CHRIS TAYLOR

UK NANOMATERIALS manufacturer Promethean Particles has given chemical engineering students at the University of Nottingham an opportunity to have significant input and responsibility over the design, construction and commissioning of a full-scale nanomaterials production plant.

Edward Lester, technical director of Promethean Particles and professor of chemical technologies at the University of Nottingham, is responsible for supervising students in their final years and gives the most promising of them the chance to design and build portions of Promethean's plant.

Originally patenting the process idea in 2004 and developing the small-scale technology within the university until 2007, the technology was spun out to the company shortly after to scale up the process to meet commercial demands. The company began developing the full-scale plant in 2013 with partner universities and businesses – including Solvay, PPG, and Repsol – as part of a European nanomaterials research programme known as Sustainable Hydrothermal Manufacturing of Nanomaterials (SHYMAN). This project was funded by the EU's Framework Programme 7 (FP7) to explore the potential of new technologies in industry.

The plant completed its commissioning phase in May this year and the project was handed over to the Promethean site in Nottingham where the first product from the reactor was collected in June.

STUDENT LEGACY

Lewis Neve, production engineer at Promethean, began working on the project in August 2013 as a research assistant at the University of Nottingham. After becoming familiar with the process by conducting a hypothetic scale-up of a supercritical hydrothermal reactor and conducting experiments with nanocrystals during his master's degree in chemical engineering, he said this put him in "good stead" when the opportunity came around to design the plant for real. Neve said he has been heavily involved with the design, procurement and construction of the plant up until May 2016 when he became an employee of Promethean. Since then he has been responsible for commissioning, operating and maintaining the plant.

"There has been a lot of individual work on my part and a lot of responsibility was handed to me by Ed [Lester]. The fact that we have a plant that has been proven to work is a huge accomplishment in my eyes," he said.

THE PROCESS

The plant's unique technology is based around a supercritcal hydrothermal synthesis process in which water or solvents, taken beyond the critical point – 374°C and 218 atm (~22 MPa) for water – flow through one half of a reactor and into a nozzle. A solution of preheated metal salt precursors flow into the nozzle from another end and the resulting encounter with the superheated water in the counter current within the nozzle allows for a near instantaneous reaction which forms the desired nanomaterial. The products are then taken out of the reactor to be cooled and depressurised before the product is collected.

What is unique about this technology is that the nozzle reactor allows the process to work continuously. The team undertook modelling work using transparent reactors and replacing superheated water with methanol, and metal salts with cold sugar water, and ran the simulation with dye to see how the fluids were mixing and to optimise the reactor design around this.

Promethean's process also allows the heat from the reaction to be recovered from the process and recycled back to the preheating stage of the inflow. Lester said the water effluent can also be recovered, depending on the materials. He said organic materials are burned off during the reaction and only water and products remain at the end of the process; however, some inorganic precursors often lead to nitric or sulphuric acid being formed with the products. He said it was still possible to reuse these effluents if there was a strategy for a precursor that required them on the next run of reactions. The technology won the Nanochallenge Open Innovation Award from BASF in the US last year.

SCALE UP, SCALE OUT AND FLEXIBILITY

Since the start of the project, Promethean has scaled from producing nanomaterial products on the gramme scale to a 1 t/y pilot plant, and now at full scale to around 1,000 t/y. Lester said that is based on a continuous flowrate of 8,000 h/y.

He said the figure covers most of the products the company makes, but larger materials such as artificial bone can only be produced at ~100–200 t/y, depending on the specified morphology. Smaller materials like nanoceramics or titanium dioxide could theoretically be produced at up to 2000 t/y.

Lester said the prospect of scaling up the project further is unlikely, as producing 1,000 t/y in the nano market is more than sufficient and that most of Promethean's client's annual needs can be met within a week of continuous production. The reactor can be adapted to operate at higher flow rates should greater demand arise.

Lester said this is a rapid and flexible process where only one reactor is needed to meet all of the annual needs for potential clients. The reactor design allows the system to be cleaned and reset for different reactions easily.

This has many advantages over the current batch process for producing nanomaterials, where the process can take hours to complete and quality can vary between batches. This would be an advantage to industry as they usually need large quantities of materials to be tested or implemented quickly.

"This is the nano-equivalent of fast food in that if we are not making it in a fraction of a second then there is less of an advantage over other slower batch technologies" said Lester.

THE PRODUCTS

Promethean can make an array of metal and metal oxide nanoparticles including silicon dioxide, titanium dioxide and zirconium dioxide. Lester said his team has so far been able to make highly crystalline zirconium dioxide of around 6 nm, which can be used as a catalyst or as a high refractive index material for inks or coatings.

Lester said the team will soon embark on making metal organic frameworks (MOFs), used for chemical filtration, carbon capture, and hydrogen storage, at a capacity of 1,000 t/y. If successful, this will make Promethean the largest MOF manufacturer in the world. There are only a handful of MOF manufacturers that can produce more than grammes per day.

"At 1,000 t/y, the economics reach the point where industries will start to take MOFs more seriously because you are selling them for ten times cheaper than you can currently buy them. That means industry can get around the catch-22 of not having test samples and start looking at how you can use them in industry," said Lester.

SHYMAN: ON THE CONTRARY, LESTER IS NO STRANGER TO TEAMING UP WITH STUDENTS

FAMILY TIES

Promethean has strong ties to the University of Nottingham. Every year, a chemical engineering student is taken on for a year's industrial placement to gain hands-on experience with the project.

Finlay Pilkington, the 2015/16 year-in-industry student has had a large personal responsibility to design, construct piping, and purchase equipment for the post-processing portion of the large-scale plant. He said the project has given him a unique experience beyond the designing he has done at university. He said constructing the piping has given him greater practical skills than he would gain at other companies and being directly involved with the physical aspects of the project has forced him to tweak his design work.

He added this project has also given him the satisfaction of seeing the project through to the commissioning phase, something that is not always possible on a one-year placement from the design phase. He said he felt a great sense of accomplishment when the reactor was first turned on successfully in June.

Pilkington said, "When we first ran the first product and it was nanomaterials, not just water or a mixture of chemicals and when I had actually seen the plant through from the design phase to producing nanomaterials [these] were my favourite achievements. Knowing I had contributed quite significantly to build the plant and had input in the design made me feel proud that it worked." ■