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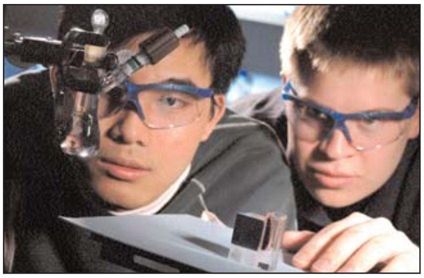
Green Chemistry: Nanometer-Scale Magnetic Particles Facilitate Separations in "One-Pot" Multi-Step Reaction Processes

Using the unique properties of new nanometer-scale magnetic particles, researchers have for the first time separated for reuse two different catalysts from a multi-step chemical reaction done in a single vessel.



By combining the new magnetic separation process with traditional gravity-driven separation, the technique could lead to more efficient production of specialty chemicals - and a reduction in waste normally produced by separation processes.

"We have developed a way to do multiple reactions in a single vessel while being able to recover the catalysts in pure form for reuse," said Christopher W. Jones, an associate professor in the School of Chemical and Biomolecular Engineering at the Georgia Institute of Technology. "By doing the reactions in a single vessel, we can cut out two or three separation steps to provide both an economic advantage and an environmentally benign process."

Separations using magnetic catalysts have been limited by a tendency of the nanoparticles to clump together because of their magnetic attraction for one another. The clumping dramatically reduces their catalytic activity.

To overcome this problem, the Georgia Tech researchers used nanometer-scale magnetic particles that are so small (5 to 20 nanometers in diameter) that they no longer exhibit a net magnetic attraction. But these superparamagnetic nanoparticles, developed by the research group of Z. John Zhang in Georgia Tech's School of Chemistry and Biochemistry, are attracted to an external magnetic source, providing a mechanism for separating them in pure form from the reaction vessel.

"These magnetic nanoparticles work well as catalyst supports because they are very small and so have a high surface area that allows creation of many catalytic sites for high activity levels," Jones said. "Because they are superparamagnetic, they remain suspended in the reaction vessel and do not clump together until a magnetic source is brought near them."

Traditional batch chemical production involves a sequence of paired chemical reaction and separation steps at the end of which the desired chemical product must be removed from the excess reactants, waste products and catalyst. The separation steps, which often require substantial energy inputs, add significant cost to the overall process.

To reduce the number of separations required, researchers have developed "one-pot" processes in which multiple reactions take place without intermediate separation. However, separations still must be done at the end of the combined reaction steps.

The new technique would allow more than one catalyst to be recovered and reused at the end of the one-pot reactions. Jones envisions the new process being used in the specialty chemical and pharmaceutical industries which produce relatively small volumes of high-value chemicals.

"For a specialty chemical company, you could imagine having a library of different catalysts that could be recovered by traditional methods and a library of magnetic catalysts recovered by magnetic means," he explained. "You could mix and match them to do different one-pot reactions depending on the needs."

In demonstrating the first example of a multi-step, one-pot reaction in which the catalysts could be recovered in pure form, the researchers controlled the reaction process by varying temperatures and pressures and controlling when reactants were introduced.

Because of its simplicity, Jones expects the new one-pot technique could be immediately put to use for chemical reactions that require only organic active sites on the catalysts. For more complex processes, additional time would be required to develop the necessary catalysts.

Ultimately, economics will determine where the process is used. "Anything that can be done in the chemical industry to reduce the number of separations can greatly reduce the cost of making a product," Jones said. "If you could cut the cost of synthesis by as little as 20 percent, that would have a huge impact."

For the future, Jones and Zhang envision using multiple catalysts whose magnetic properties would be tuned for activation at different temperatures, allowing them to be separated independently.

After the reaction, the non-magnetic catalyst was removed from the vessel by decantation while a small permanent magnet held the magnetic catalyst to the vessel wall. After separation, the recovered catalysts we analyzed for signs of contamination and then reused in other multi-step one-pot chemical reactions without loss of catalytic activity.

Supported by an exploratory research grant from the National Science Foundation and by Georgia Tech internal research funding, the project demonstrates how the unique properties of nanometer-scale materials can find real-world applications.

"Here, nanotechnology allows us to do something that is commercially relevant and environmentally benign," Jones said. "The understanding of magnetic properties at the nanoscale allowed us to put a magnetic catalyst and a non-magnetic catalyst together, do a reaction, and then separate them."

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