Researchers electrify polymerization

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Scientists led by Carnegie Mellon University chemist Krzysztof Matyjaszewski are using electricity from a battery to drive atom transfer radical polymerization (ATRP), a widely used method of creating industrial plastics. The environmentally friendly approach, reported in the April 1 issue of *Science*, represents a breakthrough in the level of control scientists can achieve over the ATRP process, which will allow for the creation of even more complex and specialized materials.

ATRP, first developed by Matyjaszewski in 1995, allows scientists to easily form polymers by putting together component parts, called monomers, in a controlled piece-by-piece fashion. Assembling polymers in such a manner has allowed scientists to create a wide range of polymers with highly specific, tailored functionalities. ATRP has been used to develop cosmetics, coatings, adhesives and drug delivery systems, and is used to develop "smart" materials — those that respond to environmental changes, such as changes in temperature, light, pressure or pH.

The current study represents the latest in a series of advances Matyjaszewski's research group has made since ATRP's inception that make the technique more precise and more environmentally friendly. In a process they are calling electrochemically mediated ATRP, or eATRP, the researchers used a computer-controlled battery to apply an electrochemical potential across the ATRP reaction.

"This marks the first time that we've paired electrochemistry with ATRP, and the results were startlingly successful," said Matyjaszewski, the J.C. Warner Professor of Natural Sciences at CMU. "We found that by adjusting the current and voltage we could slow and speed up, or even start and stop the reaction on-demand. This gives us a great deal more flexibility in conducting our reactions that should lead to the development of precisely engineered materials."

In traditional ATRP reactions scientists use a copper catalyst to grow a complex polymer structure by adding a few monomeric units at a time to the polymer chain. The process relies on paired reduction-oxidation (redox) reactions between two species of copper — the activator CuI and deactivator CuII — where the two catalysts exchange electrons back and forth. Occasionally, one of the exchanges will spontaneously stop, called a radical termination, resulting in the accumulation of CuII. To keep the polymerization going, researchers must rebalance the system by compensating for the excess CuII.

In the early ATRP experiments, scientists addressed this problem by adding more CuI to the system. This generated materials with high, sometimes toxic, levels of copper, reaching around 5,000 parts-per-million (ppm). Such levels of copper are hard to remove using current industrial equipment. As an alternative, Matyjaszewski and colleagues developed novel methods for using activators and reducing agents to reactivate the CuII. Most notably, they found that environmentally friendly reducing agents like sugars or vitamin C were highly effective in reducing the amount of copper catalyst used in ATRP reactions.

In the current study, Matyjaszewski and Visiting Assistant Professor of Chemistry Andrew Magenau looked to electrochemistry as a means for maintaining balance in ATRP reactions. They found that adding electricity capitalized on the redox reaction by moderating the transfer of electrons. This allowed them to compensate for the radical terminations and reduce the amount of copper needed to run ATRP. As a result the amount of copper in the system was reduced to 50 ppm, a

100-fold decrease. In terms of creating a greener, less toxic form of ATRP, this amount rivaled Matyjaszewski's previous studies that used vitamin C and sugars as reducing agents, but has the added benefit of not requiring the addition of any additional organic or inorganic reducing agents.

The researchers found that applying electricity to the system also gave them more precise control over the reaction. The computer-controlled battery allowed them to manipulate the ATRP process in real-time by changing the current or voltage.

The researchers have used this process to create the standard types of polymers made with ATRP: star, brush and block copolymers. They believe that the meticulous control eATRP gives them over the rate of polymerization will allow for the creation of polymers with even more complex architectures.

Provided by Carnegie Mellon University

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