Developing a New Microreactor for Organic Synthesis Using Microwave Heating

Microwave heating can deliver faster chemical reaction rates than conventional heating methods. Researchers from MIT are investigating ways to improve microreactor designs for uniform temperature distributions and better handling of chemical processes.

BY LEXI CARVER

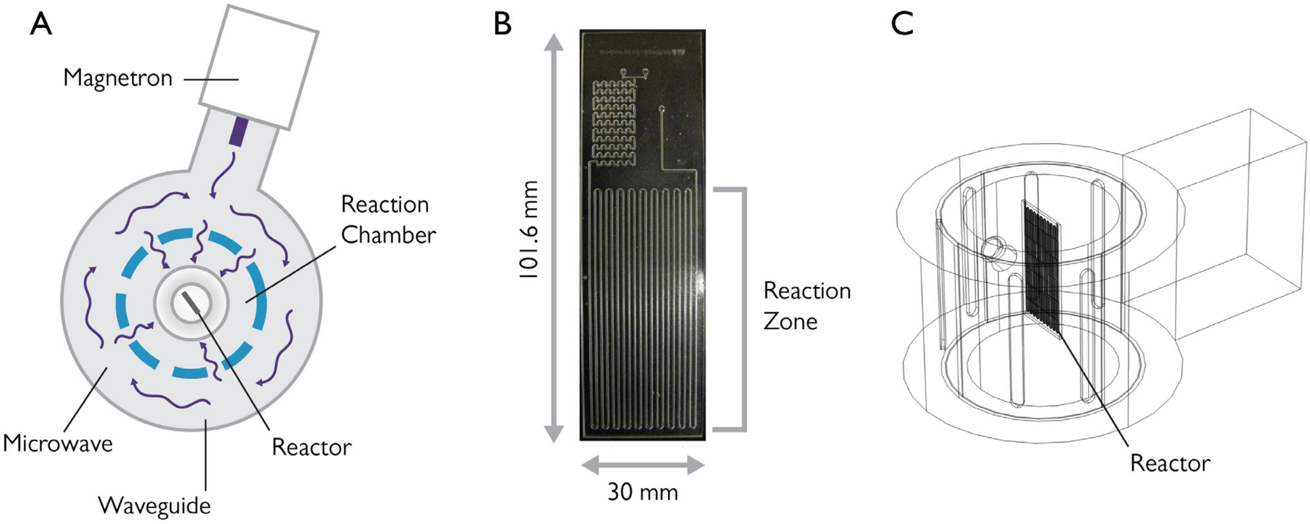

FIGURE 1. (A) Schematic top view of the microreactor system. (B) Borosilicate glass plate. (C) 3D geometry used in simulation.

Precisely controlled chemical reactions are the magic behind many materials used in everyday products, such as solvents, pharmaceuticals, and fluids used in engines and cleaning agents. Such materials are often created by organic synthesis. This process combines individual chemicals and can be done through microwave heating in a microreactor system, which is direct, localized, and allows for extremely fast and efficient high-temperature heating.

Despite its promising benefits, the uniform temperature distribution that is crucial for organic chemistry is often difficult to achieve. Chemical reactions can be inhibited by unwanted temperature changes, and by heating that occurs too slowly or does not reach a high enough temperature. Wen-Hsuan Lee, researcher at Massachusetts Institute of Technology (MIT, Cambridge, MA, U.S.) discovered this challenge when a microreactor she initially fabricated to conduct microwave organic synthesis would not reach the necessary temperature. Under sponsorship from the National Institute of Health (NIH, Bethesda, MD, U.S.) and the supervision of Klavs Jensen, director of the Jensen Research Group, Lee investigated the slow heating rates and non-uniform temperature distribution—eventually, she designed a new reactor to enable more efficient chemical processes.

INVESTIGATING UNEVEN HEATING: GEOMETRIC CHALLENGES

The microwave system Lee used to test her microreactor (see Figure 1A, 1C) was provided by CEM Corporation (Matthews, NC, U.S.), a manufacturer of laboratory microwave equipment. The device comprises a magnetron generating microwaves; a circular waveguide that joins to the magnetron and guides the microwave path; and a cavity where the chemistry reactors are loaded. The inner wall of the waveguide has vertical gaps spaced around it to let microwaves enter the reaction chamber.

Lee designed a borosilicate glass microreactor with flow channels for the reaction fluid to pass through (see Figure 1B), which sits at the center of the reaction chamber. The fluid absorbs microwaves and heats up at the reaction zone, and the serpentine shape of the channels maximizes the amount of fluid contained and heated. Uniform heating of the reaction is crucial—without this, the complete chemical process will not occur properly.

But as Lee discovered, the resulting temperature distribution was not, in fact, uniform. The waveguide shape caused microwaves to enter the chamber at unpredictable intervals and angles, making it difficult to ensure the desired temperature. She then turned to COMSOL Multiphysics® for a simulation to explain how the microreactor geometry affected the heating process. "The maximum steady-state temperatures in the reactor were too low and the temperature changes too slow for the chemical reactions we wanted," Lee explained. "We used COMSOL software to diagnose the problem—and discovered it's a problem inherent in microwave irradiation that can be solved by adjusting the reactor design." From her model, she was able to predict the heat convection in the air inside the cavity and the fluid in the microreactor channels.

TEMPERATURE TRENDS IN THE MICROREACTOR CHANNELS

After examining the heating profiles, Lee realized that the shape and orientation of the reactor influenced the microwave and temperature distributions; results

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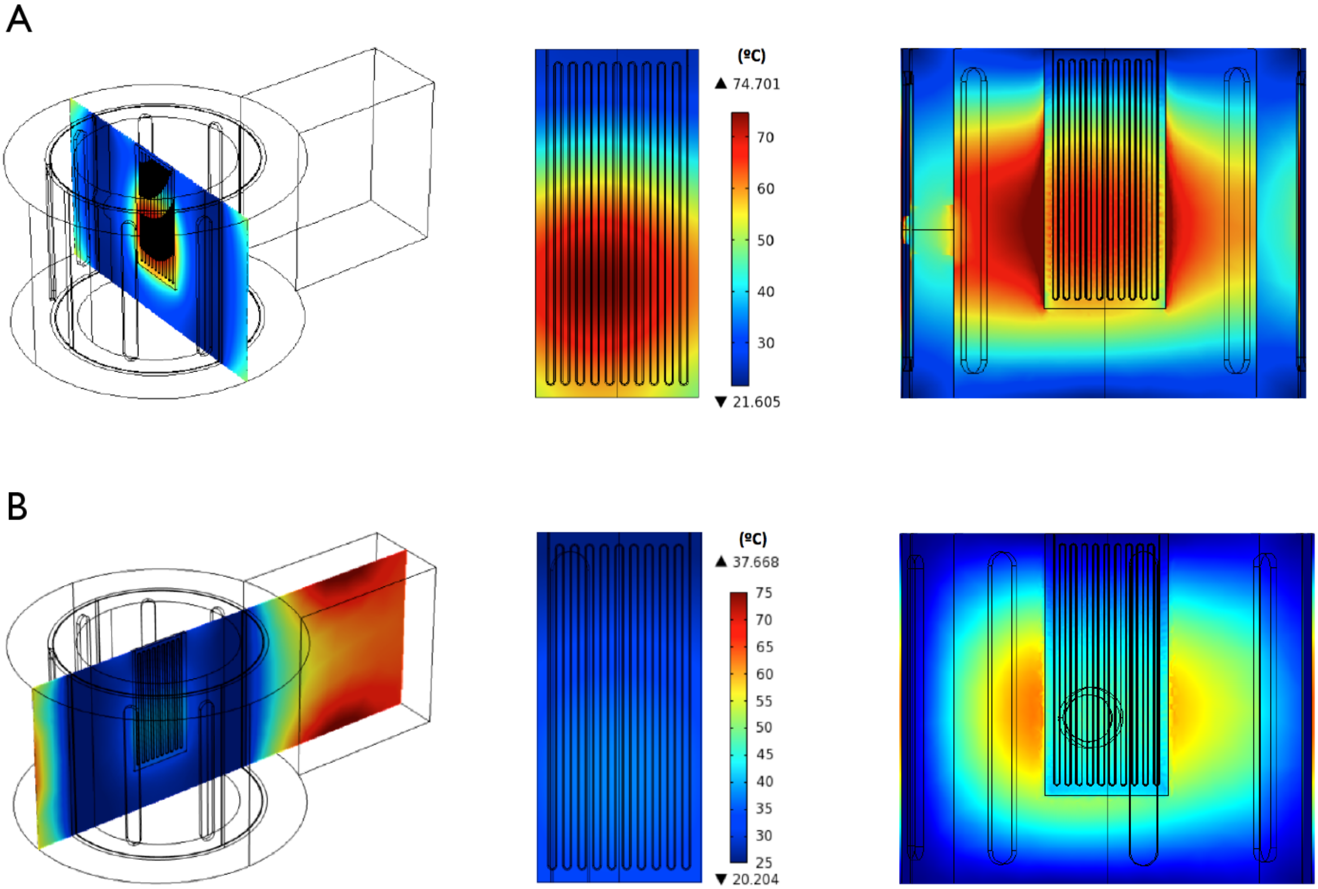

FIGURE 2. Simulation results showing heat distribution changing with reactor orientation. (A) Highest-temperature results. (B) Lowest-temperature results.

varied widely with the position of the microreactor. She used her COMSOL simulation to optimize its location and orientation, aiming to maximize the strength of the electric field. Testing out different rotation angles (including those in Figure 2), she determined the position that resulted in the highest and fastest heat transfer, which allowed her to focus the reaction channels at the point of greatest electric field strength.

Her simulation indicated significant changes in temperature for different rotation angles; the optimal position for the highest temperature occurred with the microreactor parallel to the microwave port (see Figures 2 and 3). Next, she experimented with different thicknesses for the borosilicate glass. Her COMSOL results indicated that the lowest electric field strength occurred at extremes of 14 mm and 1.4 mm, and the highest around 7 mm.

Optimizing the rotation angle and thickness of the glass, Lee greatly improved the efficiency of the system. The discoveries from her simulations guided her new reactor setup; after refining the existing system, she developed a configuration containing several layers of channels arranged in the area with the highest localized heat.

Her new microreactor design arranges the channel shapes to maximize the amount of fluid positioned in the region with the greatest electric field strength (see Figure 4).

ENABLING MORE EFFICIENT CHEMICAL REACTIONS

Though the original microreactor setup made it difficult to deliver a uniform temperature distribution and high enough heat for the desired chemical processes, Lee's simulation enabled her to optimize the design and orientation of the borosilicate glass. Her findings, which were crucial for achieving even heat distribution, will help Lee and her colleagues to continue developing a microreactor that can couple with microwave irradiation for organic synthesis. ■

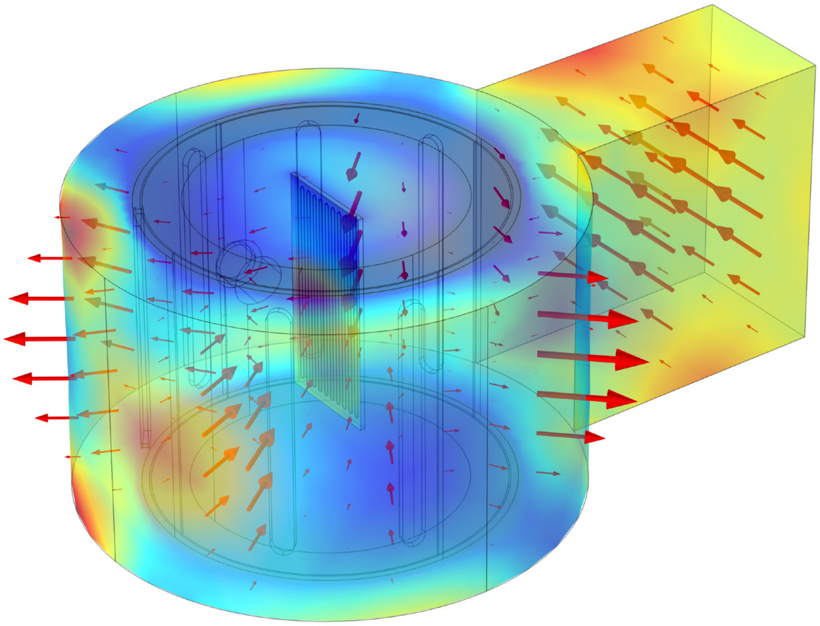

FIGURE 3. Temperature and electric field distribution in the microreactor system.

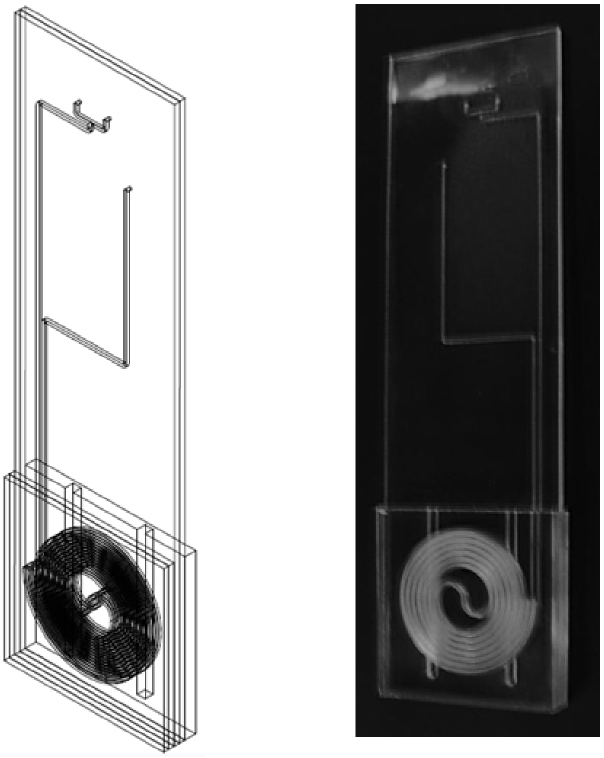

FIGURE 4. Lee's microreactor design.