Miniature grinding mill provides observation of 'green' chemical reactions

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Person in laboratory holding a flask. Credit: Chokniti Khongchum from Pexels

Scientists at the University of Cambridge have developed a new approach for observing mechanochemical reactions—where simple ingredients are ground up to make new chemical compounds and materials that can be used in anything from the pharmaceutical to the metallurgical, cement and mineral industries.

The study, published in *Nature Communications* and led by Cambridge

Earth Sciences' Dr. Giulio Lampronti, observed reactions as materials were pulverized inside a miniaturized grinding mill—providing new detail on the structure and formation of crystals.

Knowledge of the structure of these newly-formed materials, which have been subjected to considerable pressures, helps scientists unravel the kinetics involved in mechanochemistry. But they are rarely able to observe it at the level of detail seen in this new work.

The study also involved Dr. Ana Belenguer and Professor Jeremy Sanders from Cambridge's Yusuf Hamied Department of Chemistry.

Mechanochemistry is touted as a 'green' tool because it can make new materials without using bulk solvents that are harmful to the environment. Despite decades of research, the process behind these reactions remains poorly understood.

To learn more about mechanochemical reactions, scientists usually observe chemical transformations in real time, as ingredients are churned and ground in a mill—like mixing a cake—to create complex chemical components and materials.

Once milling has stopped, however, the material can keep morphing into something completely different, so scientists need to record the reaction with as little disturbance as possible—using an imaging technique called time-resolved in-situ analysis to essentially capture a movie of the reactions. But, until now, this method has only offered a grainy picture of the unfolding reactions.

By shrinking the mills and taking the sample size down from several hundred milligrams to less than ten milligrams, Lampronti and the team were able to more accurately capture the size and microscopic structure of crystals using a technique called X-ray diffraction.

The down-scaled analysis could also allow scientists to study smaller, safer, quantities of toxic or expensive materials. "We realized that this miniaturized setup had several other important advantages, aside from better structural analysis," said Lampronti. "The smaller sample size also means that more challenging analyses of scarce and toxic materials becomes possible, and it's also exciting because it opens up the study of mechanochemistry to all areas of chemistry and materials science."

"The combination of new miniature jars designed by Ana, and the experimental and analytical techniques introduced by Giulio, promise to transform our ability to follow and understand solid-state reactions as they happen," said Sanders.

The team observed a range of reactions with their new miniaturized setup, covering organic and inorganic materials as well as metal-organic materials—proving their technique could be applied to a wide range of industry problems. One of the materials they studied, ZIF-8, could be used for carbon capture and storage, because of its ability to capture large amounts of CO_2. The new view on these materials meant they were able to uncover previously undetected structural details, including distortion of the crystal lattice in the ZIF-8 framework.

Lampronti says their new developments could not only become routine practice for the study of mechanochemistry, but also offer up completely new directions for research in this influential field, "Our method allows for much faster kinetics, and will open up doors for previously inaccessible reactions—this could really change the playing field of mechanochemistry as we know it."

More information: Giulio I. Lampronti et al, Changing the game of time resolved X-ray diffraction on the mechanochemistry playground by downsizing, *Nature Communications* (2021). DOI: 10.1038/s41467-021-26264-1

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