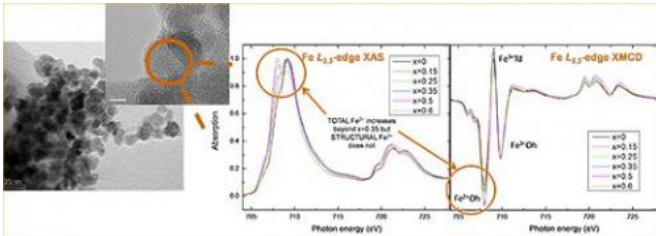


Designer particles stand in for layers of subsurface minerals

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Tuning the particles does not involve adding more iron, but rather, forcing the iron in the particles to become reactive or passive. Here, microscopic images of iron particles and extensive tests demonstrate that the passive iron is becoming reactive, rather than adding more iron.

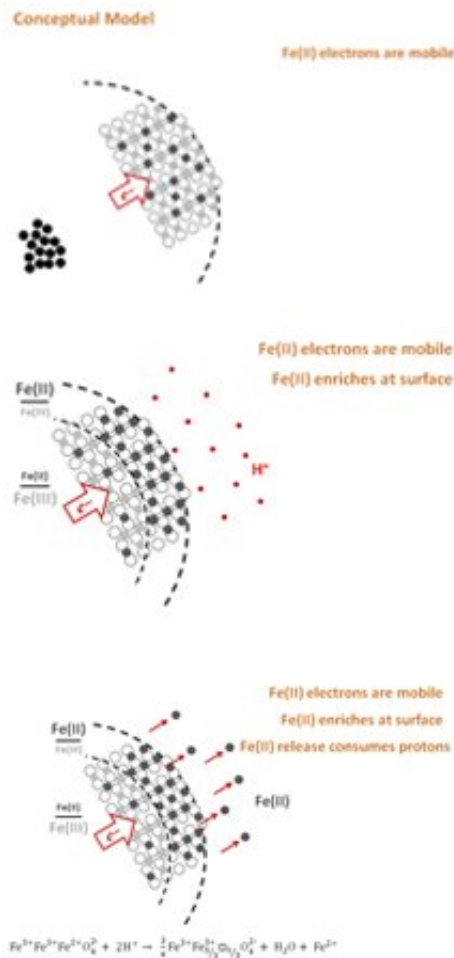
(Phys.org)—To understand how underground pollutants react with magnetite and other minerals, scientists need an easy-to-use mineral stand-in. An international team led by Pacific Northwest National Laboratory created analogous particles with precisely tuned amounts of relatively reactive iron, or Fe(II), and less reactive iron, Fe(III), to match natural conditions. Tuning this Fe(II)/Fe(III) ratio dials in the desired amount of iron reactivity. To test their process, the team created tiny iron oxide spheres similar to magnetite, except with titanium added to control the Fe(II)/Fe(III) ratio directly.

"This nanoparticle system lets us fine-tune the iron in a predictable manner and determine the reactivity systematically," said Dr. Carolyn Pearce, a PNNL [geochemist](#) who led the study.

Geochemists want to know how contaminants, such as technetium, interact with the reactive fraction of minerals at former [nuclear weapons](#) sites. But, to unravel these kinds of complex problems the researchers need well-defined samples they can analyze in the laboratory. These new [particles](#) appear to be good understudies for

these minerals. The particles are also of interest in development of iron-based fluids, pioneering [cancer therapy](#), [drug delivery](#), [chemical sensors](#), [catalytic activity](#), photoconductive materials, and more traditional uses in data storage.

"PNNL's materials have already been used in next-generation bioassays for cellular nanoparticle uptake," said Pearce. "Their ability to exchange electrons with substances in the liquid that surrounds them makes them an intriguing prospect for a host of remediation uses as well."



When placed in a dilute, slightly acidic liquid, the reactive iron in the particles moves to the surface and then out into the environment, where it reacts.

the surface to the solution.

The team synthesized the particles on a bench top with simple aqueous chemistry but containing precise amounts of titanium doped into their crystal structures, which tunes the Fe(II)/Fe(III) ratio. The team performed and reported a comprehensive set of spectroscopic and microscopic studies on these particles on everything from their atomic structure to their shapes and reactivity.

"Creating the particles on a bench top makes it easy, but understanding what you've got in detail requires a lot of characterization and tools," said Pearce. "With the suite of instruments now available, here in the EMSL and at synchrotron user facilities, we were able to push this fundamental science to an unprecedented level."

After performing basic chemical tests, the team turned to micro-x-ray diffraction on aqueous suspensions of the nanoparticle to look at the box-like structure of the particle's atoms. This diffraction technique also showed that the team could only synthesize particles up to a certain level of titanium at room temperature.

Next, the team characterized particles Mössbauer spectroscopy and x-ray photoelectron spectroscopy with hard x-rays, which allowed them to examine the particles' interiors. They then used softer, less invasive x-rays at the synchrotron to glean details about the particles' surfaces, where a lot of the chemistry takes place.

They used a transmission electron microscope to see the particles' crystallinity and morphology. They found the particles were generally spherical but with some crystal facets and a 10- to 12-nanometer diameter on average.

Then, the team put the particles in a more dilute system and ran all the tests again, giving the team a before and after look at the particles. The results provided the team with the composition, structure, and magnetic properties of the titanomagnetite particles, with distinctions between the way the particles behave inside relative to their surfaces. They found that in a slightly acidic or proton-rich liquid, the Fe(II) moves from the particle's interior to

"It is really difficult to track the iron movement in samples, particularly in the few atomic layers near the surface," said Dr. Kevin Rosso, who leads the Geochemistry group at PNNL and worked on this study. "But, in this system we did just that."

By determining the complex formulas that explain how the nanoparticles behave with different levels of iron, the team is now taking that data and comparing it to the behavior of titanomagnetite found on the Hanford Site. These comparisons are helping scientists better predict how the natural material will behave as it encounters different types of waste over thousands of years.

More information: CI Pearce, et al. 2012. "[Synthesis and Properties of Titanomagnetite \(Fe_{3-x}Ti_xO₄\) Nanoparticles: A Tunable solid-state Fe\(II/III\) Redox System.](#)" *Journal of Colloid and Interface Science*, 387(1):24-38. [DOI:10.1016/j.jcis.2012.06.092](#)

Provided by Pacific Northwest National Laboratory

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