SFE537: Conservation of Corroded Iron Artifacts Using Subcritical Water

Introduction

Multiple corroded iron artifacts, including some recovered from the H.L. Hunley, a civil war submarine, were conserved using a novel subcritical water procedure. The use of subcritical water enhanced the stabilization of the artifacts by the rapid exchange of chloride ions (Cl^-) with hydroxide ions (OH^-). Subcritical water significantly improved diffusion constants of the anionic exchange of chloride and hydroxide ions. In addition, long term stability is enhanced by transformation of corrosion products into more stable forms.

Subcritical water

The term subcritical water describes water in the liquid state at a temperature above its boiling point of 100 C and its critical temperature of 374 C. The water is liquified by applying pressure to the system. Liquid water exhibits many changes in its characteristics as it is heated to the subcritical state. The viscosity and surface tension of the liquid water decreases, and diffusivity increases. In addition, the dielectric constant of water decreases significantly as it is heated, and water behaves like a mixture of water and methanol when in the subcritical temperature range. Thus, subcritical water may also be used to extract many organic molecules such as phenolic monomers with substantial environmental benefits compared to the use of conventional organic solvents.

Equipment

Applied Separations 40 Liter Subcritical Water Pilot Plant (Allentown, PA, USA).

High-pressure pumps introduce the treatment solution from the NaOH feeder vessel into the reactor pressure vessel. The reactor vessel is heated by a heating jacket and pressure is controlled via a backpressure regulator. Mixing of the 40 L reactor was improved by the addition of a high flow recirculation line operated by a variable speed pump.

Temperature and pressure at various locations in the system are monitored, recorded, and controlled via computer. Filters with a range of pore sizes are installed to protect valves and tubing from debris or sediment. A heat exchanger was added to the reactor to control the cooling of the system after treatment.

The loading and unloading of heavy artifacts is facilitated by a crane that is also used to lift and close the reactor vessel endcap. A set of perforated stainless-steel baskets are used to support the artifacts during treatment.

Materials

- ✓ 0.5 wt% NaOH degassed via sonication

Method

(See references for details)

1, Add 0.5% NaOH to the feed reservoir.
2. Place artifact into the treatment vessel

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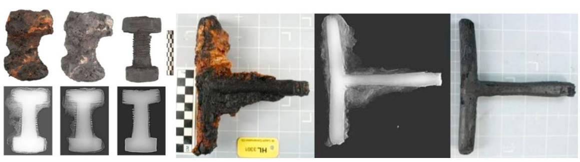
3. Prime the fluid flow path and flush any air out of the system.
4. Close the treatment vessel.
5. Pressurize the vessel to 50 bar.
6. Establish and stabilize flow of 0.5%NaOH to specified flow rate.
7. Increase temperature to 180 C.
8. Once the system is stable, the eluent is sampled periodically at the exit of the vessel for Cl- concentration.
9. When this concentration reaches the level of the feed solution, the heating elements are turned off.
10. Continue to pump cold feed solution until the vessel reaches room temperature.
11. Turn off the feed pump and depressurize the vessel.
12. Open the vessel and retrieve the artifact.
13. Rinse artifact thoroughly with deionized water until the rinse is pH neutral.
14. Dry artifact quickly to prevent flash corrosion.

Results



Conclusion

The subcritical water desalination process significantly reduces the processing time required to reduce the Cl- concentrations of artifacts. In addition, the conserved specimens are more stable and allow for post treatment deconcretion. The process allows for drying to take place prior to any mechanical cleaning due to the stability of the artifact and its corrosion matrix. This result makes visual identification of the corrosion layers easier to remove.

References

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