Unlocking 4D Elemental Quantification: Metal Leaching in 4D Using TESCAN Spectral CT

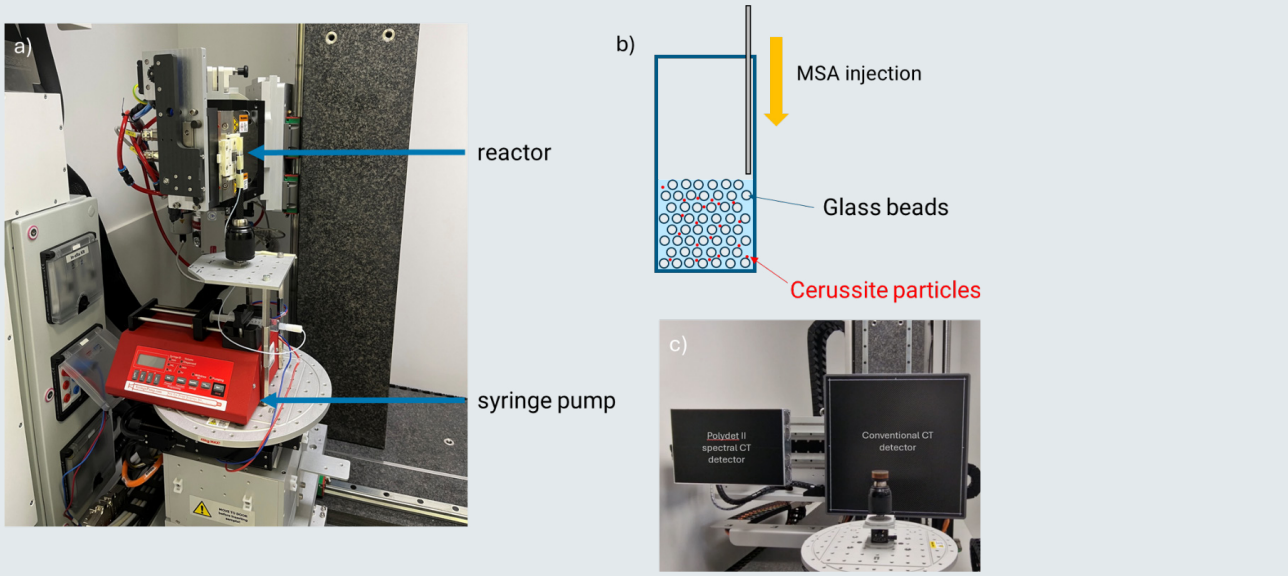
Introduction

The global shift towards electrification depends on a stable supply of critical metals such as copper, lithium and rare earth elements (REEs). These metals can be extracted from ores, scrap, or industrial residues through hydrometallurgy-a process that dissolves metals into an aqueous solution. Hydrometallurgy is particularly suited for low-grade or complex ores and is widely recognized as a more environmentally friendly alternative to other metal extraction methods. Conventional hydrometallurgical processes typically rely on inorganic acids like hydrochloric and sulfuric acid. While these acids are inexpensive and widely available, they have considerable disadvantages, including the formation of poorly soluble salts (e.g., with Ag, Pb, or Zn) and issues with gypsum scaling. To overcome these challenges, researchers are investigating more efficient and sustainable leaching agents (e.g., Binnemans and Jones, 2023).

The TESCAN UniTOM XL Spectral, with its unique 4D imaging capabilities, is especially suited to investigate the complex, time-dependent nature of metal leaching. This study leverages the system's advanced elemental and structural imaging to gain unprecedented insight into the leaching behavior of cerussite ($PbCO_3$), a naturally occurring lead ore, when exposed to a high-potential alternative leaching agent.

Experimental Setup and Protocol

The experiment was conducted using a custom setup mounted on the rotation stage of a TESCAN UniTOM XL, equipped with a TESCAN Polydet II spectral detector to study the leaching process of lead carbonate ($PbCO_3$),



▲ **Figure 1:** *Overview of the used setup*

a. Experimental configuration mounted on the rotation stage
b. Schematic of the reactor vessel
c. Dual detector in the TESCAN UniTOM XL Spectral

A custom reactor vessel was constructed using a polymeric vial (PTFE, 30 mm high, 6.3 mm I.D.), connected to a syringe pump (New Era Syringe Pumps Inc., NE-300) via 1/16" polymer tubing. The syringe pump powered through the rotation stage's slip ring, allowing for continuous rotation of the test-setup during image acquisition.

The reactor was partially filled (~ 30%) with 300 µm glass beads, into which 10mg of crushed cerussite ($PbCO_3$, a naturally occurring lead mineral) was mixed. The remaining volume was saturated with de-ionized water, just covering the top of the bead bed.

During the experiment, 0.4 ml of 0.25 M Methanesulfonic acid (MSA, CH_3SO_3H) was added into the reactor over a period of 2 hours at a flow rate of 3.3 µl/min. MSA is a strong, biodegradable acid known for its high solubility with metal salts and is increasingly used in hydrometallurgical applications involving lead, zinc and silver, as well as in recycling processes for Pb- and Li-ion batteries, PV-modules and phosphor lamps. (Binnemans and Jones, 2023). The formula for the dissolution reaction of cerussite with MSA is:

$$PbCO_{3\,(s)} + 2\ CH_3SO_3H_{\,(aq)} \rightarrow Pb(CH_3SO_3)_{2\,(aq)} + CO_{2\,(g)} + H_2O_{\,(l)}$$

The experiment was repeated on sister samples and imaged using both dynamic micro-CT and 4D spectral CT.

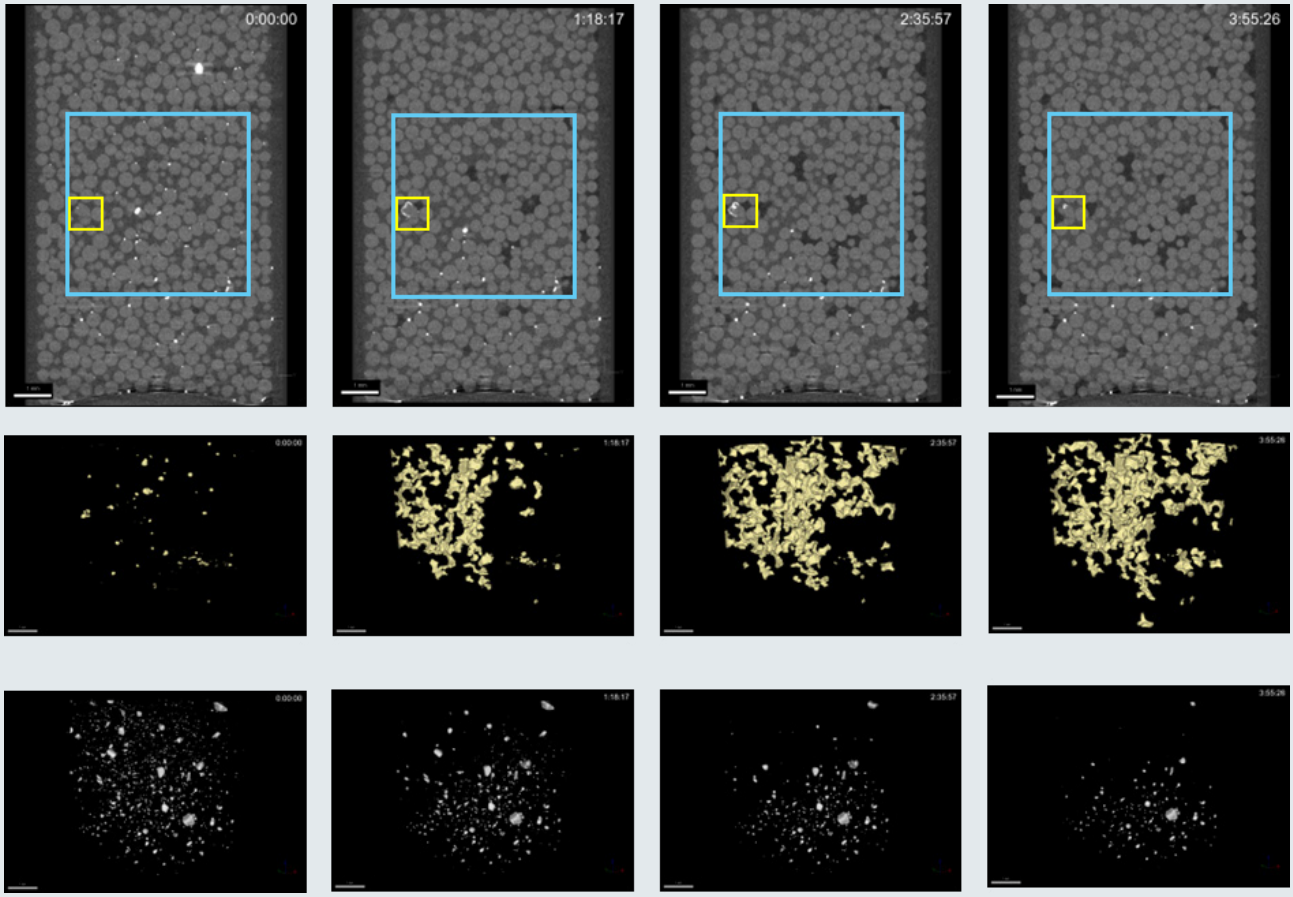
Investigating Metal Leaching u With Dynamic Micro-CT

Given the time-sensitive nature of the metal leaching process, scanner settings were optimized for rapid image acquisition, achieving a temporal resolution of 60 seconds per full 360° rotation. A total of 229 continuous rotations were captured over a 230-minute period. This was accomplished by maximizing the X-ray flux within a compact experimental geometry (see Table 1).

The first signs of cerussite dissolution are observed about 10 minutes after the start of methanesulfonic acid injection-corresponding to the time required for the acid front to diffuse through the pore network. Simultaneously, the formation of CO_2 gas bubbles becomes visible within the pore space. As expected, the finest cerussite dissolve due to their low volume and high surface-area-to-volume ratio. An interesting reaction is observed in the center-left region of the yellow box in Figure 2. First a precipitation reaction around a grain is observed followed by removal of the precipitate.

	Dynamic micro-CT	Spectral CT
Tube voltage	180 kV	160 kV
Tube power	15 W	15 W
Filter	1 mm Cu	no filter
Exposure time	85 ms	83 ms
Number of projections	710	1200
Source-Detector Distance	550 mm	743 mm
Reconstructed voxel size	9.8 µm	13 µm
Time per 360 rotation	60 s	5-10 min
Scan rotations	229	46

▲ **Table1:** *Overview of scan parameters used in the experiment.*

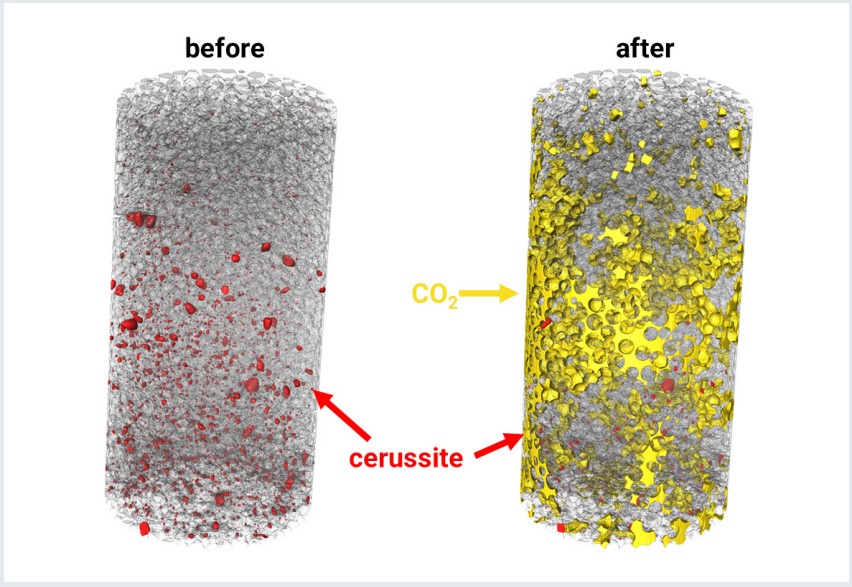


▲ **Figure 2:** *Results of dynamic micro-CT imaging*

Animated sequences of these frames are available here

From top to bottom:

a. 2D YZ cross-sectional slices through the sample
b. 3D rendering of the gas phase
c. 3D rendering of the cerussite distribution

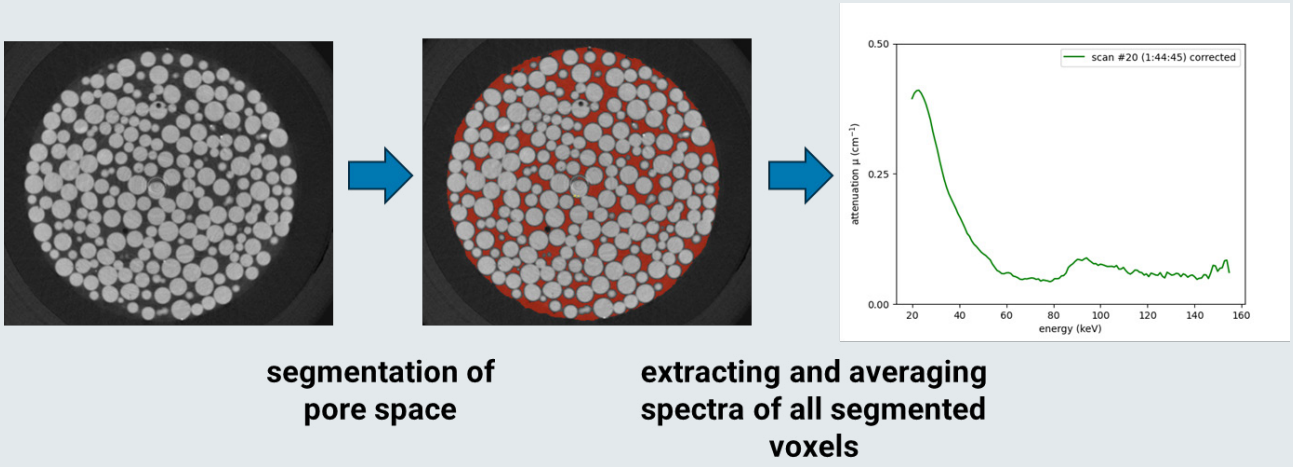


◀ **Figure 3:** *3D rendering of the sample before and after metal leaching.*

4D Spectral CT

TESCAN Spectral CT marks a significant advancement in lab-based micro-CT technology by capturing the full X-ray energy spectrum, allowing direct, voxel-level measurements of chemical composition and material density. In this study, we demonstrate how 4D Spectral CT can be used to investigate complex chemical processes- specifically, the time-resolved behavior of metal leaching. To understand the metal leaching process, we aim to identify the lead in the pore water of the sample by tracking the K-edge of lead. Moreover, we also recorded the density and effective atomic number of the pore-water solution over time.

The dissolution experiment was imaged by acquiring 5-minute back-to-back Spectral CT scans for 160 min followed by a 12-hour time-lapse of a10 minute Spectral scan followed by a 50-minute wait (i.e. one 10 min Spectral CT scan every hour). The spectral data was acquired and reconstructed using TESCAN Spectral Suite software (see Figure 4). After reconstructing the Spectral CT data, the pore space was segmented, and the Spectral CT data from all voxels classified as pore space were averaged to calculate a single spectrum, density and effective atomic number (Z_{eff}) per time step.



▲ **Figure 4:** *Spectral CT processing workflow for K-edge identification of lead*

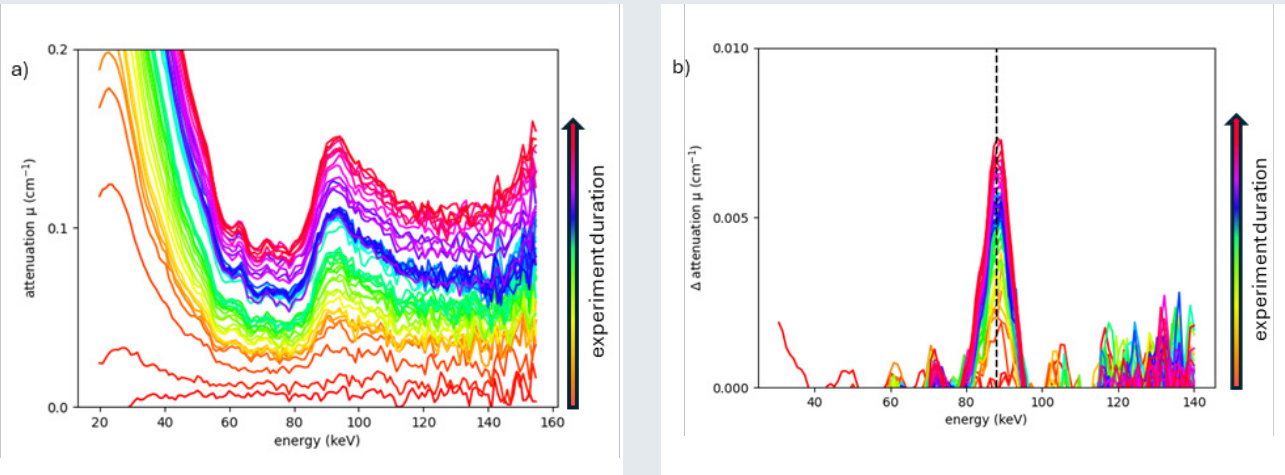
The acquired spectral data are presented in figure 5a, with the corresponding K-edge peak locations shown in figure 5b. The spectra change notably in three ways over the duration of the experiments.

1. The spectral curves rise over time, indicating an increase in total X-ray attenuation. This is caused by the growing density of the pore water as cerussite dissolves and releases lead into the solution.

2. The overall shape of the spectral curve evolves over time, due to the change in the effective atomic number of the pore water.

3. A distinct inflection point-commonly referred to as a K-edge-appears and becomes more pronounced between 80-100 keV. This issue corresponds to a sudden increase in X-ray absorption that occurs when the photon energy exceeds the binding energy of the K-shell electrons of an atom. Since the energy at which this occurs is element specific, the K-edge serves as a reliable indicator for elemental identification. In this case, the first derivatives of the spectra show a peak at 88.8 keV (± 1 kev), closely aligning with the theoretical value of the K-edge of lead at 88keV. The spectral signature becomes clearly visible after about 15 minutes, shortly after the onset of dissolution observed in the dynamic micro-CT experiment described above.

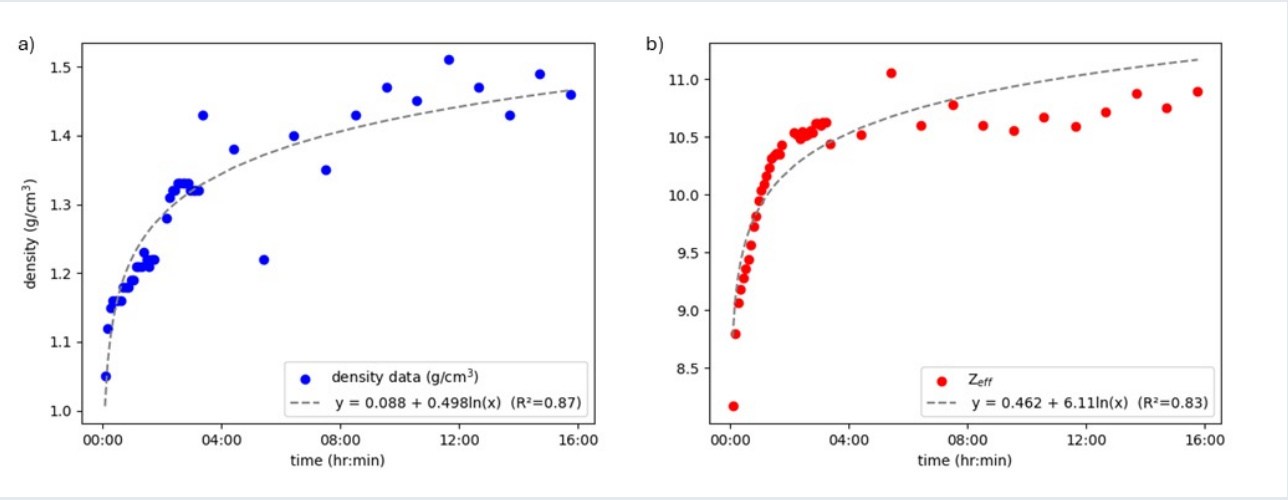
Figure 6 presents the calculated density and mean atomic number of the pore water These two values are directly derived from the spectral datasets. Notably, the initial measurements exceed the theoretical values for pure water (1 g/cm^3 density and 7.54 Z_{eff}). This indicates that while the main dissolution commences after about 10 minutes, MSA may already be present in the pore water during the first scan.



▲ **Figure 5:** Recorded X-ray spectrum of the segmented pore water

a) Note how the attenuation increased over the duration as well as the increasingly more pronounced kink in the spectrum.

b) K-edge peak extracted from the recorded spectra. The dotted line indicates 88 keV energy level.



▲ **Figure 6:** Temporal evolution of pore water properties

Calculated (a) density and (b) mean atomic number (Z_{eff}) of the pore water at each time step.

Conclusions

This application note demonstrates how TESCAN Spectral CT, when combined with dynamic micro-CT, enables 4D investigation of complex chemical processes such as those found in hydrometallurgy. We demonstrate how we can identify the presence of lead with high certainty, and that both the density and mean atomic number can be tracked over time. In the future this may pave the way for direct in-situ concentration measurements.

References

Binnemans, Koen, and Peter Tom Jones. *„Methanesulfonic acid (MSA) in hydrometallurgy."* Journal of Sustainable Metallurgy 9.1 (2023): 26-45.

TESCAN UniTOM XL Spectral

TESCAN UniTOM XL offers institutional and industrial research facilities, a highly versatile micro-CT platform capable of handling a wide range of sample sizes and types. It enables data acquisition across multiple length scales, supporting both standard 3D non-destructive imaging and continuous dynamic CT for true 4D in situ experimentation.

TESCAN SPECTRAL CT provides genuine spectral imaging by capturing the full X-ray spectrum before and after interacting with your samples. This spectral information enhances microstructural imaging by providing complementary chemical and compositional insights. It also enables contrast differentiation in scenarios where conventional attenuation based micro-CT reaches its limitations.

Key specifications of TESCAN UniTOM XL

Max. temporal resolution	<3 seconds for 360-degree rotation
Max. spatial resolution (line-pair)[1]	3µm or less
Geometry	Rotating sample
X-ray Source	30-180 kV or 30-230 kV Open type reflection source
X-ray detector	2856x2856 pixel; Amorphous silicon flat panel
Max. CT FOV (⌀xh)	1000 x 600mm
Max. Sample weight	45 kg
Motorization	10 motorized stages on high precision granite
System dimensions (approx.)	3.50 m × 1.58 m × 2.15 m (LxWxH)
System weight (approx.)	6550 - 7550 kg

[1] **Spatial resolution determined based on JIMA line pattern**

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