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First oxygen isotopic analyses in clay minerals within polymineralic samples using NanoSIMS

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Oxygen isotope compositions of clay minerals provide key information on their genetic environmental conditions, but their small grain size and frequent coexistence with other minerals often limit analysis by conventional bulk techniques [1]. Here, we present first oxygen isotopic measurements by nanoscale secondary ion mass spectrometry (NanoSIMS) on different clay minerals formed in different geological contexts.

NanoSIMS analyses were carried out on neoformed kaolinite and smectite from lower Barremian palaeosols (Cretaceous, Iberian Range, NE Spain), and on coexisting glauconite and celadonite related to hydrothermal alteration of Jurassic pillow lavas (Betic Cordillera, SE Spain).

In the palaeosol samples, NanoSIMS elemental mapping of the ions $^{12}C^-$, $^{16}O^-$, $^{18}O^-$, $^{28}Si^-$, $^{27}Al^{16}O^-$ and $^{56}Fe^{16}O^-$ were first carried out in conventional thin sections to identify kaolinite or smectite-rich areas. $^{16}O/^{18}O$ ratios were later obtained at selected 1 × 1 μm² points. The average $\delta^{18}O$ values obtained from four different samples for kaolinite ($\delta^{18}O_{SMOW}$=18 ±15‰ and $\delta^{18}O_{SMOW}$=14 ±4‰) and smectite ($\delta^{18}O_{SMOW}$=13 ±14‰ and $\delta^{18}O_{SMOW}$ =11 ±6‰) are consistent with crystallization in weathering conditions. However, the standard deviations of the measurements were relatively large, which may be due to the presence of epoxy resin, which degasses under vacuum conditions in the NanoSIMS chamber [1].

For glauconite and celadonite, focused ion beam–scanning electron microscopy (FIB-SEM) was used to extract two individual lamellae of each mineral (≈10 × 5 μm, 1 μm thick). This approach eliminated the need for preliminary NanoSIMS elemental mapping for mineral detection, but resulted in rapid consumption of the lamellae during isotopic analyses due to their small thickness. Elemental mapping of the ions $^{18}O^-$ and $^{16}O^-$ were obtained by NanoSIMS in 12 x 12 μm^2 areas rather than by point analyses. The average $\delta^{18}O$ values obtained for glauconite ($\delta^{18}O_{SMOW}$=32 ±6‰) and celadonite ($\delta^{18}O_{SMOW}$=24± 5‰) reflect a different temperature range for these minerals, but with a partial overlap in their formation conditions.

This study demonstrates that, despite the standard deviations, NanoSIMS could be applicable for analysing oxygen isotopic composition of coexisting small-size clay minerals within the same sample.

[1] Laita, E., et al. (2023). Chem. Geol. 615, 121213.