Time resolved in situ monitoring of mechanochemical transformations by X-ray spectroscopy (XAS)

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1. Introduction (section 1)

Mechanochemical reactions promise a new direction for environmentally benign preparation of materials, and has been dubbed by IUPAC as one of the 10 chemical innovations that will change our world [1]. Despite this significant promise, very little is known about the mechanisms that drive mechanochemical transformations, posing significant barriers to realizing their full potential. To this end, there is growing need to follow mechanochemical reactions in situ and in real time. We here describe advances in the development and application of XAS methods to monitor material synthesis in real time under mechanochemical conditions. We demonstrate the generality of our approaches by describing mechanochemical syntheses of materials by both vibratory ball milling and by Resonant Acoustic Mixing (RAM), where a time resolution of 1 second is for a whole XAS spectrum was achieved. Moreover, we describe how spectroscopic methods can be coupled to diffraction-based approaches (Figure 1), thereby providing new dimensions in understanding mechanochemical synthesis [2].

2. Results

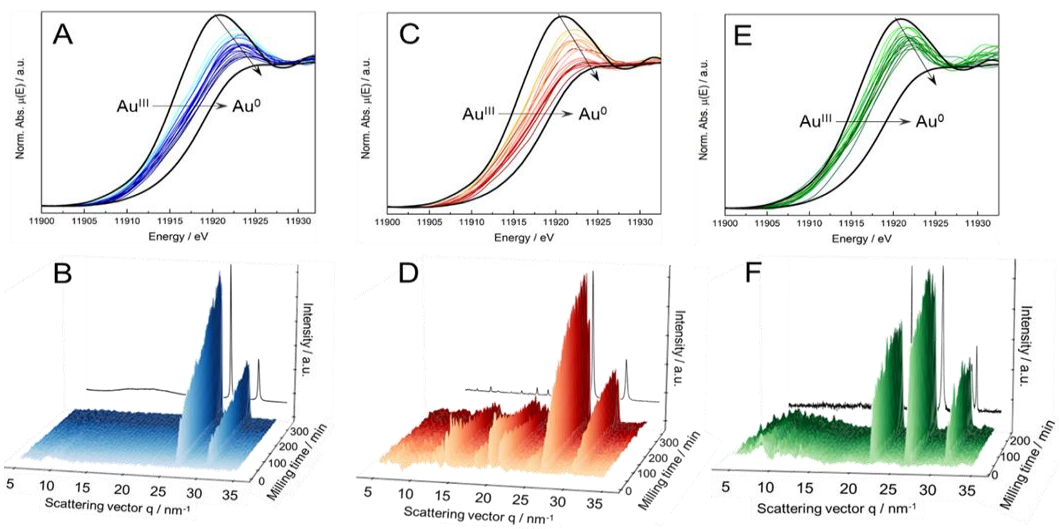

Figure 1 - Time-resolved-XANES (Au-LIII edge) (top) and time-resolved-XRD patterns (bottom) for the synthesis of Au NPs under ball milling conditions using hydroquinone (HQ) (A and B), ascorbic acid (AA) (C and D) and NaBH4 (E and F) as reducing agents. The black spectra in A, C and E correspond to the AuIII (HAuCl4·3H2O) and Au0 (foil) standards. The XRD patterns shown as the black traces in B, D, and F are data from ex situ measurements after the milling period.

3. References

[1] F. Gomollón-Bel, Chem. Int., 2019, 41, 12–17
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