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Mineralogical characterization of magnesium-based nanoparticles recovered from a swirl-stabilized magnesium flame by analytical and scanning/transmission electron microscopy

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S.1 Optical microscopy

The sample appeared as an agglomerate of small white particles (Fig. S.1).

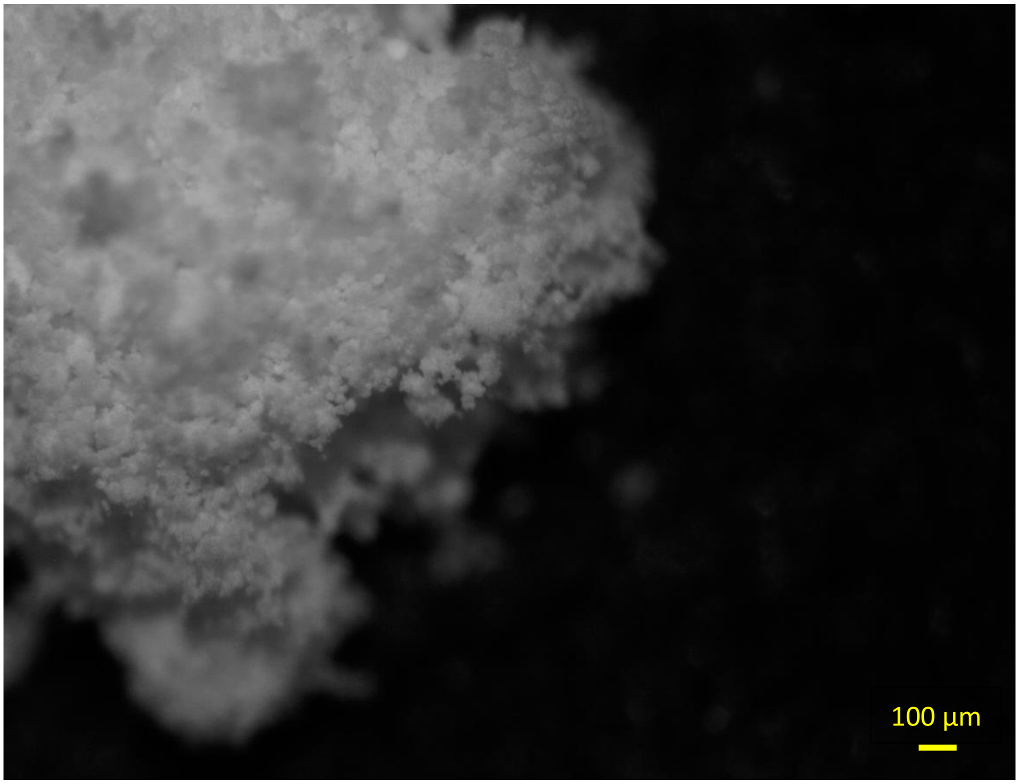

Figure S.1: MgO powder observed by optical microscopy on a cardboard surface.

S.2 SEM-EDXS

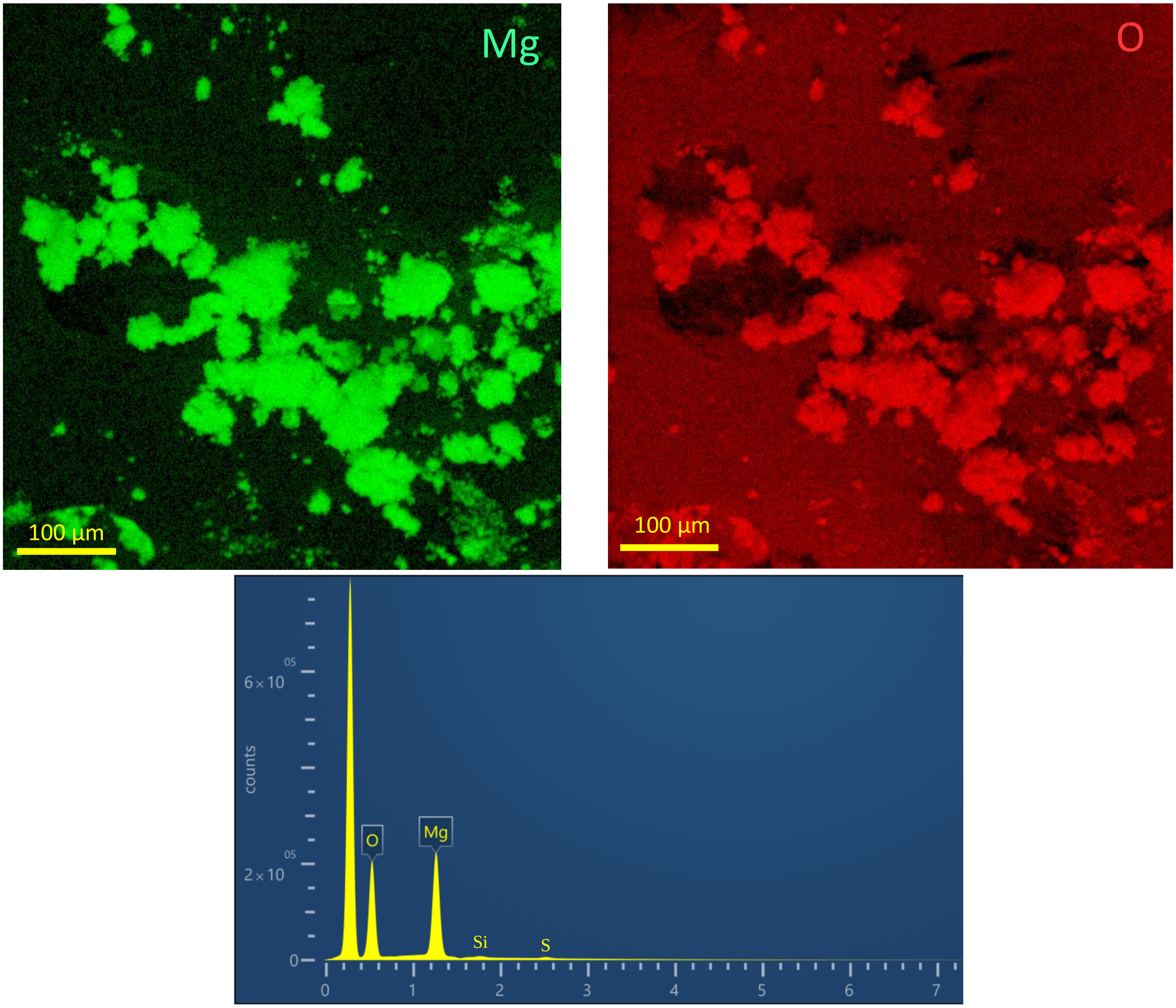

Figure S.2.1: EDXS map collected on MgO aggregate (Mg green, and O red), and related EDXS spectrum (elements shown in the mapping are within the blue squares).

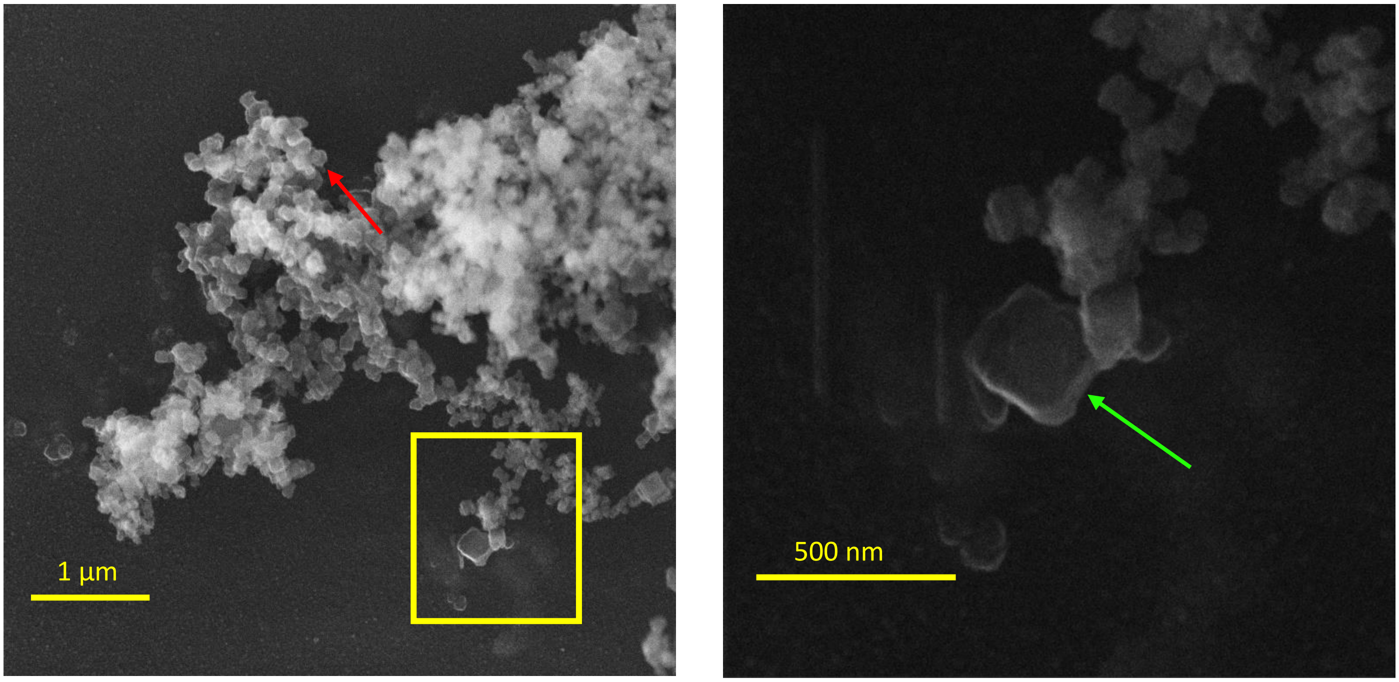

Fig. S.2.2: a) SEM SE image of MgO aggregates. Energy 30 keV, beam current 5 pA, spot size 8.67 nm. The yellow box indicates the ROI. b) Zoom in the ROI.

Particles having a projected hexagonal shape (e.g. red arrow) resulted more difficult to detect at this level of investigation (Fig. S.2.2). This might be a consequence of the resolution limit of the instrument, the similarity with rounded shapes and, possibly, as a consequence of the relatively low conductivity of the sample. The more frequent and easily recognizable particles having a squared projected shape (e.g. green arrow) were identified through SEM SE images. These squared particles show rounded stepped-like edges.

The listed limitations did not allow us to obtain high-quality high-resolution images of our sample (e.g. Fig. 2.2 b, 350,000x magnification) in SEM SE mode, thus we have focused our study on ARM (see the main text on the manuscript).

S.3 TEM: morphologies and dimensional distribution

We have detected the rare presence of particles having a more elongated rectangular projection (Fig. S.3.1a). These particles were distinguishable from the squared projection of the more commonly observed cubic structures. Often, particles were melded together by shapeless amorphous material (Fig. S.3.1a) or crystalline to poorly crystalline material (Fig. S.3.1b).

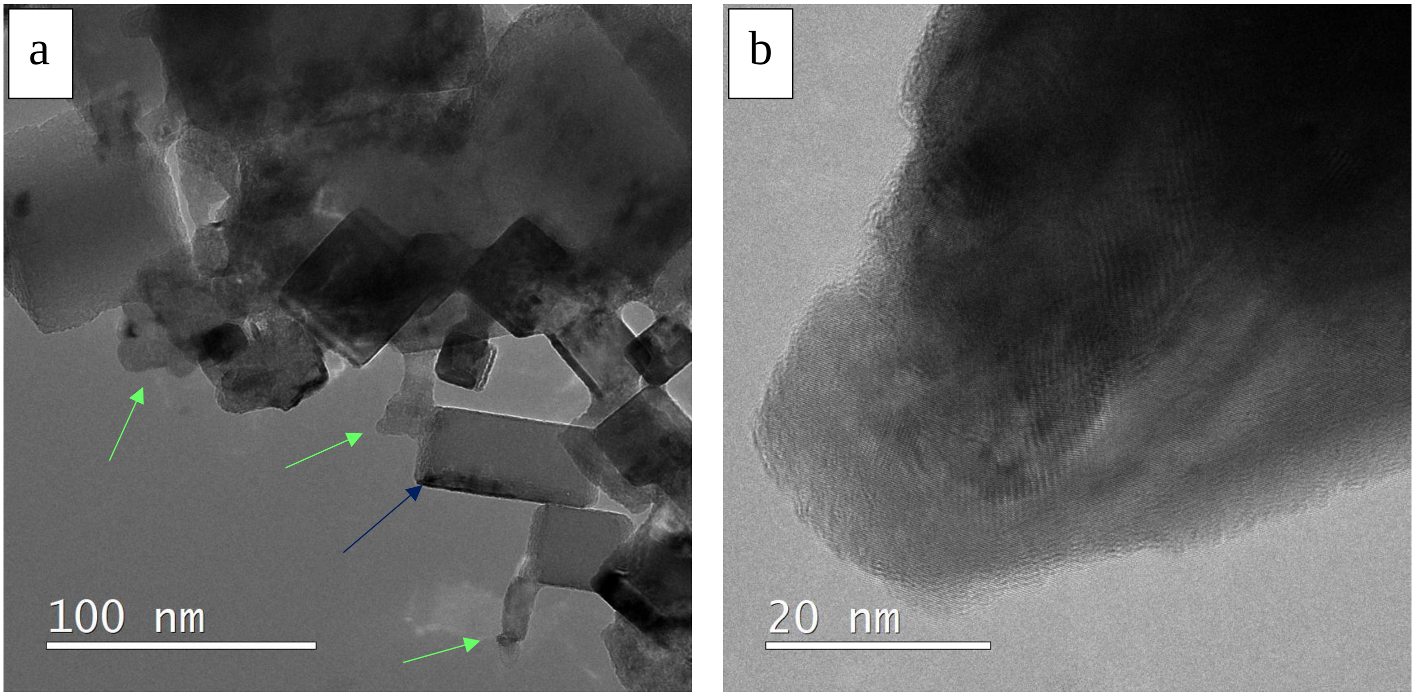

Figure S.3.1: (a) BF-TEM images showing shapeless amorphous connective material (light green arrows) and a rectangular particle (dark blue arrow), (b) a detail of the shapeless, mostly amorphous connective material.

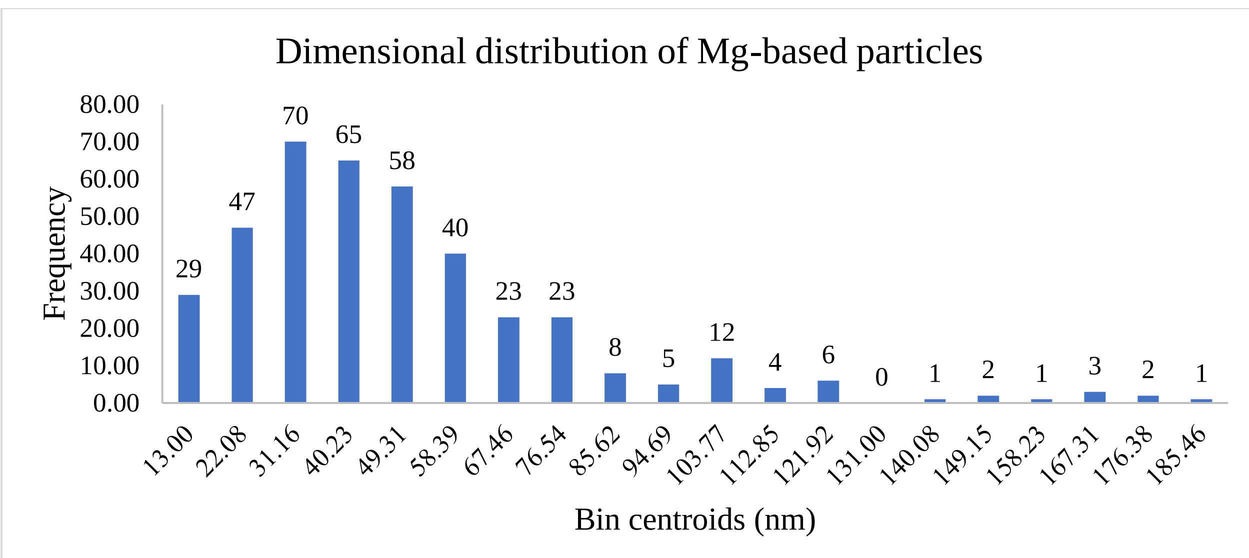

Figure S.3.2: Dimensional distribution (D_{ev}) of individual Mg-based particles measured at the TEM.

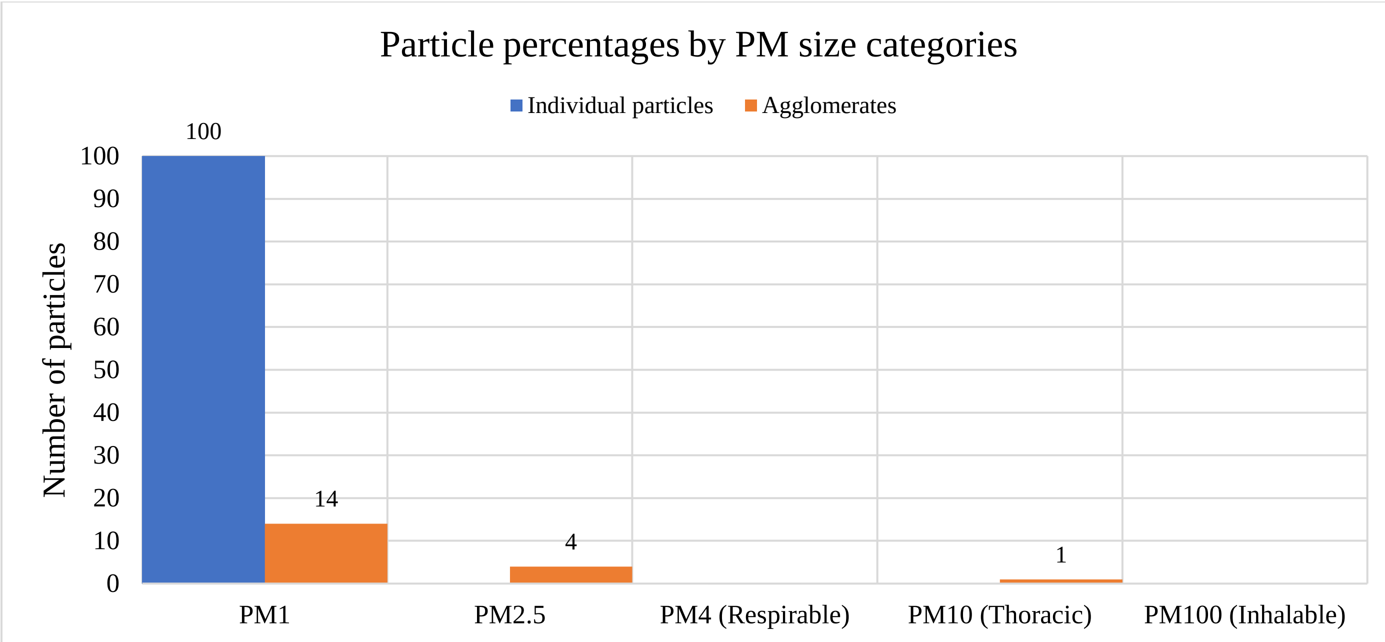

Figure S.3.3: Percentage of particles that belong to a specific particle-size category based on their D_{ev}.

S.4 SAED measured d-spacing summary

Tab. S.4: Additional d-spacings (in Å) measured on TEM-SAED patterns. The d_{hkl} values highlighted in red represent the spacing of spots measured on complex diffraction patterns, in which there was a large number of spots that we were not able to reconduct to a specific symmetry and/or that were overlapped to polycrystalline patterns.

	Rings (d_{hkl} in descending order)											
Photographic film number	1st - d_{hkl}	2nd - d_{hkl}	3rd - d_{hkl}	4th - d_{hkl}	5th - d_{hkl}	6th - d_{hkl}	7th - d_{hkl}	8th - d_{hkl}	9th - d_{hkl}	10th - d_{hkl}	11th - d_{hkl}	12th - d_{hkl}
77	2.41 d_{111}	2.06 d_{200}	1.42 d_{220}	1.15	0.96	0.86	-	-	-	-	-	-
89	2.02 d_{200}	1.80	1.60	1.45 d_{220}	1.31	-	-	-	-	-	-	-
90	2.09 d_{200}	1.57	1.22	1.13	0.93	0.85	-	-	-	-	-	-
94	2.36 d_{111}	2.29	2.25	2.03 d_{200}	1.97	1.85	1.49	1.32	1.18	1.10	0.91	0.85
98	2.48 d_{111}	2.17	2.00 d_{200}	1.91	1.81	1.41 d_{220}	1.32	1.14	1.06	0.88	-	-

S.5 STEM-EDXS Mapping

The chemical mapping performed on aggregates showed a homogeneous distribution of Mg and O signals.

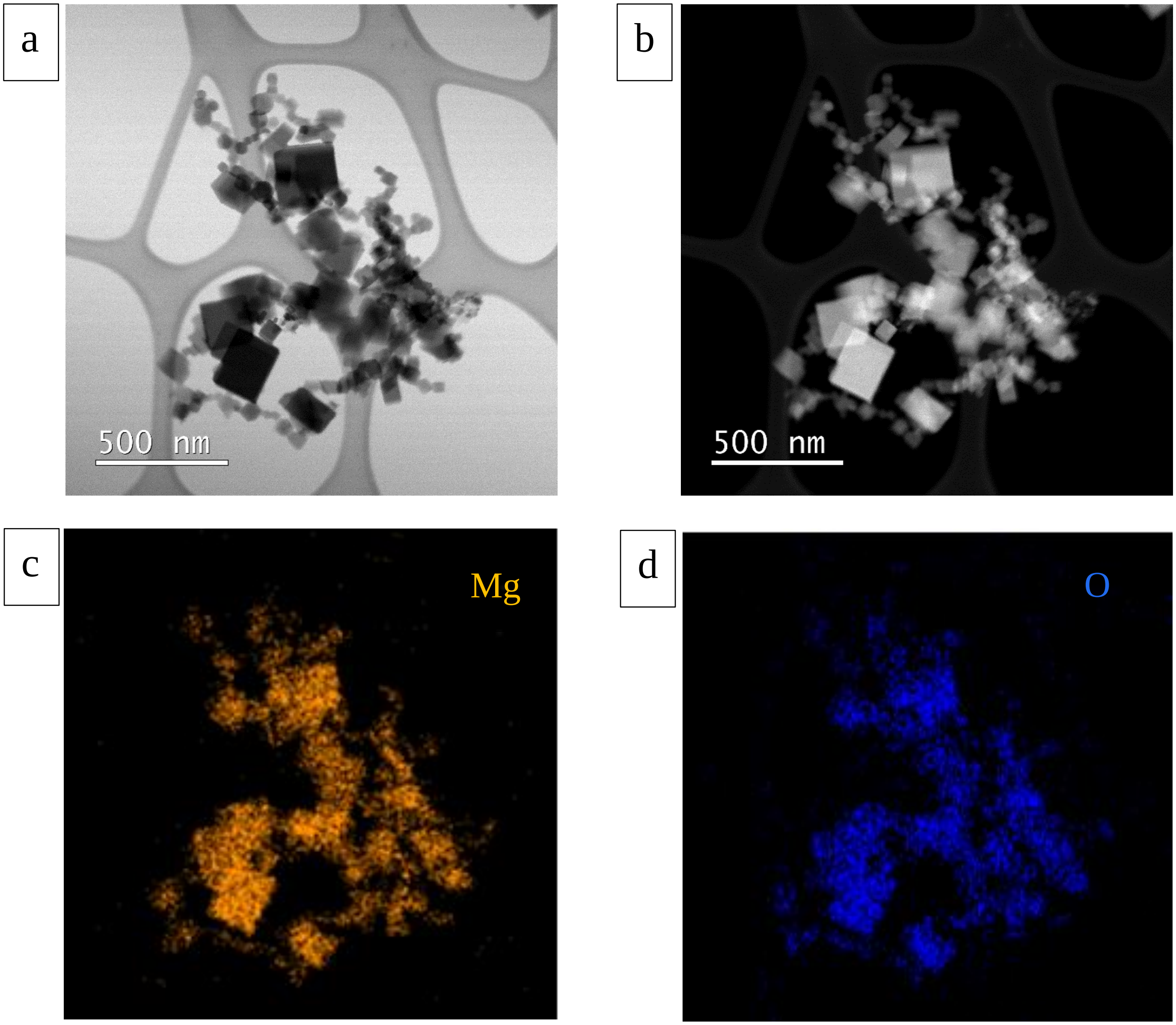

Figure S.5.1: An aggregate of Mg-based particles viewed in STEM (a) Bright Field (BF) and (b) Medium Angle Annular Dark Field (MAADF). The chemical maps (EDXS) collected on the same aggregate show the presence of (c) Mg and (d) O. The chemical maps scale is omitted.

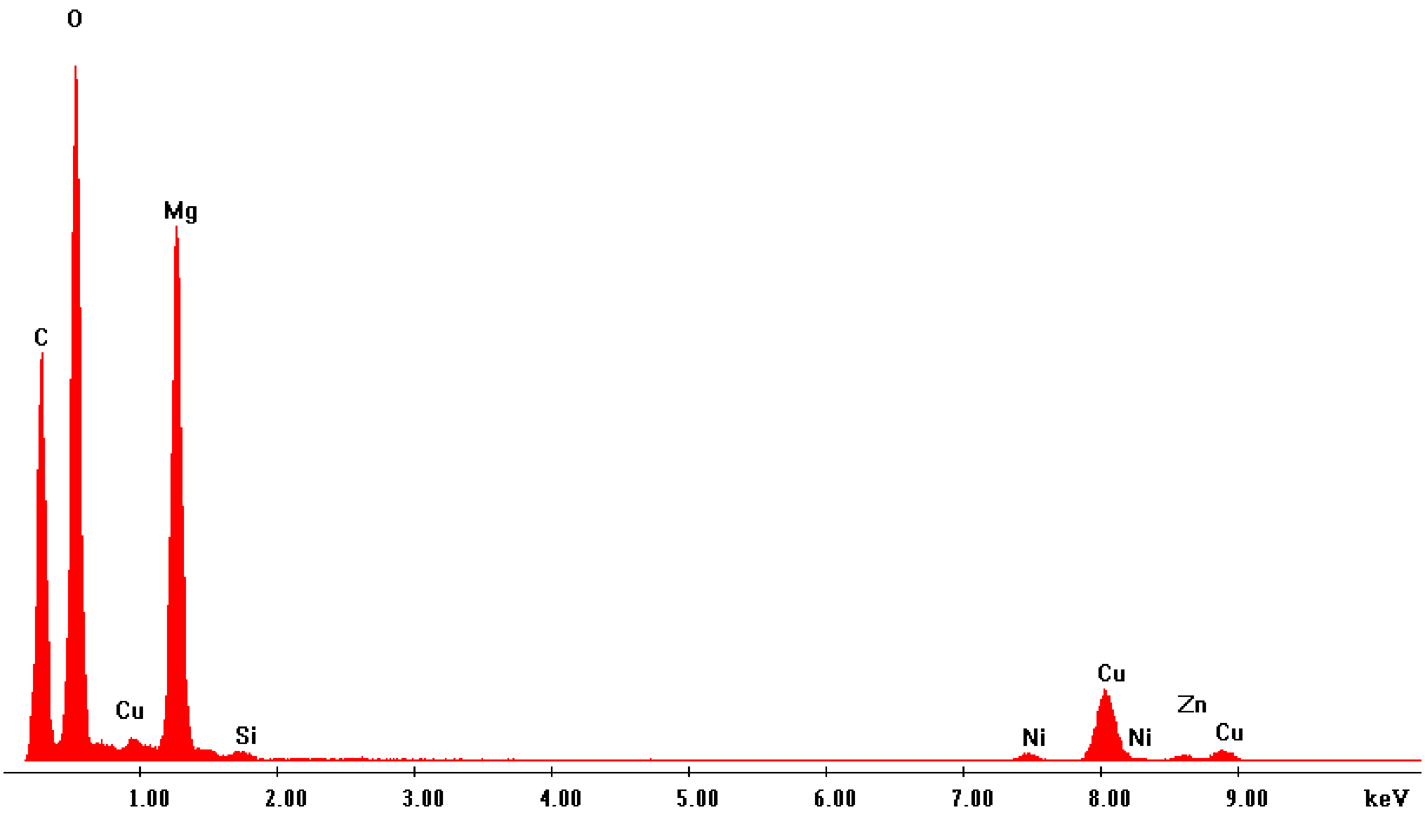

Fig. S.5.2 The EDXS spectrum collected on a representative aggregate. Fe was occasionally detected in some of the aggregates, but this aspect will be studied using the ARM (The C, Si, Ni, Cu and Zn signals are generated by the TEM grid).