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Supplementary Information

**In situ evidence of mineral physical protection and carbon
stabilization revealed by nanoscale 3-D tomography**

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3-D tomography computation and illustration

The final 3-D tomographic structures for visualization and illustration are generated using *Amira* 3-D software for post-image processing (Fig. 1). The reconstructed datasets first go through the *Median* and the *Gauss* filter processes to

1 enhance the S/N ratio before 3-D computation. To eliminate the noise surrounding the
2 reconstructed datasets, the *LabelField* function is used to define a 3-D mask for the
3 specimens of interest. The *Arithmetic* function is used to segment the specimens from
4 the surrounding noise according to the 3-D mask. After the above-mentioned post-
5 image processing, the dataset is illustrated using *Voltex* and *Isosurface*. In general, OC
6 is demonstrated by *Voltex* with a proper contrast value, and minerals and gold particles
7 with high intensity are shown by *Isosurface* with a reasonable threshold. Organic C and
8 minerals are bound in the specific spatial region using *SelectRoi*. The *CameraRotate*
9 module is used to show the rotating motion of tomography along a specific axis. The
10 internal structure of the specimen is shown under the *ClippingPlane* module. The
11 *DemoMaker* module is applied to make an animated sequence of operations for
12 advanced movie recording, and *MovieMaker* is used to export the animated operation
13 to a video file.

14

15 **Supplementary Figure Captions**

16 **Figure S1.** The flowchart for 3-D tomography reconstruction and subsequent 3-D
17 computation for illustration using TXM. Reconstructed 3-D tomography datasets are
18 generated based on the measured distributions, and 3-D tomography illustration is
19 generated by the image post-process and computation.

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2 **Figure S2.** The 2-D X-ray absorption-contrast composite image in all focus depths for
3 OC-mineral consortium from Mt. Nanhua. The grey scale is proportional to the X-ray
4 attenuation coefficients of different materials. The dark laminal texture pointed by the
5 white arrow reveals a sheet-like mineral coating on OC surface, which is a dense and
6 thin layer, likely indicating a high level of physical protection. We propose such texture
7 originates from adsorption. The red arrow points to the other distinct texture of OC-
8 mineral nano-aggregates/clusters, with dark minerals in the core and light OC
9 surrounding, which is not necessarily on the rim. This texture indicates a possible
10 microsite of OC-mineral co-precipitation. It should be noted that the light region
11 surrounding the dark region (minerals) could be either OC or air/cavity, as their
12 attenuation coefficients are very difficult to distinguish from each other in X-ray images.
13 There are numerous such OC-mineral clusters in the image, with a large, round-shaped
14 one chosen for illustration purposes. In reality, the OC-mineral clusters can be of any
15 shape. The scale bar is 3 microns.

16

17 **Figure S3.** A cross-sectional view of the reconstructed 3-D tomography, under
18 absorption-contrast mode for the OC-mineral consortium from Mt. Nanhua, along the
19 X-Z plane. The grey scale is inversely proportional to the X-ray attenuation coefficients
20 of different materials. The white and the red arrows point to the mineral layer and OC-

1 mineral clusters respectively. The yellow arrow points to a potential nucleus for mineral
2 clusters development that could be a light material such as OC. On the other hand, this
3 microsite may be a cavity. The scale bar is 3 microns.

4
5 **Figure S4.** The X-ray diffraction patterns of the particulate OC and the mountain soil.

6 There is a distinct difference between the particulate OC and the mountain soil in
7 mineralogy as shown in the stacking graph. The major crystal phases in the soil is quartz
8 (ICDD 0-033-1161), amesite (ICDD 01-080-1772), and muscovite (ICDD 04-012-
9 1956). The X-ray diffraction pattern also shows a few minor phases in the mountain
10 soil, such as chlorite (ICDD 01-075-8791), phlogopite (ICDD 00-016-0344), magnetite
11 (ICDD 01-080-6407), and hematite (ICDD 01-080-5408), but these diffraction peaks
12 are hardly recognizable due to their low intensity.

13
14 **Supplementary Movie (Video clips) Captions**

15 **Figure SMOV1.** Video illustration extracted from 3-D absorption-contrast tomography
16 of lab-made BC and mineral nanoparticle consortium. The yellow particle is a gold
17 nanoparticle for position reference. All minerals are shown in a silver color. The dark
18 grey part contours the structure and boundary of OC.

19 <https://doi.org/10.5446/36090>

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21 **Figure SMOV2.** Video illustration extracted from 3-D phase-contrast tomography of
22 lab-made BC and mineral nanoparticle consortium. The yellow particle is a gold
23 nanoparticle for position reference. All minerals are shown in a silver color. The dark

1 grey part contours the structure and boundary of OC.

2 <https://doi.org/10.5446/36091>

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4 **Figure SMOV3.** Video illustration obtained from 3-D absorption-contrast tomography
5 of the particulate mineral-bearing OC from the mountain soil. The yellow particle is a
6 gold nanoparticle for position reference. All minerals are shown in a rust color. The
7 dark grey part contours the structure and boundary of OC.

8 <https://doi.org/10.5446/36092>

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1 **Table S1.** XRD peak positions of mineral-bearing OC sample from Mt. Nanhua.

	d (Å)	d-reference (Å)	hkl
Ferrihydrite	2.5644	2.5634	100
	2.2502	2.2504	012
	2.0046	1.9840	013
	1.7344	1.7322	014
	1.5090	1.5160	015
	1.4779	1.4800	110
Goethite	4.9831	5.0000	020
	4.2063	4.2089	110
	2.6992	2.7071	130
	2.5914	2.5913	021
	2.4595	2.4591	111
	2.2625	2.2624	121
	1.7210	1.7284	221
	1.6990	1.7005	240
	1.5650	1.5706	151
1.5135	1.5150	002	
Lepidocrocite	6.2651	6.2700	200
	3.2921	3.2940	210
	2.4747	2.4730	301
	2.4333	2.4340	410
	2.3616	2.3620	111
	1.9402	1.9400	501
	1.9370	1.9350	020
	1.7367	1.7350	511
	1.5333	1.5340	002
	1.5258	1.5240	321
1.3684	1.3710	521	
Quartz	4.2532	4.254	100
	3.3422	3.342	101
	2.4571	2.456	110
	2.2806	2.280	102
	2.2361	2.236	111
	1.9788	1.979	201
	1.8173	1.817	112
1.6715	1.671	202	

1.5412

1.541

211

1.3818

1.374

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1 **Table S2.** FTIR peak assignment of mineral-bearing OC sample from Mt. Nanhua.

Wavenumber (cm⁻¹)	Model	Reference	Ref. value
1758	Carbonyl C=O stretching	Parikh et al., 2014	1765
1706	Aromatic carbonyl/carboxyl C=O stretching	Özçimen and Ersoy- Meriçboyu, 2010	1709
1596	ν C=C in aromatic	Sharma et al., 2004	1597
1454	CH deformation and aromatic ring vibrations	Sharma et al., 2004	1460
1386	Carboxyl C–O symmetric stretching	Parikh et al., 2014	1384
1274	Carboxyl C–O stretching	Parikh et al., 2014	1280
1247	ν (C-O) phenolic	Parikh et al., 2014	1240
1113	Si–O stretching	Vaculikova et al., 2011	1113
1062	Si–O stretching	Harsh et al., 2002	1060
1025	Aliphatic ether C–O and alcohol C–O stretching	Parikh et al., 2014	1029
910	OH deformation	Vaculikova et al., 2011	913
875	1 adjacent H deformation	Parikh et al., 2014	870
798	2 adjacent H deformation	Parikh et al., 2014	804
754	4 adjacent H deformation	Parikh et al., 2014	750
694	Fe-OH stretching	Blanch et al. 2008	690
674	In-plane O-H bend	Blanch et al. 2008	670
626	Fe–O stretching	Blanch et al. 2008	633
534	Fe-OH stretching	Blanch et al. 2008	533
497	Fe–O asymmetric stretching	Blanch et al. 2008	497
476	Fe-O vibrations	Parikh et al., 2014	480

2 (Blanch et al., 2008; Harsh et al., 2002; Özçimen and Ersoy-Meriçboyu, 2010; Parikh
3 et al., 2014; Sharma et al., 2004; Vaculíková et al., 2011)

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