

Supplementary Information

The Effects of Surfactants and Essential Oils on Microwave-Assisted Hydrothermal Synthesis of Iron Oxides

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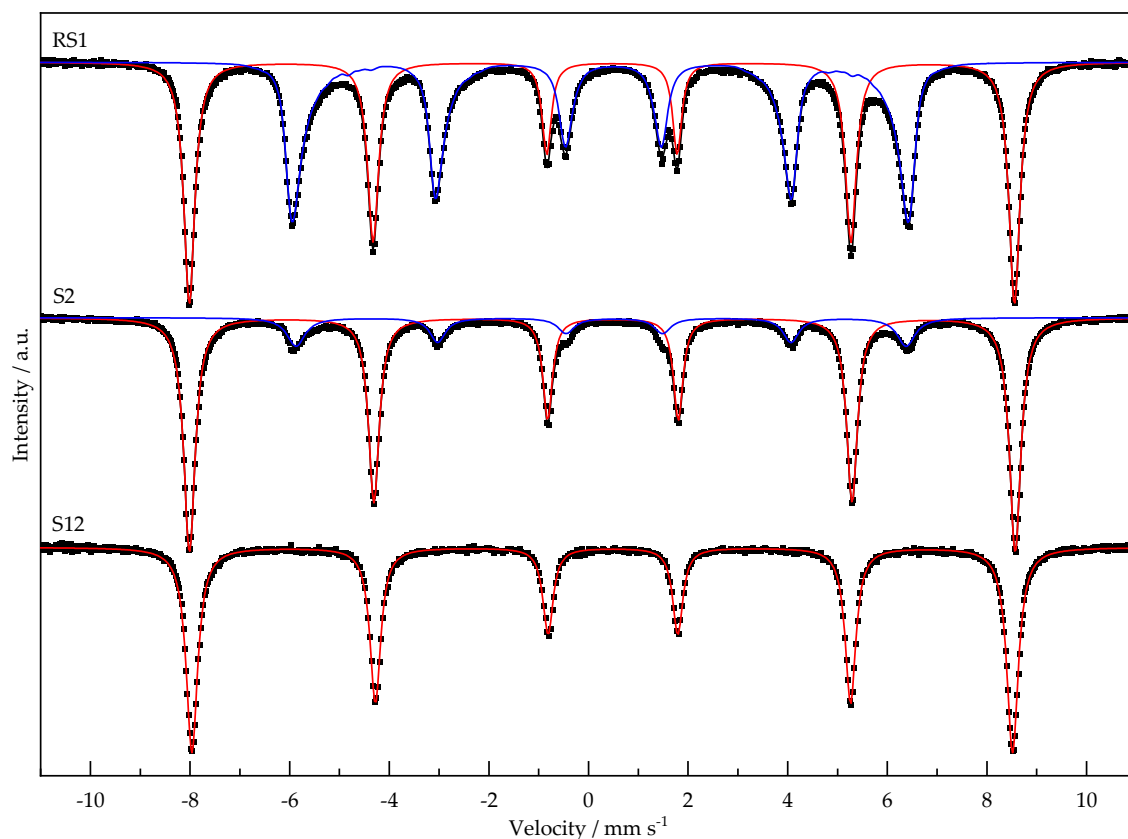
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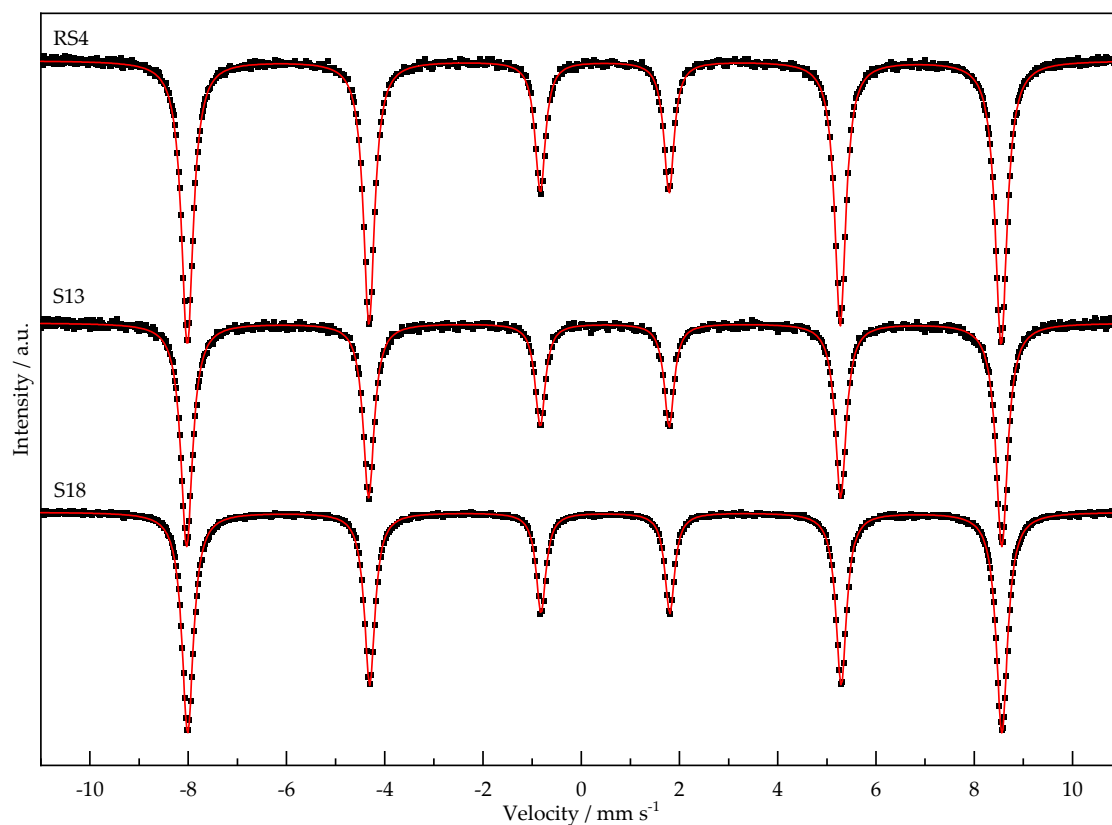
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⁵⁷Fe Mössbauer spectroscopy

Supplementary Figure S1 and Supplementary Figure S2 show ⁵⁷Fe Mössbauer spectra of prepared samples (recorded at 20 °C), while Supplementary Table S1 lists the Mössbauer parameters determined by fitting the recorded spectra with the MossWinn program [1]. No additional polymorphic forms of goethite phase were observed. Mössbauer spectra of the reference sample RS1, prepared by the microwave treatment at 200 °C for 20 min, and sample S2, prepared under the same conditions with addition of PEG (Supplementary Figure S1), show the presence of two sextets with Mössbauer parameters (Supplementary Table S1) that can be attributed to magnetically ordered Fe(III) oxyhydroxide goethite and Fe(III) oxide hematite phases [2], in line with PXRD results. The Mössbauer spectrum of sample S12, prepared under the same conditions with the addition of lavender essential oil, however, contains only a sextet of pure hematite phase in a weakly ferromagnetic state, which is typical for hematite at RT [2]. The Mössbauer spectra of reference sample RS4 and samples S13 and S18, prepared by the microwave treatment at 260 °C for 5 min, show the presence of the sextet characteristic for pure hematite phase in a weakly ferromagnetic state [2] regardless of the addition of PEG in sample S13 or lavender essential oil in sample S18. The slightly increased hyperfine magnetic field (B_{hf}) in samples S13 and S18 can be attributed to the improved hematite crystallinity in the samples prepared with PEG and lavender essential oil compared to the reference sample. This is in line with the typical crystallite size found in these samples using Rietveld refinement of PXRD data.



Supplementary Figure S1. ^{57}Fe Mössbauer spectra (recorded at 20 °C) of reference sample RS1, sample S2 with 4.5% PEG and sample S12 with 4.5% lavender essential oil, prepared at 200 °C for 20 min.



Supplementary Figure S2. ^{57}Fe Mössbauer spectra (recorded at 20 °C) of reference sample RS4, sample S13 with 1% PEG and sample S18 with 1% lavender essential oil, prepared at 260 °C for 5 min.

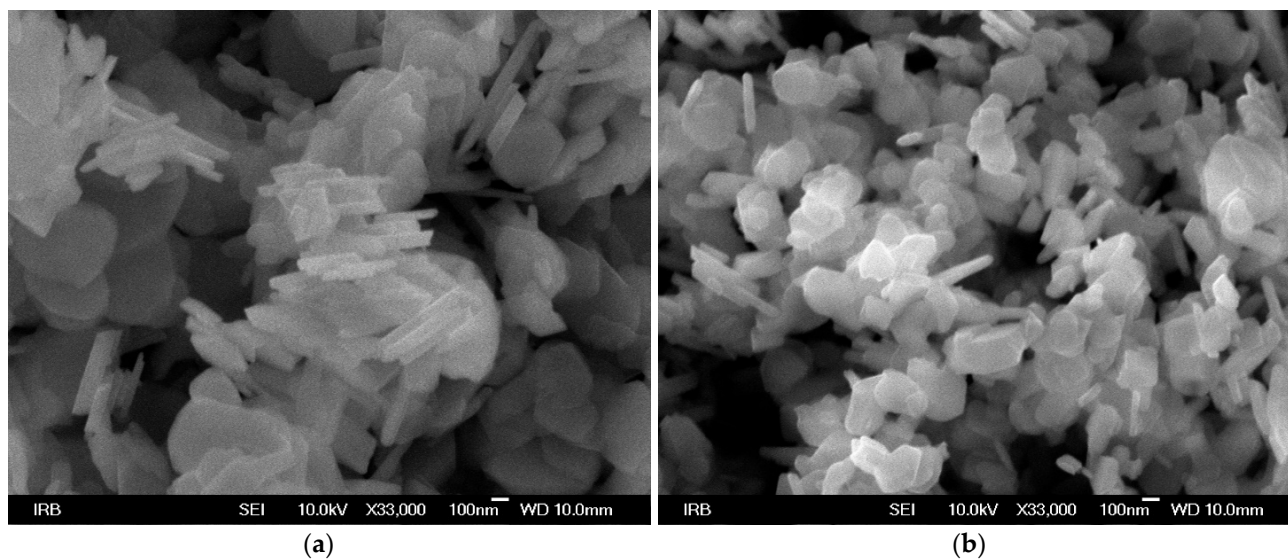
Supplementary Table S1. Mössbauer parameters for reference samples and samples with PEG and lavender essential oil, prepared at 200 °C for 20 min and 260 °C for 5 min.

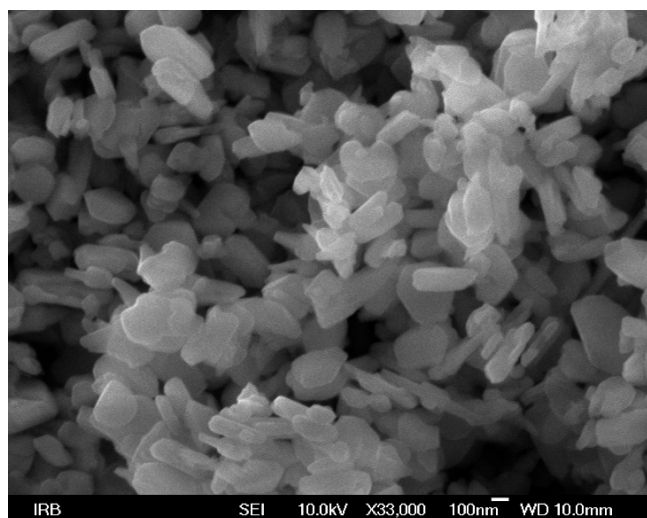
Sample	$\delta/\text{mm s}^{-1}$	$\Delta E_Q/\text{mm s}^{-1}$	B_{hf}/T	Area/%	Phase
RS1	0.37	−0.20	51.49	49.59	$\alpha\text{-Fe}_2\text{O}_3$
	0.37	−0.26	37.37	50.41	$\alpha\text{-FeOOH}$
S2	0.37	−0.21	51.53	82.94	$\alpha\text{-Fe}_2\text{O}_3$
	0.37	−0.27	38.08	17.06	$\alpha\text{-FeOOH}$
S12	0.38	−0.21	51.21	100.00	$\alpha\text{-Fe}_2\text{O}_3$
RS4	0.37	−0.21	51.49	100.00	$\alpha\text{-Fe}_2\text{O}_3$
S13	0.37	−0.21	51.57	100.00	$\alpha\text{-Fe}_2\text{O}_3$
S18	0.38	−0.22	51.51	100.00	$\alpha\text{-Fe}_2\text{O}_3$

δ – isomer shift relative to $\alpha\text{-Fe}$, B_{hf} – hyperfine magnetic field, ΔE_Q – quadrupole splitting

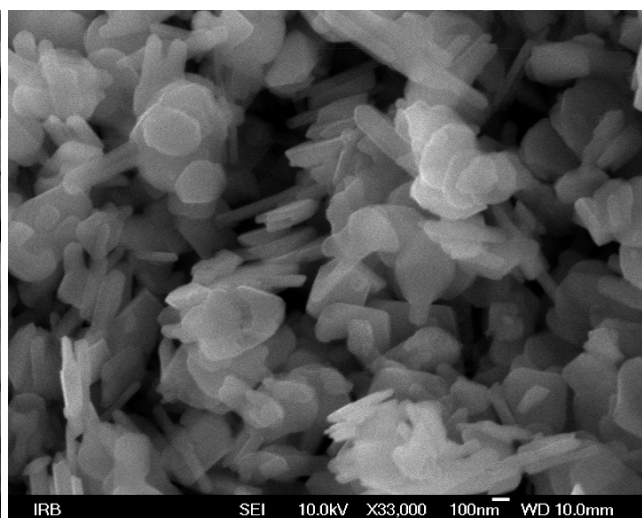
SEM analysis

The FE-SEM images of other samples that are not shown in article, prepared at 200 °C for 20 min with addition of surfactants and essential oil are shown in Supplementary Figure S3, while images of samples prepared at 260 °C for 5 minutes are shown at Supplementary Figure S4.

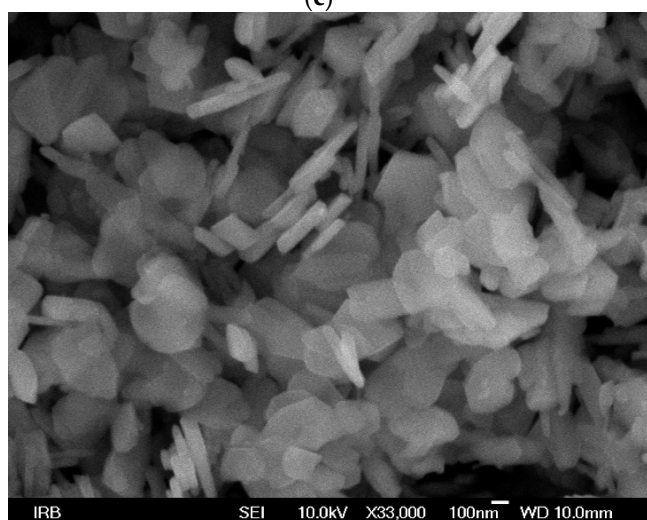




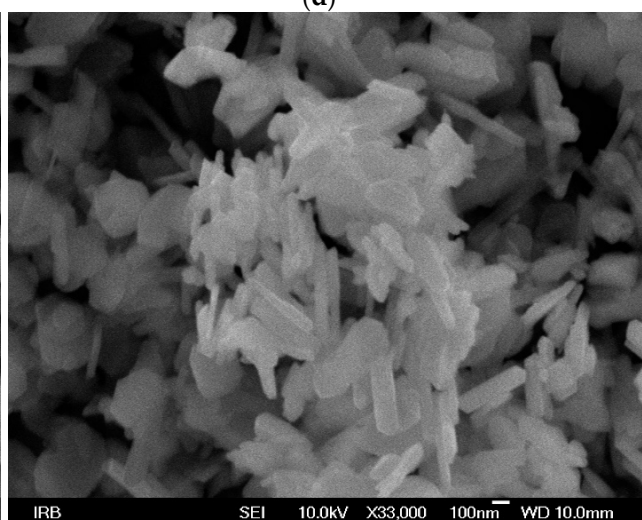
(c)



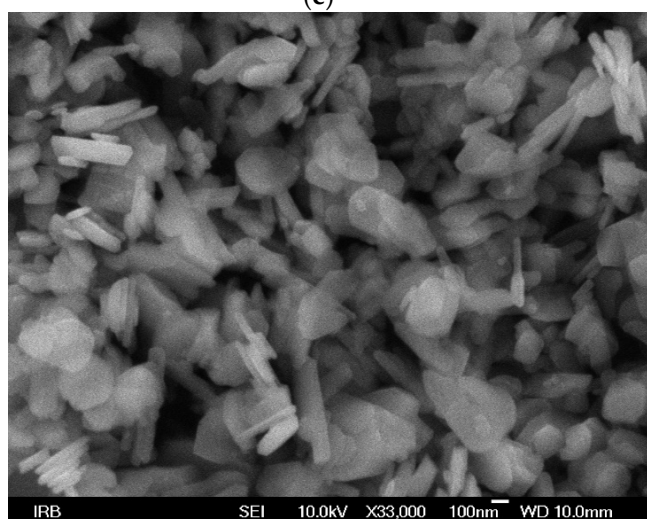
(d)



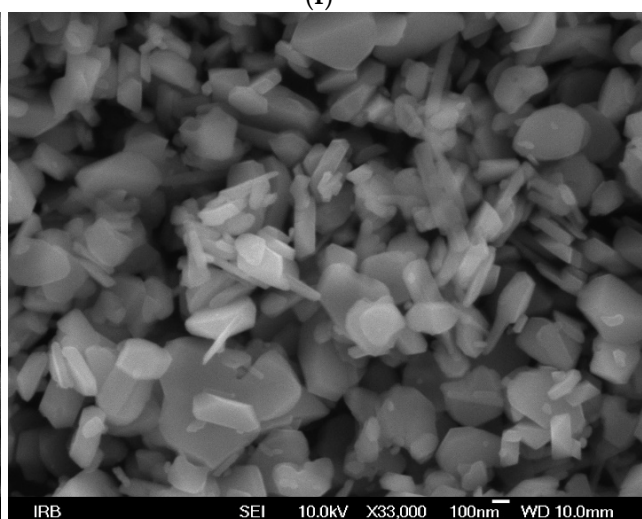
(e)



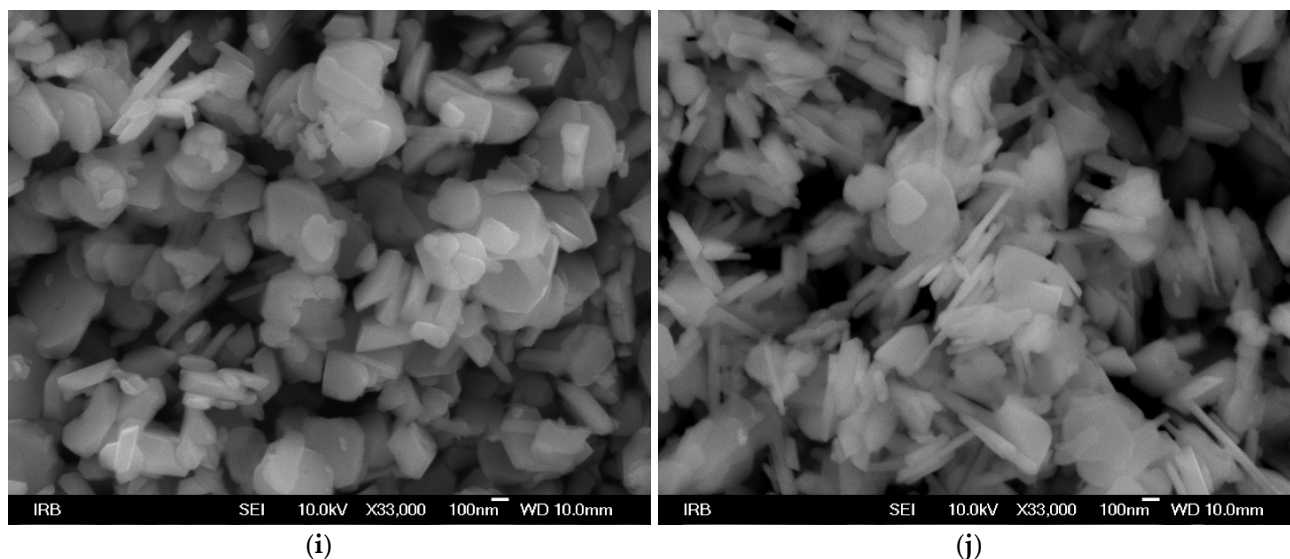
(f)



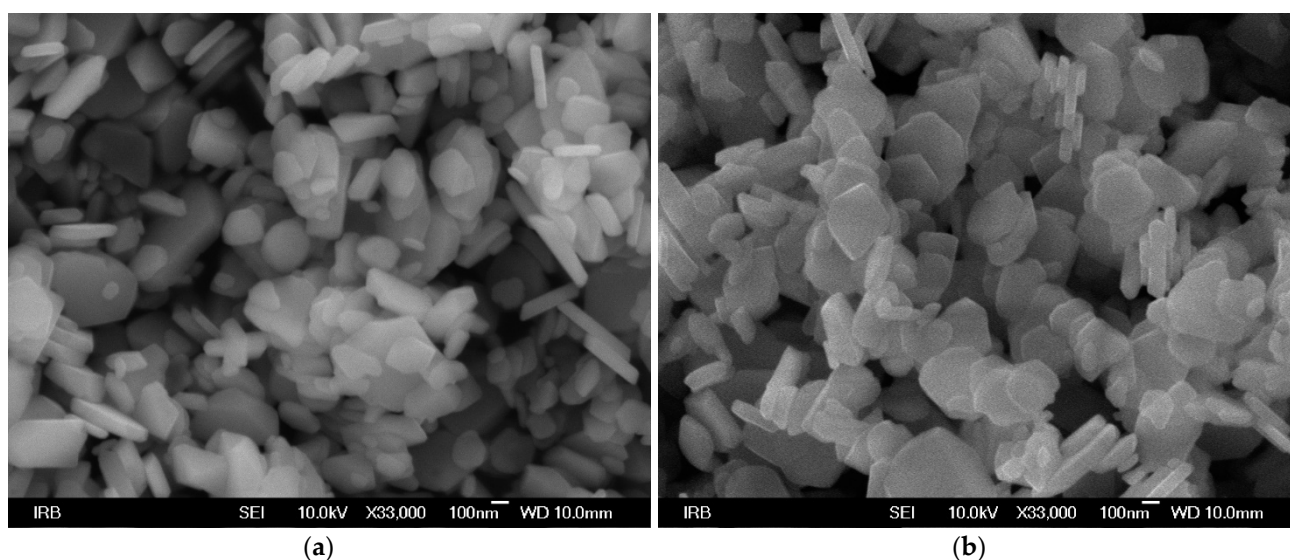
(g)

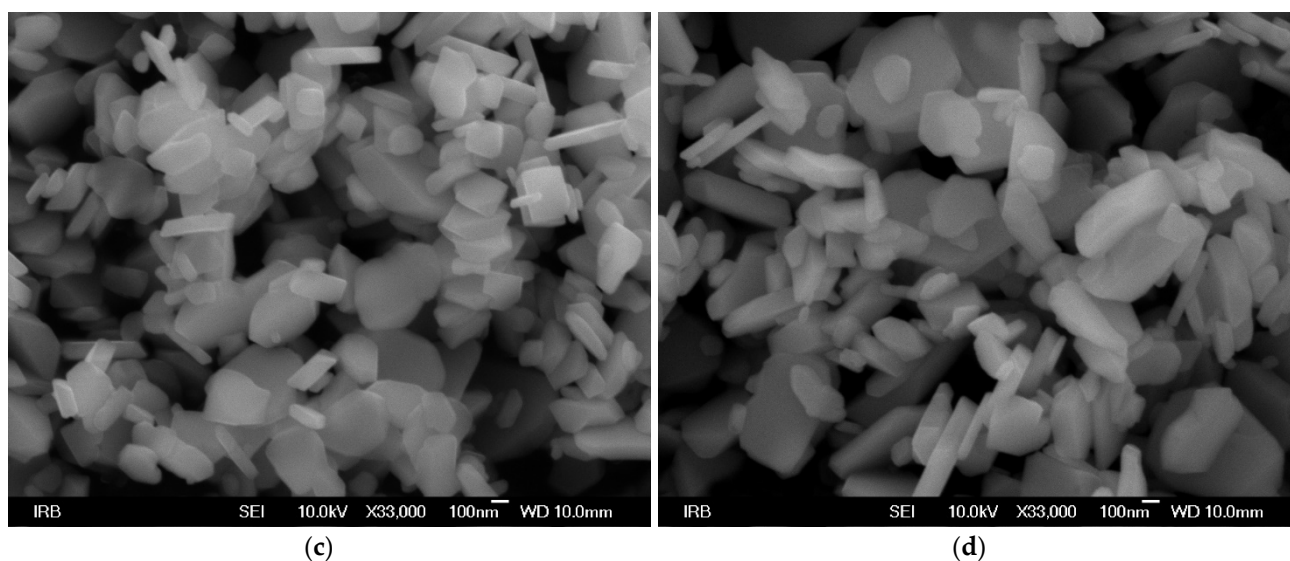


(h)



Supplementary Figure S3. The FE–SEM images of (a) sample S1 with 1% PEG, (b) sample S3 with 1% NGS, (c) sample S4 with 4.5% NGS, (d) sample S5 with 1% SDS, (e) sample S6 with 4.5% SDS, (f) sample S7 with 1% sage essential oil, (g) sample S8 with 4.5% sage essential oil, (h) sample S9 with 1% rosemary essential oil, (i) sample S10 with 4.5% rosemary essential oil and (j) sample S11 with 1% lavender essential oil, prepared at 200 °C for 20 min, taken at 33000 × magnification.





Supplementary Figure S4. The FE-SEM images of (a) sample S14 with 1% NGS, (b) sample S15 with 1% SDS, and (c) sample S16 with 1% sage essential oil and (d) sample S17 with 1% rosemary essential oil, prepared at 260 °C for 5 min, taken at 33000 × magnification.

References

1. Klencsár, Z.; Kuzmann, E.; Vértés, A. User-friendly software for Mössbauer spectrum analysis. *J. Radioanal. Nucl. Chem.* **1996**, *210*, 105–118, <https://doi.org/10.1007/BF02055410>.
2. Murad, E.; Cashion, J. In *Mössbauer Spectroscopy of Environmental Materials and Their Industrial Utilization*; Kluwer Academic Publishers: Boston, 2004.