

One-Step Synthesis of Iron and Titanium-Based Compounds Using Black Mineral Sands and Oxalic Acid under Subcritical Water Conditions

Carla S. Valdivieso-Ramírez ¹, Patricia I. Pontón ¹, Anja Dosen ², Bojan A. Marinkovic ^{2,*} and Victor H. Guerrero ^{1,*}

¹ Department of Materials, Escuela Politécnica Nacional, Quito 170525, Ecuador; carla.valdivieso@epn.edu.ec (C.S.V.-R.); patricia.ponton@epn.edu.ec (P.I.P.)

² Department of Chemical and Materials Engineering, Pontifical Catholic University of Rio de Janeiro (PUC-Rio), Rio de Janeiro 38097, Brazil; adosen@puc-rio.br

* Correspondence: bojan@puc-rio.br (B.A.M.); victor.guerrero@epn.edu.ec (V.H.G.)

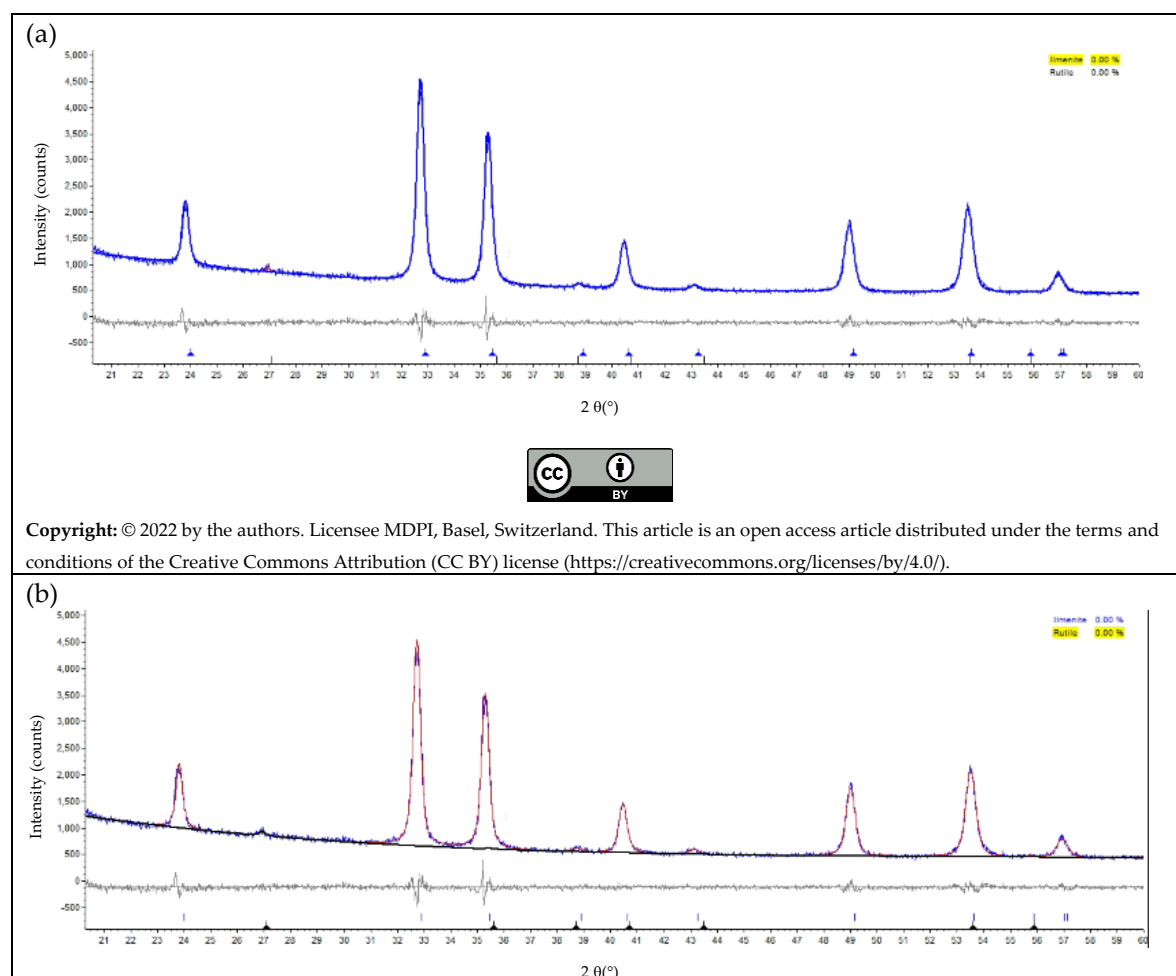


Figure S1. Le Bail fitting of SCW sample: (a) the main phase is an ilmenite-hematite solid solution, and (b) the secondary phase is rutile.

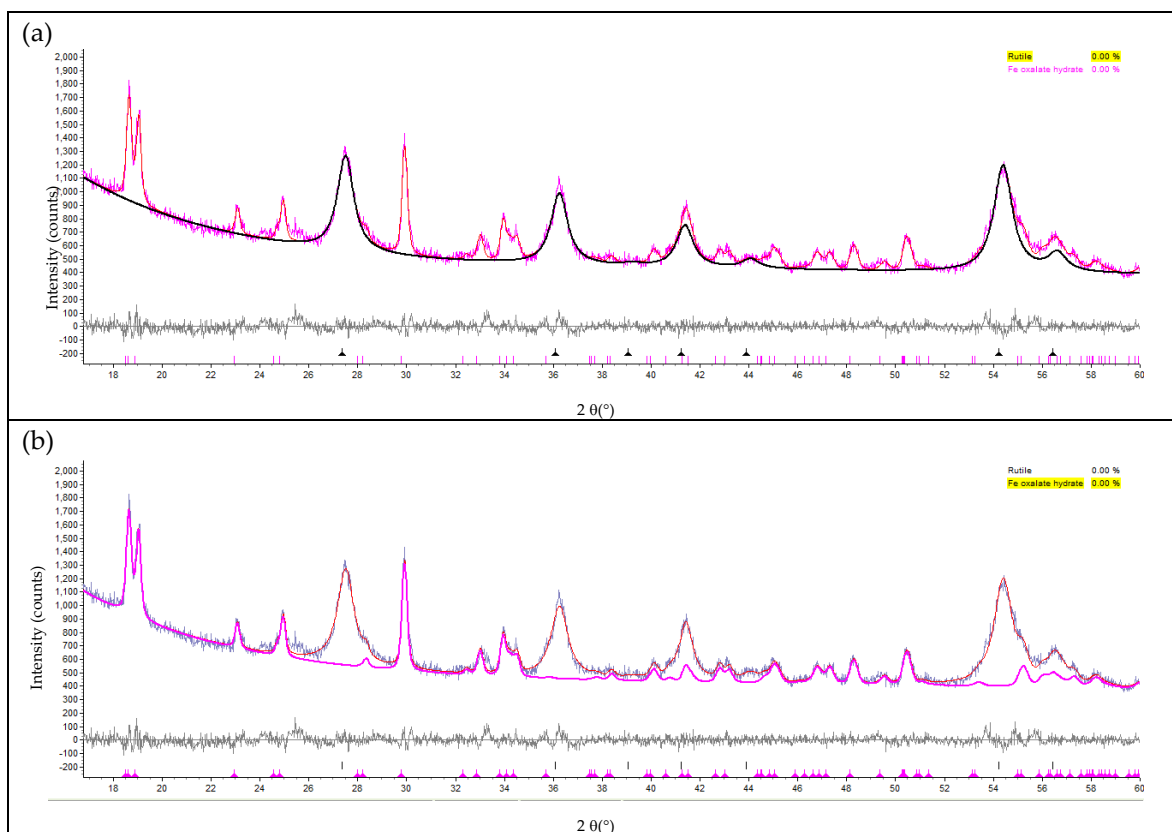


Figure S2. Le Bail fitting of WA fraction for 0.1 M, demonstrating the presence of (a) rutile and (b) Fe oxalate hydrate (PDF 23-0293, $a = 9.84 \text{ \AA}$).

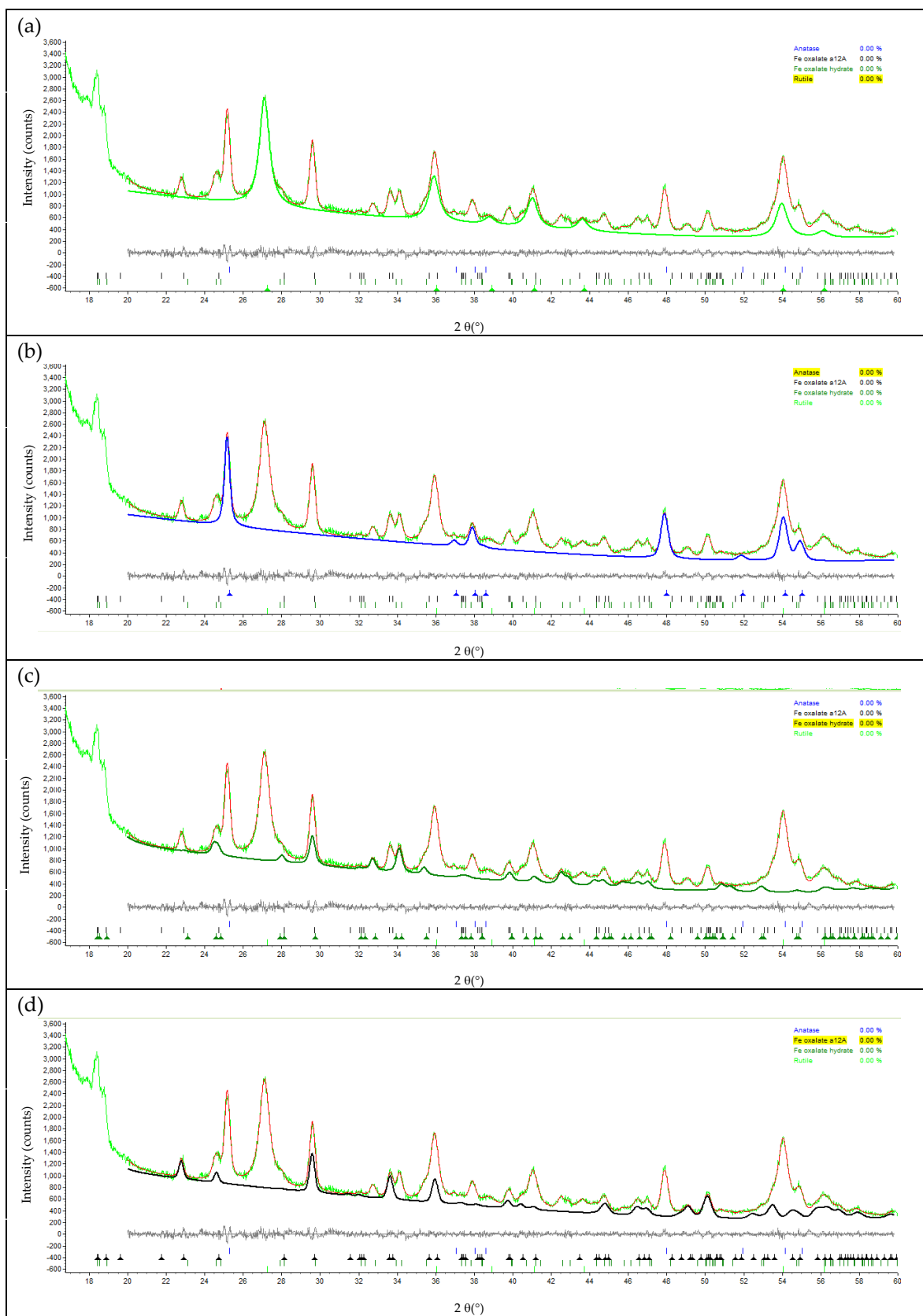


Figure S3. Le Bail fitting of WA fraction for 0.5 M, showing the presence of (a) rutile, (b) anatase, (c) ferrous oxalate hydrate (PDF 23-0293, $a=9.84 \text{ \AA}$), and (d) ferrous oxalate hydrate (PDF 72-1305, $a = 12.06 \text{ \AA}$).

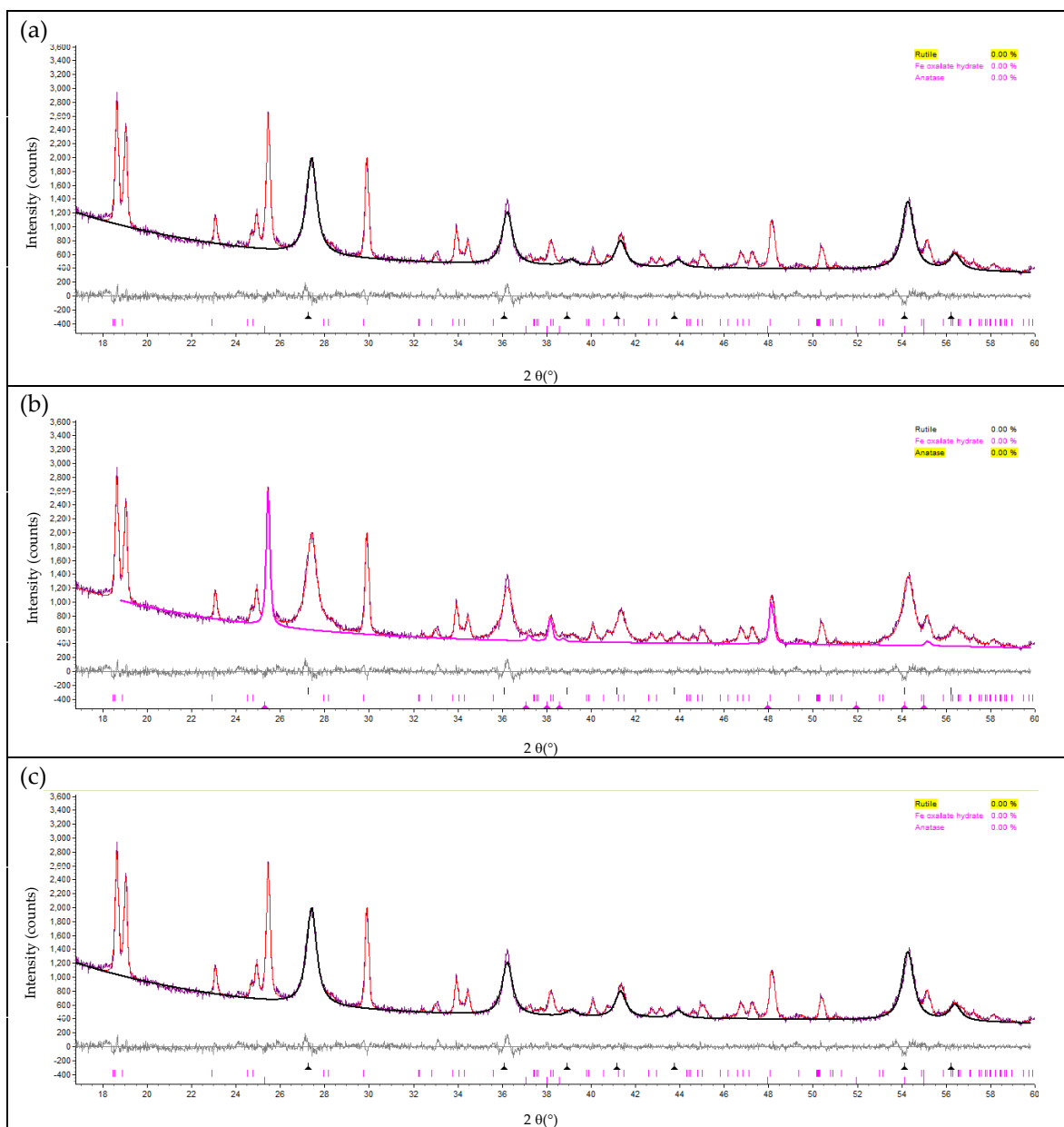


Figure S4. Le Bail fitting of WA fraction for 1.0 M, showing the presence of (a) rutile, (b) anatase and (c) ferrous oxalate hydrate (PDF 23-0293, $a = 9.84 \text{ \AA}$).

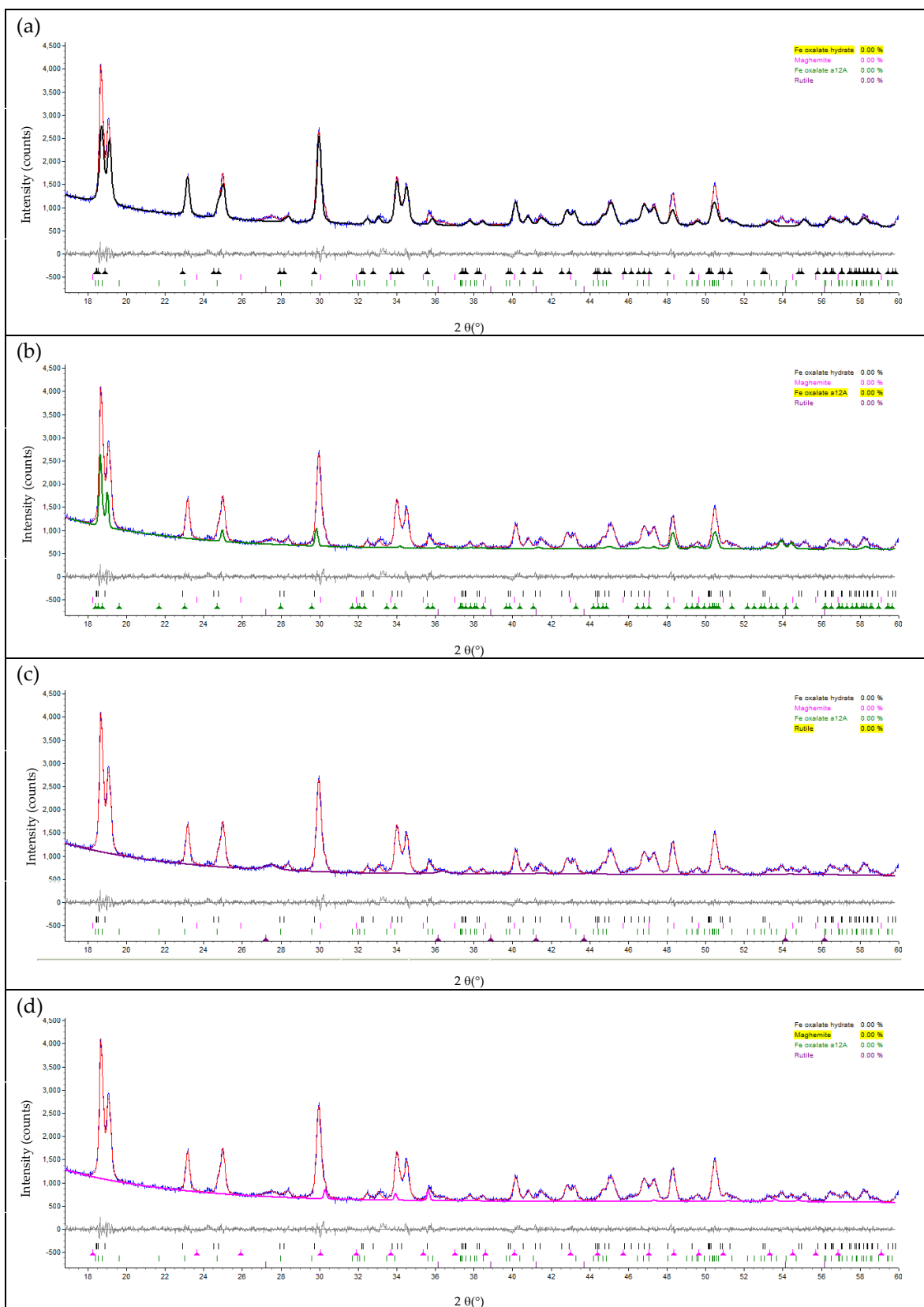


Figure S5. Le Bail fitting of WB fraction for 0.1 M, showing the presence of main phases: (a) ferrous oxalate hydrate (PDF 23-0293, $a = 9.84 \text{ \AA}$), (b) ferrous oxalate hydrate (PDF 72-1305, $a = 12.06 \text{ \AA}$), and secondary phases: (c) rutile and (d) maghemite.

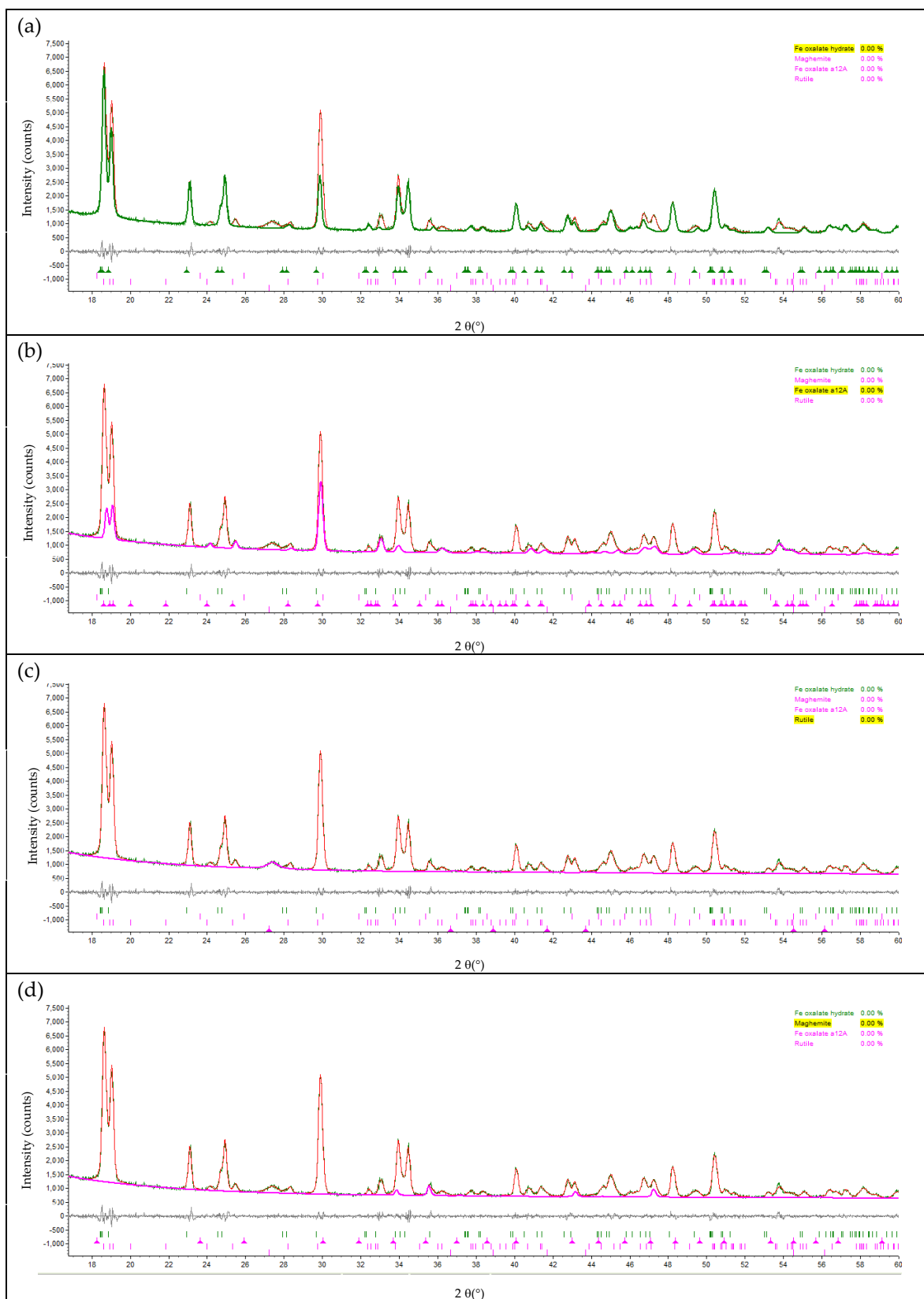


Figure S6. Le Bail fitting of WB fraction for 0.5 M, showing the presence of main phases: (a) ferrous oxalate hydrate (PDF 23-0293, $a = 9.84 \text{ \AA}$), (b) ferrous oxalate hydrate (PDF 72-1305, $a = 12.06 \text{ \AA}$), and secondary phases: (c) rutile and (d) maghemite.

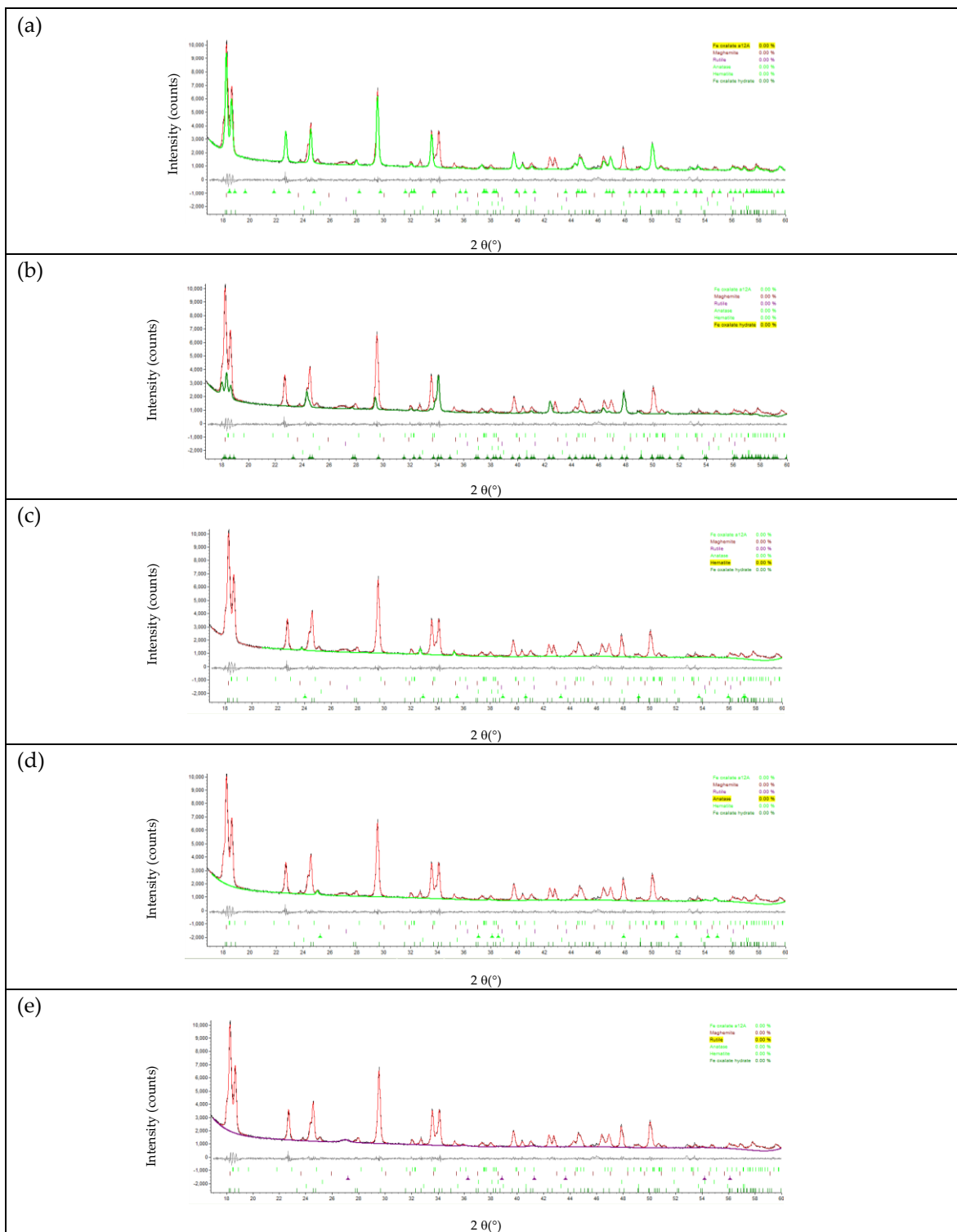
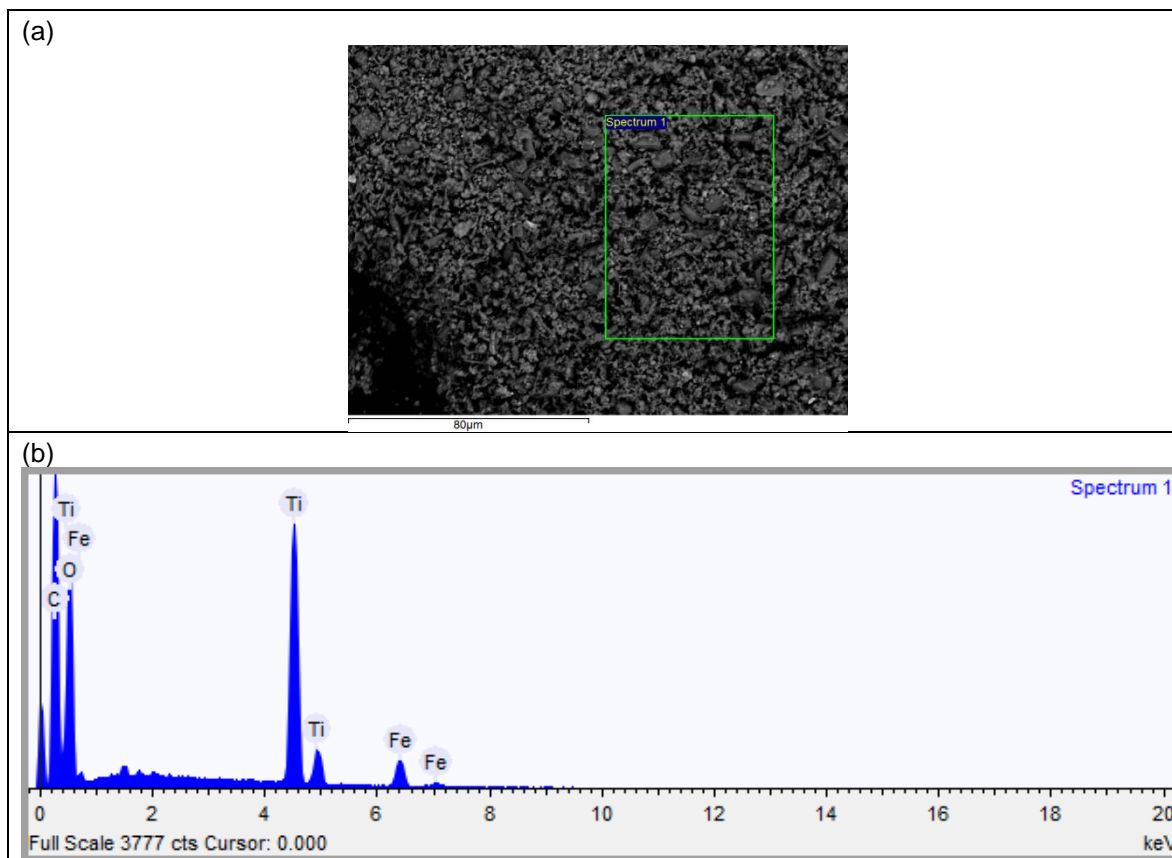


Figure S7. Le Bail fitting of WB fraction for 1.0 M, showing the presence of main phases: (a) ferrous oxalate hydrate (PDF 72-1305, $a = 12.06 \text{ \AA}$), (b) ferrous oxalate hydrate (PDF 23-0293, $a = 9.84 \text{ \AA}$), and secondary phases: (c) hematite, (d) anatase and (e) maghemite.



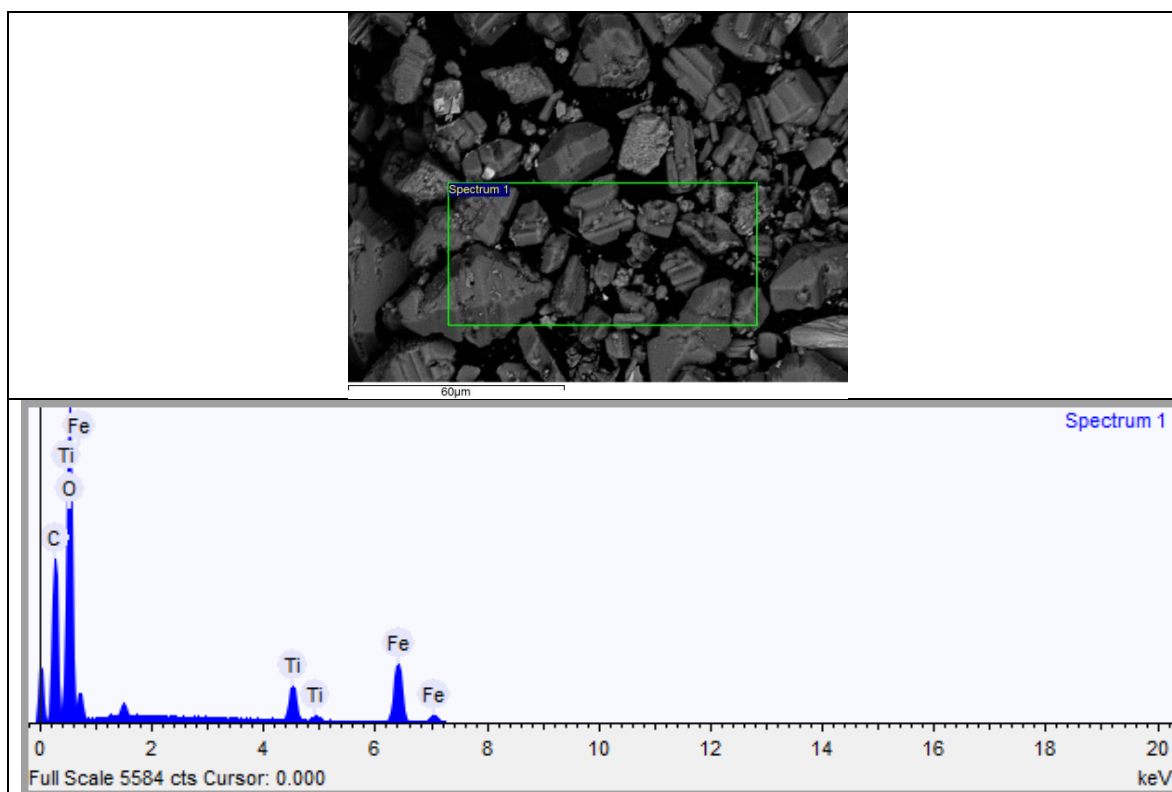


Figure S9. (a) EDS mapping and (b) EDS spectrum of WA fraction for 1.0 M.

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IRON OXALATE HYDRATE

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Single-crystal X-ray diffraction and spectroscopic studies on
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 $'-x,3/4+y,3/4+z'$
 $'-x,1/4+y,1/4+z'$
 $'1/2-x,3/4+y,1/4+z'$
 $'1/2-x,1/4+y,3/4+z'$
 $'z,3/4-x,3/4-y'$
 $'z,1/4-x,1/4-y'$
 $'1/2+z,3/4-x,1/4-y'$
 $'1/2+z,1/4-x,3/4-y'$
 $'-y,3/4+z,3/4+x'$
 $'-y,1/4+z,1/4+x'$
 $'1/2-y,3/4+z,1/4+x'$
 $'1/2-y,1/4+z,3/4+x'$
 $'x,3/4-y,3/4-z'$
 $'x,1/4-y,1/4-z'$
 $'1/2+x,3/4-y,1/4-z'$

$'1/2+x,1/4-y,3/4-z'$
 $'1/4-x,1/2+z,3/4-y'$
 $'1/4-x,+z,1/4-y'$
 $'3/4-x,1/2+z,1/4-y'$
 $'3/4-x,+z,3/4-y'$
 $'1/4+z,1/2-y,3/4+x'$
 $'1/4+z,-y,1/4+x'$
 $'3/4+z,1/2-y,1/4+x'$
 $'3/4+z,-y,3/4+x'$
 $'1/4-y,1/2+x,3/4-z'$
 $'1/4-y,+x,1/4-z'$
 $'3/4-y,1/2+x,1/4-z'$
 $'3/4-y,+x,3/4-z'$
 $'1/4+x,1/2-z,3/4+y'$
 $'1/4+x,-z,1/4+y'$
 $'3/4+x,1/2-z,1/4+y'$
 $'3/4+x,-z,3/4+y'$
 $'1/4-z,1/2+y,3/4-x'$
 $'1/4-z,+y,1/4-x'$
 $'3/4-z,1/2+y,1/4-x'$
 $'3/4-z,+y,3/4-x'$
 $'1/4+y,1/2-x,3/4+z'$
 $'1/4+y,-x,1/4+z'$
 $'3/4+y,1/2-x,1/4+z'$
 $'3/4+y,-x,3/4+z'$
 $'3/4-x,3/4-z,y'$
 $'3/4-x,1/4-z,1/2+y'$
 $'1/4-x,3/4-z,1/2+y'$
 $'1/4-x,1/4-z,y'$
 $'3/4+z,3/4+y,-x'$
 $'3/4+z,1/4+y,1/2-x'$
 $'1/4+z,3/4+y,1/2-x'$
 $'1/4+z,1/4+y,-x'$
 $'3/4-y,3/4-x,z'$
 $'3/4-y,1/4-x,1/2+z'$
 $'1/4-y,3/4-x,1/2+z'$
 $'1/4-y,1/4-x,z'$
 $'3/4+x,3/4+z,-y'$
 $'3/4+x,1/4+z,1/2-y'$
 $'1/4+x,3/4+z,1/2-y'$
 $'1/4+x,1/4+z,-y'$
 $'3/4-z,3/4-y,x'$
 $'3/4-z,1/4-y,1/2+x'$
 $'1/4-z,3/4-y,1/2+x'$
 $'1/4-z,1/4-y,x'$
 $'3/4+y,3/4+x,-z'$
 $'3/4+y,1/4+x,1/2-z'$
 $'1/4+y,3/4+x,1/2-z'$
 $'1/4+y,1/4+x,-z'$

```

'-z,-x,-y'
'-z,1/2-x,1/2-y'
'1/2-z,-x,1/2-y'
'1/2-z,1/2-x,-y'
'y,z,x'
'y,1/2+z,1/2+x'
'1/2+y,z,1/2+x'
'1/2+y,1/2+z,x'
'-x,-y,-z'
'-x,1/2-y,1/2-z'
'1/2-x,-y,1/2-z'
'1/2-x,1/2-y,-z'
'z,x,y'
'z,1/2+x,1/2+y'
'1/2+z,x,1/2+y'
'1/2+z,1/2+x,y'
'-y,-z,-x'
'-y,1/2-z,1/2-x'
'1/2-y,-z,1/2-x'
'1/2-y,1/2-z,-x'
loop_
_atom_site_label
_atom_site_fract_x
_atom_site_fract_y
_atom_site_fract_z
_atom_site_occupancy
Fe-oct 0.50000 0.50000 0.50000 0.83333
Fe-tet 0.12500 0.12500 0.12500 1.00000
O 0.25000 0.25000 0.25000 1.00000

```

ILMENITE

```

data_global
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loop_
_publ_author_name
'Wechsler B A'
'Prewitt C T'
_journal_name_full 'American Mineralogist'
_journal_volume 69
_journal_year 1984
_journal_page_first 176
_journal_page_last 185
_publ_section_title
;
Crystal structure of ilmenite (FeTiO3) at high temperature and high pressure
P = 25.4 kbar
;
_database_code_amcsd 0000932

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```

_chemical_formula_sum 'Fe Ti O3'
_cell_length_a 5.0691
_cell_length_b 5.0691
_cell_length_c 13.9849
_cell_angle_alpha 90
_cell_angle_beta 90
_cell_angle_gamma 120
_cell_volume 311.209
_exptl_crystal_density_diffn 4.857
_symmetry_space_group_name_H-M 'R -3'
loop_
_space_group_symop_operation_xyz
  'x,y,z'
  '2/3+x,1/3+y,1/3+z'
  '1/3+x,2/3+y,2/3+z'
  'y,-x+y,-z'
  '2/3+y,1/3-x+y,1/3-z'
  '1/3+y,2/3-x+y,2/3-z'
  '-x+y,-x,z'
  '2/3-x+y,1/3-x,1/3+z'
  '1/3-x+y,2/3-x,2/3+z'
  '-x,-y,-z'
  '2/3-x,1/3-y,1/3-z'
  '1/3-x,2/3-y,2/3-z'
  '-y,x-y,z'
  '2/3-y,1/3+x-y,1/3+z'
  '1/3-y,2/3+x-y,2/3+z'
  'x-y,x,-z'
  '2/3+x-y,1/3+x,1/3-z'
  '1/3+x-y,2/3+x,2/3-z'
loop_
_atom_site_label
_atom_site_fract_x
_atom_site_fract_y
_atom_site_fract_z
_atom_site_U_iso_or_equiv
Fe 0.00000 0.00000 0.35562 0.00861
Ti 0.00000 0.00000 0.14637 0.00671
O 0.31596 0.02301 0.24537 0.00773

```