**Effects of peroxo precursors and annealing temperature on properties and photocatalytic activity of nanoscale titania**

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# Supplementary material

## X-ray diffraction patterns

**Fig. s1.** XRD patterns of titanyl sulphate treated with H2O2 and annealed at various temperatures: TSP250 (*1*), TSP600 (*2*), TSP800 (*3*) and TSP900 (*4*).



**Fig. s2.** XRD patterns of titanyl sulphate stored at room temperature for 48 h and annealed at 850 °C after treatment with H2O2 (TSP(48h)850) (*1*), (NH4)2S2O8 (TSAPS(48h)850) (*2*), and (NH2)2CO.H2O2 (TSUP(48h)850) (*3*).

## IR spectra



**Fig. s3.** IR spectra of titanyl sulphate treated with H2O2 and annealed at 550 °C (TSP550) (*1*) and 700 °C (TSP700) (*2*).



**Fig. s4.** IR spectra of titanyl sulphate treated with (NH2)2CO.H2O2 (TSUP25) (*1*) and annealed at 185 °C (TSUP185) (*2*), 515 °C (TSUP515) (*3*), and 850 °C TSUP850 (*4*).

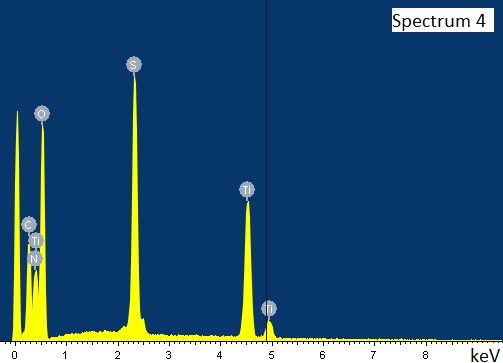
## Elemental analysis

Anal. Calcd for (NH2)2CO.H2O2 (%): C 12.77; H 6.38; N 29.79. Found (%): C 13.02; H 6.51; N 30.15. Anal. Calcd for 1.9TiO2 + (NH2)2CO.H2O2 (TSUP185) (%): C 4.88; H 2.44; N 11.38. Found (%): C 4.90; H 3.15; N 13.70. Anal. Calcd for TiO2 (TSUP515) (%): C 0; H 0; N 0. Found (%): C 0; H 0.32; N 0.94.

## X-ray microanalysis

Sample TSUP100

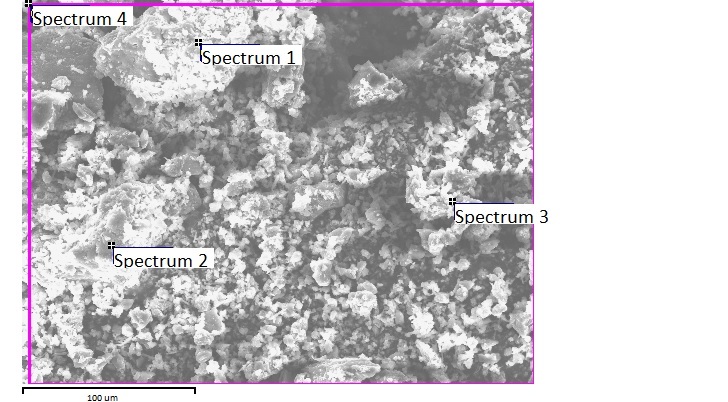


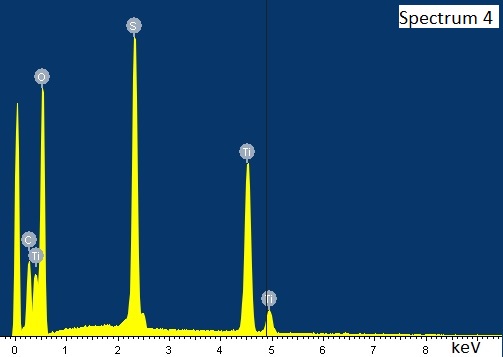


Atomic %

|  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- |
| Spectrum | C | N | O | S | Ti |
| 1 | 9.83 | 15.86 | 65.40 | 5.02 | 3.89 |
| 2 | 10.19 | 11.55 | 60.04 | 9.88 | 8.33 |
| 3 | 23.80 | 8.73 | 44.90 | 11.41 | 11.15 |
| 4 | 26.30 | 17.27 | 48.22 | 4.54 | 3.67 |
| Average | 17.53 | 13.35 | 54.64 | 7.71 | 6.76 |
| Standard deviation | 8.74 | 3.92 | 9.68 | 3.45 | 3.63 |
| Max | 26.30 | 17.27 | 65.40 | 11.41 | 11.15 |
| Min | 9.83 | 8.73 | 44.90 | 4.54 | 3.67 |

Sample TSUP160

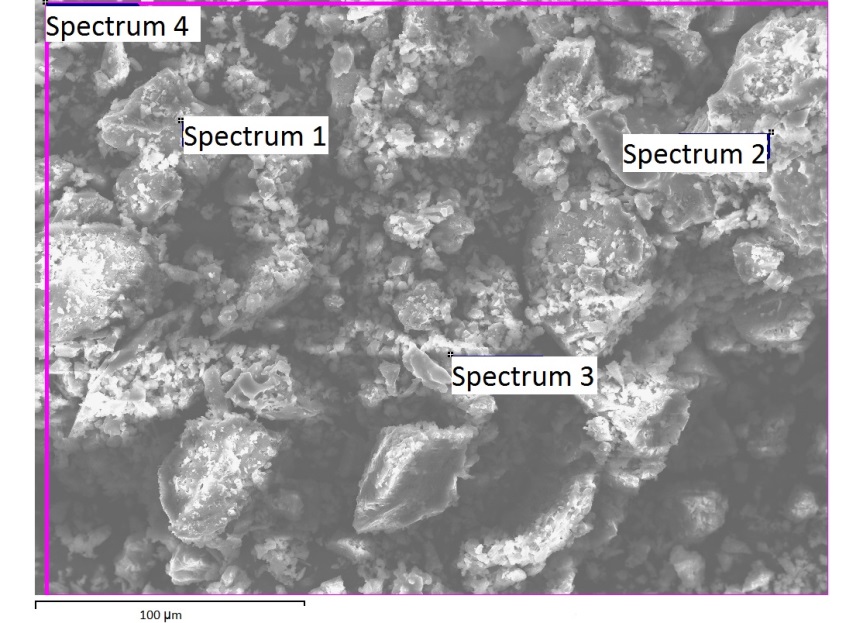


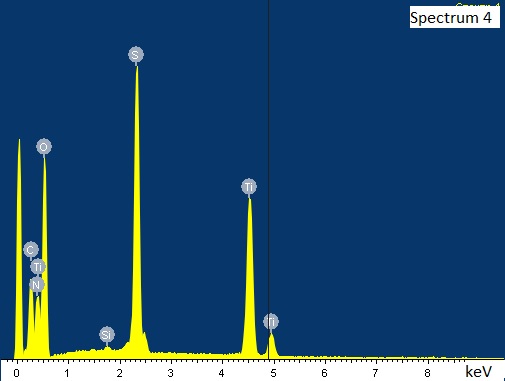


Atomic %

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
| Spectrum | C | O | S | Ti |
| 1 | 8.78 | 74.71 | 9.34 | 7.17 |
| 2 | 7.61 | 72.43 | 10.93 | 9.03 |
| 3 | 7.61 | 45.45 | 22.25 | 24.69 |
| 4 | 27.74 | 60.14 | 6.51 | 5.61 |
| Average | 12.93 | 63.18 | 12.26 | 11.62 |
| Standard deviation | 9.89 | 13.44 | 6.91 | 8.82 |
| Max | 27.74 | 74.71 | 22.25 | 24.69 |
| Min | 7.61 | 45.45 | 6.51 | 5.61 |

Sample TSUP185

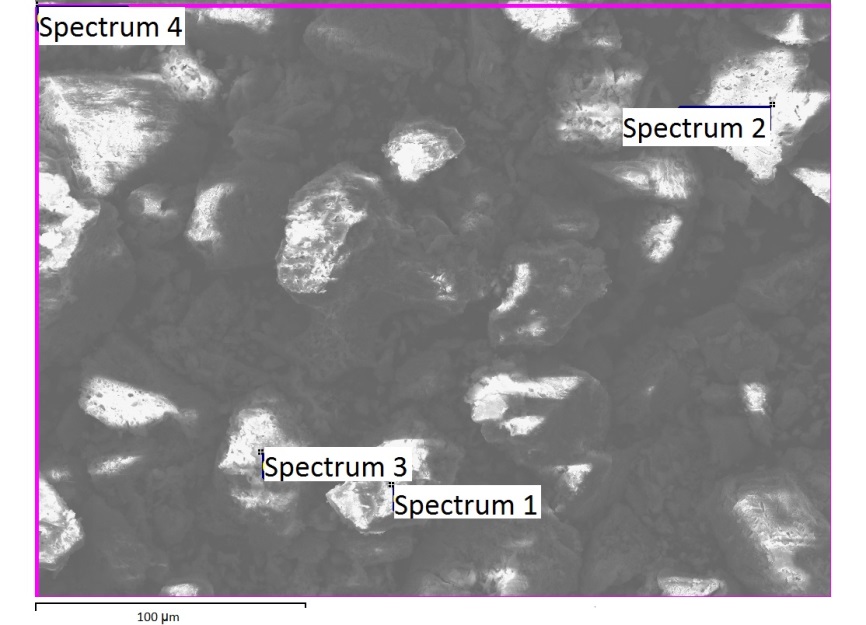


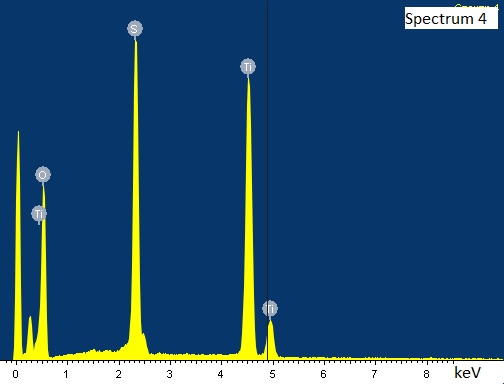


Atomic %

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| Spectrum | C | N | O | Si | S | Ti |
| 1 | 7.54 | 12.26 | 62.26 |  | 9.96 | 7.98 |
| 2 | 9.17 | 15.40 | 59.19 |  | 9.12 | 7.12 |
| 3 | 9.64 | 13.25 | 47.27 | 0.16 | 15.00 | 14.68 |
| 4 | 25.62 | 15.23 | 49.00 | 0.07 | 5.48 | 4.60 |
| Max | 25.62 | 15.40 | 62.26 | 0.16 | 15.00 | 14.68 |
| Min | 7.54 | 12.26 | 47.27 | 0.07 | 5.48 | 4.60 |

Sample TSUP515





Atomic %

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
| Spectrum | O | Al | S | Ti |
| 1 | 76.39 | 0.20 | 11.60 | 11.81 |
| 2 | 29.03 |  | 29.29 | 41.68 |
| 3 | 79.94 |  | 9.20 | 10.86 |
| 4 | 74.89 |  | 10.90 | 14.21 |
| Max | 79.94 | 0.20 | 29.29 | 41.68 |
| Min | 29.03 | 0.20 | 9.20 | 10.86 |

Details of preparing samples for elemental analysis and X-ray microanalysis

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
| Sample | 1st reagent | 2nd reagent | Annealing temperature, °C | Annealing time, min |
| TSUP100 | TiOSO4∙2H2O  (2.55 g, 13.5 mmol) | (NH2)2CO∙H2O2  (1.5 g, 16.5 mmol) | 100 | 60 |
| TSUP160 | TiOSO4∙2H2O  (2.55 g, 13.5 mmol) | (NH2)2CO∙H2O2  (1.5 g, 16.5 mmol) | 160 | 90 |
| TSUP185 | TiOSO4∙2H2O  (1.7 g, 9 mmol) | (NH2)2CO∙H2O2  (1 g, 11 mmol) | 185 | 120 |
| TSUP515 | TiOSO4∙2H2O  (1.7 g, 9 mmol) | (NH2)2CO∙H2O2  (1 g, 11 mmol) | 515 | 330 |

## X-ray photoelectron spectroscopy

Oxygen peaks

|  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- |
| Sample | Peak (O2) area | Peak (O) area | Total area | Peak (O2) fraction | Peak (O) fraction |
| TSP700 | 27393,3 | 55490,4 | 82883,7 | 0,3305 | 0,6695 |
| TSPSA700 | 18364,8 | 48837,4 | 67202,2 | 0,27328 | 0,72672 |
| TSUP700 | 31685,4 | 58221,7 | 89907,1 | 0,35242 | 0,64758 |