

Supporting Information

- Figure 1. Powder X-ray diffraction patterns of the glycolate precursors.
- Figure 2. FTIR spectra of (a) Titanium glycolate, (b) Zirconium glycolate and (c) Vanadium glycolate
- Figure 3. Thermogravimetric data for all the three glycolate precursors
- Figure 4. XRD pattern of calcined NPs at 500 °C of TiO₂ synthesized using (a) Microemulsion route containing OBG and (b) Microemulsion without OBG
- Figure 5. TEM image of calcined TiO₂ NP at 500 °C synthesized using Microemulsion without containing OBG

Figure 1

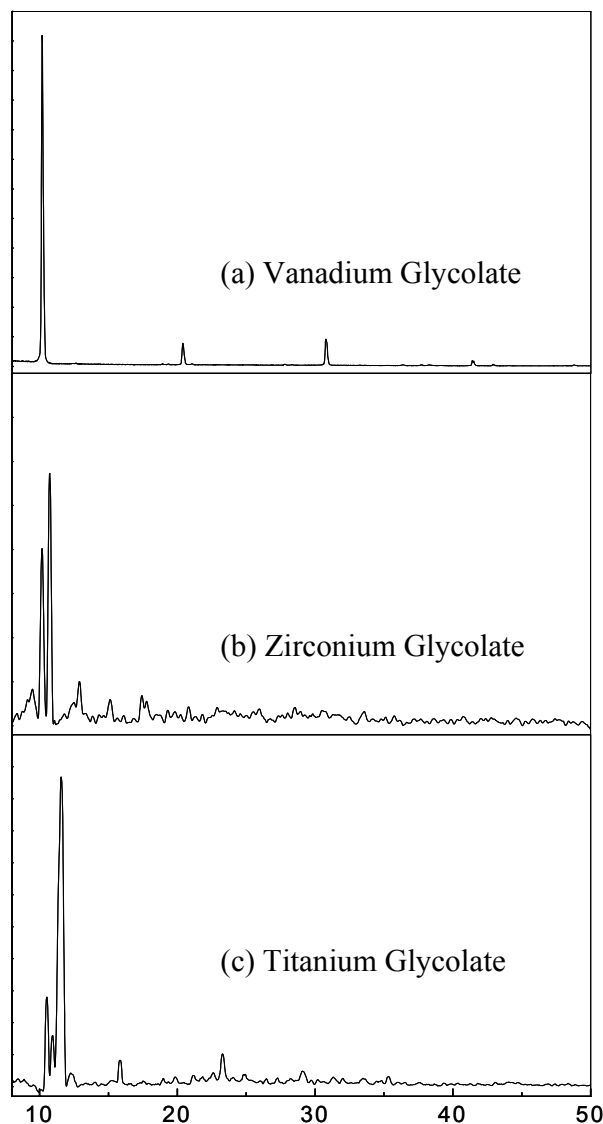


Figure 1 shows the representative XRD patterns of the synthesized glycolate precursors. The presence of relatively intense peaks suggest an ordered structure, however, so far there is no JCPDS data available for these glycolates. Only Rietveld analysis data for vanadium glycolate synthesized by a different approach has been reported (Weeks *et. al. J. Mater. Chem.*, 2003, **13**, 1420). The peak position observed for vanadium glycolate (d spacing: 4.36, 3.20, 3.15, 2.38, 2.34, 2.17 and 2.10 °Å) have been found to match with the reported data. Currently, the exact structure determination for these glycolate precursors is underway. It should also be noted that there is an absence of the respective oxide phases in the as-synthesized glycolate precursors.

Figure 2

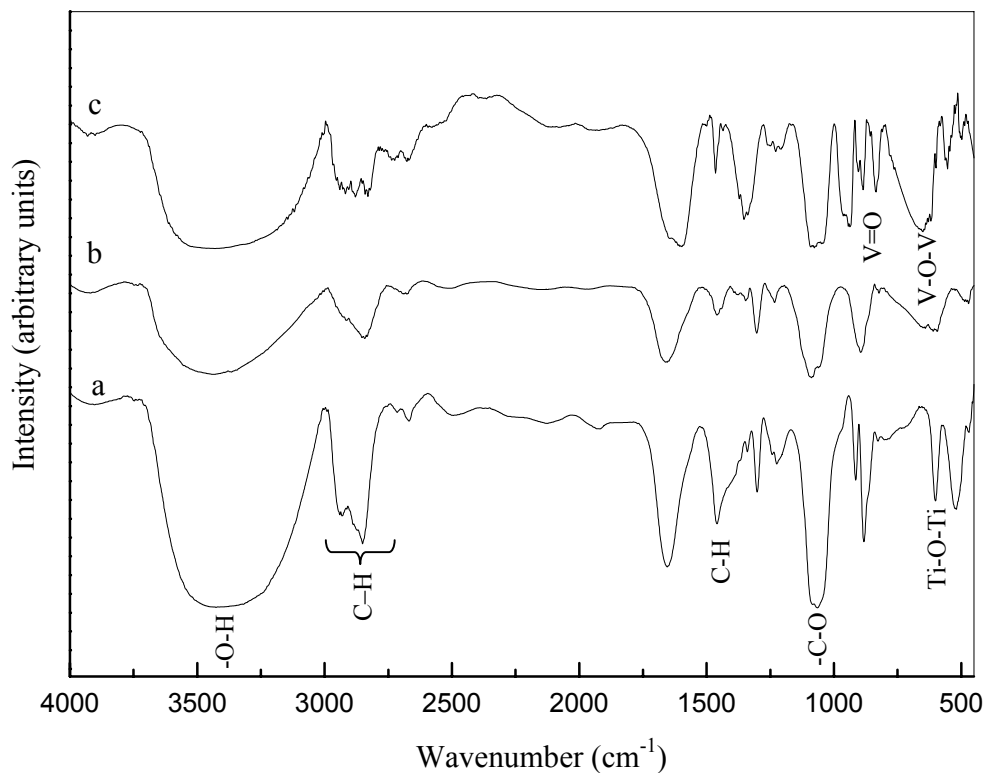
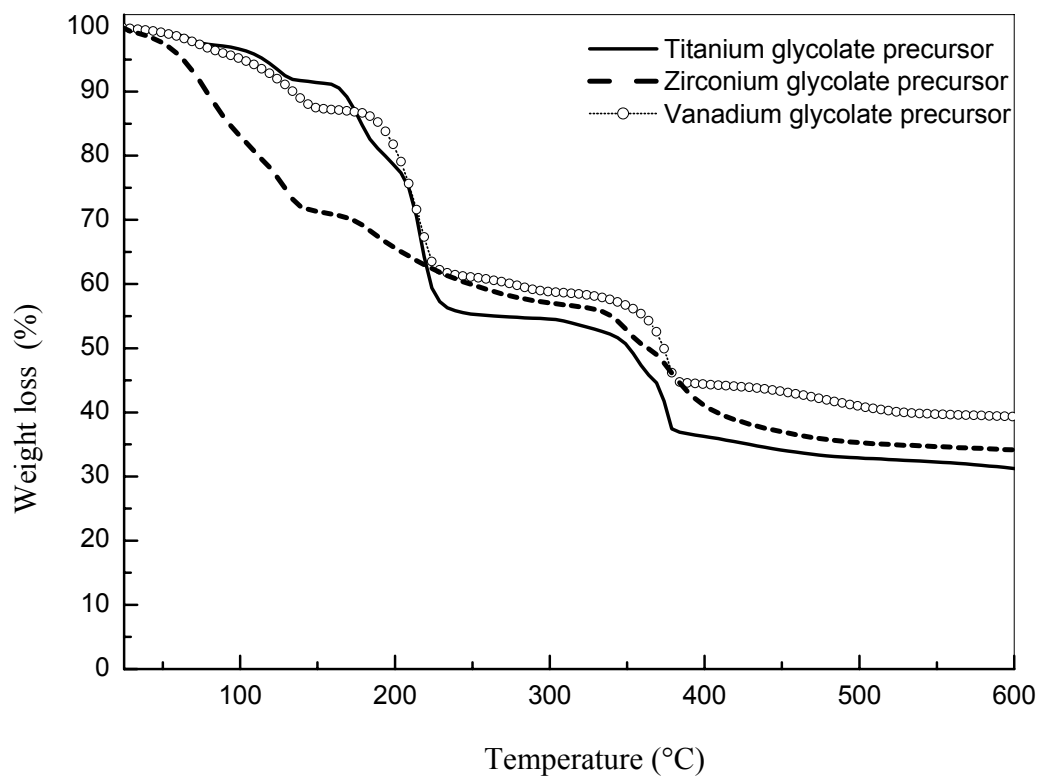


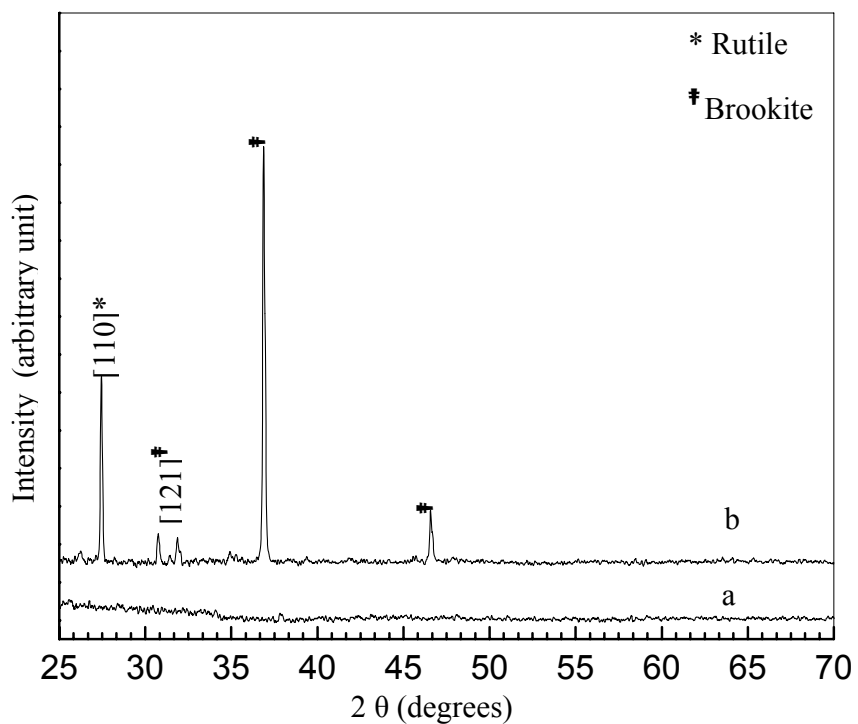
Figure 2 shows the FTIR spectrum with an intense broad absorption band at about 3400 cm^{-1} due to the presence of O–H stretching vibrations of metal glycolate moiety. The appearance of other intense band at *ca.* 1080 cm^{-1} suggests the presence of C–O stretching. While the peaks observed at 1340 and 2800 cm^{-1} indicate the presence of metal coordinated ethylene glycol ligand and C–H stretching bands, respectively. These peaks are also present in zirconium/vanadium glycolate precursor. In case of titanium glycolate precursor, the Ti–O–Ti network can be seen by the presence of the peak at 650 cm^{-1} . While the peaks observed in Fig. 2c at 651 and 880 cm^{-1} correspond to V–O–V and V=O vibration bands, respectively (Chen *et. al.*, *J. Mater. Sci.*, 2004, **39**, 2625).

Figure 3



In figure 3, predominantly two regions of weight loss are observed. The loss in the temperature range of 25 °C to 210 °C is mainly attributed to physisorbed water and the decomposition of physisorbed ethylene glycol. This was confirmed by recording the TGA curve for pure ethylene glycol, the weight loss for which occurs just below 200 °C. The second weight loss takes place at *ca.* 350 °C and this has been ascribed to the decomposition of ethylene glycol moiety coordinated to the metal atoms (Ti/Zr/V).

Figure 4



In Figure 4, the XRD pattern exhibit the appearance of rutile (JCPDS Card 00-034-0180) and brookite (JCPDS Card 00-029-1360) phase on heat treatment at 500 °C, while the control sample (synthesized using microemulsion mixture without containing OBG) showed almost amorphous nature.

Figure 5

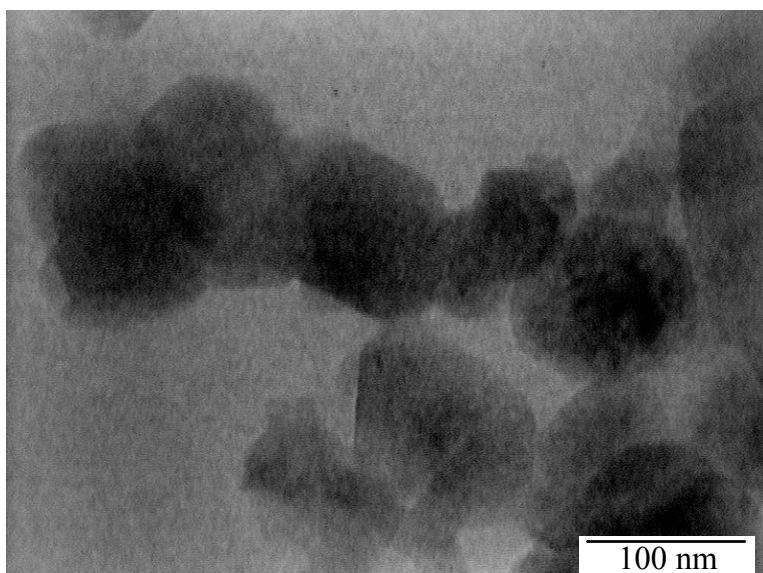


Figure 5 shows the TEM image in which the expected increase in the crystal size (on the order of *ca.* 100 nm) due to the coalescence of small NPs can be observed.