High-temperature stabilized anatase TiO₂ from an aluminum-doped TiCl₃ precursor

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† Electronic Supplementary Information (ESI) available: [detailed experimental procedures, ICP and weight change data, and SEM images for calcined TiO₂ from TiCl₃(com-25%Al) and TiCl₃(com-25%Al,sub)]. See http://dx.doi.org/10.1039/b000000x/

Summary

The solid-state hydrolysis and air calcination of aluminum-doped TiCl₃ leads to crystalline anatase TiO₂ that is stable on heating to 1000 °C, in contrast to control studies with related AlCl₃ and TiCl₃ physical mixtures that produce rutile TiO₂ under the same conditions. Particle and crystallite sizes are at or above 100 nm, which is much larger than sizes observed for unstabilized anatase TiO₂ systems.

\[
\text{TiCl}_4 + \text{Me}_3\text{Al} \rightarrow \text{Ti}(3 \text{ mol}\% \text{ Al})\text{Cl}_3(s) + \text{Me}_2\text{AlCl} + 1/2 \text{C}_2\text{H}_6
\]

\[
\text{Ti(Al)Cl}_3 + \text{H}_2\text{O/O}_2 \rightarrow \text{Ti(Al)O}_3(\text{OH})_2\text{Cl}_2 \rightarrow \text{Ti}(1.5 \text{ mol}\% \text{ Al})\text{O}_2
\]

[retains > 80 % anatase at 1000 °C]

Images of hydrolyzed then calcined aluminum doped TiCl₃