PROCESS FOR SYNTHESIS OF WELL-ORDERED MESOPOROUS TITANIA HAVING MONOCLINIC AND ANATASE PHASES

IITM Technology Available for Licensing

Problem Statement

- In the present era, semiconductor-based photo-catalysts are being explored worldwide for many purposes and the photocatalytic activity of such materials depends on many properties, viz., surface area, recombination rate of excitons, crystal phase, crystallite size, morphology, etc.
- One of the major challenges lies in the synthesis of high-quality mesoporous materials like titania precursor during hydrolysis leading to **uncontrolled phase separation**, which normally **results in disordered phases**.
- Hence, there is a need to mitigate these issues.

Technology Category/ Market

Technology: Synthesis Of Well-ordered Mesoporous Titania; **Industry:** Semiconductor, Catalyst/Photocatalyst**,** Chemical Engineering; **Applications:** Catalyst, Porous Materials, etc. **Market:** The Global Titanium Market is projected to **$33.5B** by **2026**, at a **CAGR** of **6.3%** during 2022-**2026**.

Technology

- The Present Patent discloses **a process for the synthesis of well-ordered mesoporous titania having monoclinic and anatase phases under optimal conditions.**

Firstly, Pluronic F127 triblock copolymer (2 g) is dissolved in (20mL) ethanol by stirring the solution for 2 h to obtain a homogenous solution.

Further, titanium tetrachloride (7mL) $TiCl_4$ (1M in methylene chloride) added dropwise to the homogenous solution under uniform stirring for 30 min till a clear solution was obtained.

The resultant solution with molar ratios TiCl4/F127/Ethanol = 1: 0.003 : 6.84, was poured onto a petri dish with uniform thickness.

- The claimed **process** further explains that the **solvent** is allowed to **evaporate in the oven** at **40°C for at least 7 days** during which inorganic precursors get hydrolyzed & and polymerized into a **metal oxide framework.**
- The as-synthesized sample obtained can be finally calcined at 350°C for 4h in the air at 0.5°C/min to remove the surfactant and obtain well-ordered highly ordered 2D hexagonal mesoporous titania. **i.e., mesoporous TiO2 (TMF-127).**
- The Rietveld refined XRD pattern clearly depicts the phase composition ratio in which **monoclinic (59 wt.%)** and **anatase (41 wt.%) phases**. (Refer Fig.1)

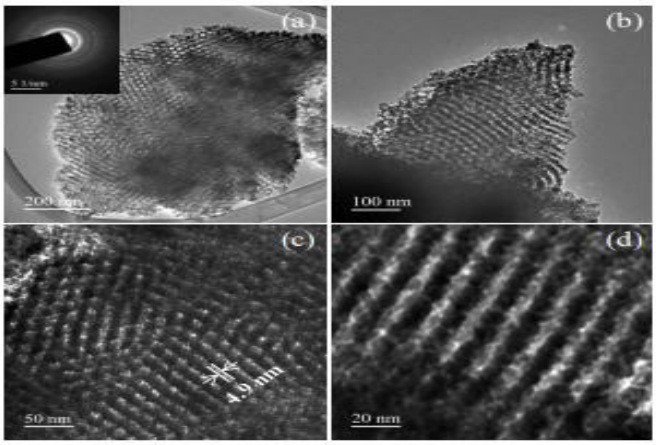


Fig. 1: Depicts TEM images of mesoporous titanium dioxide, TiO_2 (TMF-127)

Intellectual Property

IITM IDF Ref.:1463; IN Patent No.386294

TRL (Technology Readiness Level)

TRL- 3/4: Technology validated in Lab

Research Lab

Prof. P. Selvam, NCCR & Dept. of Chemistry

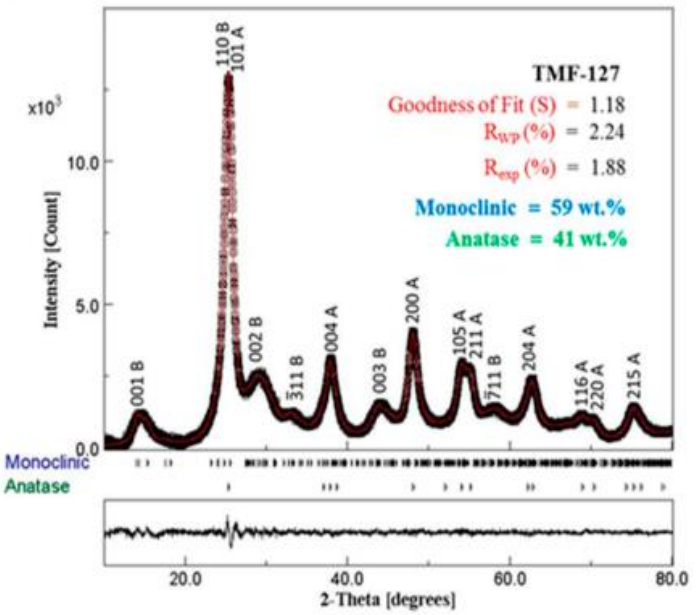
CONTACT US
Dr. Dara Ajay, Head
Technology Transfer Office,
IPM Cell- IC&SR, IIT Madras

IITM TTO Website:
https://ipm.icsr.in/ipm/

Email: smipm-icsr@icsrpis.iitm.ac.in
sm-marketing@imail.iitm.ac.in
Phone: +91-44-2257-9756 / 9719

Image

FIG. 2: Illustrates of the Rietveld refined XRD pattern of mesoporous titania, TMF-127.



Experimental Results

Table 1: Shown the corresponding structural parameters of mesoporous titania, TMF-127.

Table 1. Structural parameter of TMF-127

Parameters	Monoclinic (Bronze)	Tetragonal (Anatase)
Crystallite size (nm)[a]	5	8
Composition (wt.%)[b]	59	41
Lattice parameters (Å)	a = 12.105(3) b = 3.7140(6) c = 6.4938(18) β(deg.) = 107.65(3)	a = 3.7832(2) c = 9.4924(4)
Unit cell volume (Å)³	V = 292.00(14)	V = 135.862(10)

[a]Scherrer crystallite size. [b]Data from Rietveld refinement.

Key Features / Value Proposition

❖ *Technical Perspective:*

- The obtained mesoporous titania (**TMF-127**) confirmed the **long-range ordered channels** as well as the **hexagonal pore system,** and the inset selected area electron diffraction (SAED) confirms structure is **well crystallized**.
- The **use of a non-aqueous medium (ethanol) and block copolymer surfactant F127**, the rate of hydrolysis, condensation, phase transformation, and phase separation between surfactant and titania precursor is **minimized**.
- **A controlled aging process** leads to the form of a **well-ordered mesoporous matrix** with **monoclinic, TiO_2(B), and anatase, TiO_2 phases**.

❖ *Industrial Perspective:*

- Provides a **cost-effective improved process** for the **synthesis** of **well-ordered mesoporous titania** having monoclinic (bronze) and tetragonal (anatase) phases.

CONTACT US
Dr. Dara Ajay, Head
Technology Transfer Office,
IPM Cell- IC&SR, IIT Madras

IITM TTO Website:
https://ipm.icsr.in/ipm/

Email: smipm-icsr@icsrpis.iitm.ac.in
sm-marketing@imail.iitm.ac.in
Phone: +91-44-2257-9756 / 9719