CTAB-Templated Synthesis of Nanocrystalline Ordered Mesoporous Titania (TMC-016)

IITM Technology Available for Licensing

Problem Statement

- **Titania (TiO2)** has **superior physic-chemical properties**, making it **structural stabile, non-toxic, highly photocatalytic active and of low cost.**
- To Improve catalytic properties of TiO2 **different morphologies, generation of defects** with various synthetic strategies.
- Therefore it is important to study **ordered mesoporous titania (OMT) materials** which is difficult to synthesize due to the **high electrophilicity of Ti4+ ion** making **uncontrolled hydrolysis-condensation and phase separation** between surfactant template and precursor, resulting in rapid dense phase disordered materials formation.
- Based on the foregoing a need therefore exists for an improved **process to synthesize an advanced material** that addresses above mentioned issues.

Technology Category/ Market

Category: Chemistry & Chemical Analysis, Advanced Material & Manufacturing

Application: purification of air and water, H2O splitting, CO2 reduction, photocatalytic

Industry: Advanced Material, Catalyst, Photocatalyst, Chemical Manufacturing

Market: The global market size of titanium in **2022** amounted to **$ 28.59 B**. As of June 2023, the market value of titanium was projected to grow to nearly **$ 31 B**. The titanium market size is forecast to grow to nearly **$ 52 B** in **yrs 2023-2030.**

Technology

The present patent proposes an improved process for **synthesis of ordered 2D-hexagonal mesoporous titania** using cationic surfactant cetyltrimethylammonium bromide **(CTAB), structure directing template Titania (TMC-016).**
Refer FIG 1 and FIG. 2 with respect to Table 1

Key Features / Value Proposition

➢ ***User Prospective:***

- **High Surface Area**
- **High Thermal Stability**
- **Tenable/Narrow Pore Diameter**
- **Active Phase Composition**
- **Thick Pore Walls**
- **Prevents mesostructure collapsing on surfactant removal.**
- **Cost efficient & Non-toxic**

➢ ***Industrial Prospective:***

- **TMC-016** is systematically characterized to study the **photocatalytic activity** of material to degrade **4-chlorophenol.**
- The **molar ratio** in resultant solution **Ti(OiPr)4/HCI/CTAB/n-butanol** was **1:2:204:0.0609:11.020.**
- The preparation of **TMC-016** takes place via **evaporation induced self-assembly (EISA) method.**
- The ordered structure has **higher hydrophobicity than ethanol**.
- It effectively prevents **nucleophile attack (H2O) on Ti4+.**
- It **controls phase separation** between surfactant and titania precursor.

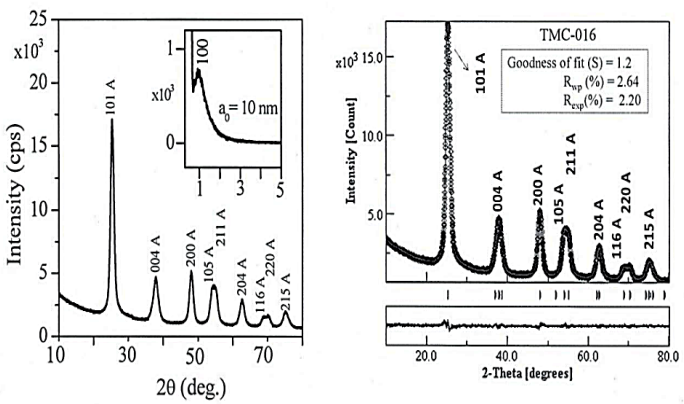

Figure 1: (a) XRD patterns of TMC-016. (b) Rietveld refined XRD pattern of TMC-016.

FIG. 1 illustrates a graph representation of the **XRD patterns** of TMC-016 (Left) & **Rietveld refined XRD patterns 2** of TMC-016 (Right)

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Process

The method comprises of the following steps:

- **Preparing a first solution (Solution A) was prepared by dissolving 0.896 mmol (326 mg) CTAB in 0.162 mo/ (12 g) n-butanol to obtain homogenous solution;**
- **Preparing a second solution (Solution-B) by dropwise addition of 0.0324 mol (3.2 g) conc. HCl into 0.0147 mol (4.2 g) titanium (IV) tetra-isopropoxide under 30 min vigorous stirring**
- **Adding Solution-A dropwise into Solution-B under uniform stirring at room temperature and further stirred for 6 h to get a clear, homogenous solution;**
- **Pouring resulting clear solution into Petri-dishes with uniform thickness and evaporating it in hot oven at 45°C for 7 days to trigger self-assembly;**
- **Forming organic-inorganic mesostructure with hydrolysis and condensation of the inorganic precursor into a periodic mesoporous network;**
- **Calcinating the synthesized resultant sample at 350°C for 4 h in air at heating rate of 0.5°C/min to obtain 2D-hexagonal ordered mesoporous TiO2 (TMC-016).**

Intellectual Property

IITM IDF Number: 1805
IP Patent Number: 369497 (Granted)

TRL (Technology Readiness Level)

TRL – 4; Technology validation in Lab

Research Lab

Prof. Selvam P
NCCR & Department of Chemistry

Images

FIG. 2 illustrates a graphical representation of **500 TEM images of TMC-016 with a confirm long range ordered channels and selected area electron diffraction (SAED) confirms crystalline framework.**

a. **200 nm; 100 nm;**
b. **10 nm- Lattice fringes with d-spacing 3.6 Å correspond to (101) plane of anatase phase;**
c. **51 nm**

The corresponding structural, textural properties are given in **Table 1**

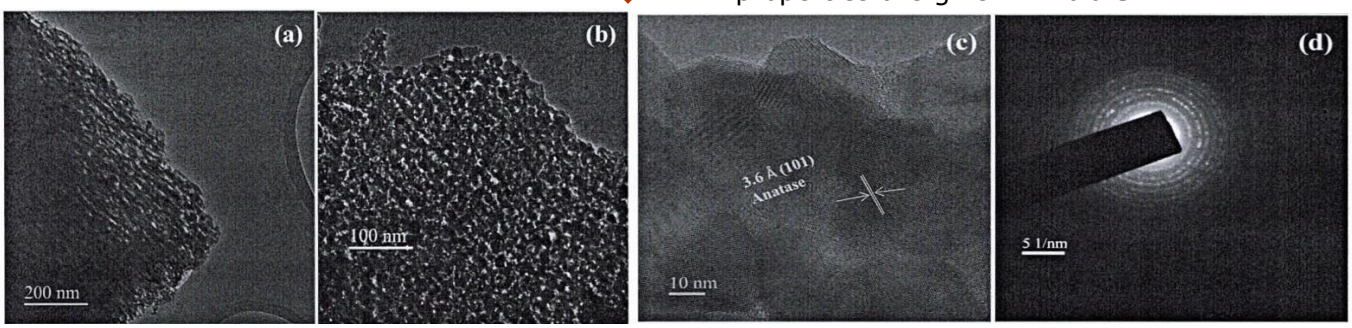

Table 1

Catalyst	[a]E_g (eV)	[b]S_{BET} ($m^2 g^{-1}$)	[c]V_P (cm^3/g)	[d]D (nm)		[e]h_w (nm)	4-CP degradation	
				BJH	TEM		[f]k (x 10^{-3} min^{-1})	[g]DE (%)
TMC-016	2.9	125	0.13	3.5	5.0	6.5	11	81
P25	3.3	53	-	-	-	-	6	53

[a]Band gap; [b]Surface area; [c]Pore volume, [d]Pore diameter; [e]Wall thickness; ($h_w = a_0 - DBJH$);
[f]First order rate constant; [g]Degradation efficiency.

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