

# Synthesis and Characterization of Titanium Mesoporous Silicate

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In this project, surfactant hegzadesil three-methyl ammonium chloride was used as a structural informant or mould for synthesis of Mesoporous silicate molecular sieves. Many of the Mesoporous titanium silicates were synthesized by adding titanium compounds to the mixture, but considerable ratio of titanium centers placed in the frame are not accessible. This disadvantage was compensated by titanium butoxide as a titanous compound after being linked to the interior surfaces of host Mesoporous. The Mesoporous silicate was synthesized by,  $S^{+}I^{-}$  method, and then titanium was linked into the cavities. The structure of synthesized compound was characterized and studied by using XRD, XRF, TGA, SEM, IR techniques, and surface adsorption of nitrogen. The results showed the formation of meso structure with hexagonal cavities, high surface area, isomorphic cavities, spherical morphology, functional groups, and the amount of titanium input into the structure.

**Keywords** Mesoporous silicate, SEM, surfactant hegzadesil, techniques three-methyl ammonium chloride, TGA, XRD, XRF

## 1. INTRODUCTION

Today, much attention has been directed toward selective oxidation by solid catalyses.<sup>[1]</sup> Until the end of the 70th decade, ion exchanging was the main method for metals coordination to Microprose matrix, which creates big difficulty for metal separation in liquid phase oxidation.<sup>[2]</sup> A break in which titanium embedded into the silicate structure was obtained by exploring titanium silicate (TS-1).<sup>[3,4]</sup> Based on this information, many titanous silicate Microproses, such as TS-2,<sup>[5]</sup> Ti-ZSM-48,<sup>[6]</sup> Ti- $\beta$ ,<sup>[7]</sup> and titanium aluminum phosphate, TAPO-5,<sup>[8]</sup> TAPO-36<sup>[9]</sup> and TAPO-34,<sup>[10]</sup> were provided. The general problem of these materials is the small size of their cavities that limits accessibility of important and large substrates.

Exploring the M41S family<sup>[11]</sup> created a new path for preparing the Titanosilicates and provided the possibility for selective oxidation of larger substrates. Many of Mesoporous titanium

silicates, including Ti-MCM-41,<sup>[12,13]</sup> Ti-HMS,<sup>[13,14]</sup> Ti-MCM-48<sup>[15]</sup> and silanated Ti-MCM-41,<sup>[16]</sup> were prepared by adding titanous compounds to a synthesized mixture. But a considerable number of titanium centers placed in its frame are inaccessible. When interior surfaces of host Mesoporous are linked to synthesized titanium catalysis precursor, this weak point can be compensated. So, the applied method for synthesis of titanium Mesoporous silicate in this project is based on linkage after synthesis. It means that at first, Mesoporous material is synthesized and then titanous compounds are linked in its cavities.

In this article, the structure is characterized and studied after synthesis process of titanium Mesoporous silicate takes place. The presented methods included X-ray diffraction measurements at low angles, infra-red spectrometry, superficial absorption of Nitrogen for studying superficial area, cavity volume, distribution of cavity sizes on the basis of BET, BJH and SEM methods, elemental analysis, and XRF.

## 2. EXPERIMENTAL SECTION AND DATA ANALYSIS

### 2.1. Synthesis of Mesoporous Silicate

According to performed tests, we need a certain ratio of surfactant to silica to obtain considered structure. Mole ratio of surfactant to silica was 0.5 and surfactant quantity was 0.05 molar. To provide silicate solution, at first 40 gr distilled water was added in balloon placed on stirrer and then 20.5 gr sodium silicate added. Then 1.2gr sulfuric acids was added to this solution and stirred for 10 min. Surfactant solution was provided by combining 31.15gr surfactant with 40.5gr distilled water. After 10 minutes, surfactant solution was added to the silicate solution. The obtained gel was stirred for half an hour, then 20gr water was added to it and refluxed for 144 h in 100°C. After setting, it was chilled to ambient temperature, the sediment was filtered and washed with twice-distilled water and placed in ambient temperature to dry overnight. The obtained white solid material divided into two parts. In order to extract the compound from the mould, one part transferred into porcelain cup and placed into furnace. As observed in Figure 1, furnace heat was increased from ambient temperature to 550°C with 3°C/min temperature slant. Then it remained in this temperature for 7 hours. The temperature decreased to ambient temperature with former slant.

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