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TEMPERATURE-INDUCED STRUCTURAL TRANSFORMATIONS OF LAYERED TITANOSILICATE JDF-L1

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The efforts to produce new materials with industrially useful properties in the past years resulted in the synthesis of a number of microporous titanosilicates with potential applications in catalysis, ion exchange, separation processes. Some of these materials do not have mineral analogues and their comprehensive characterization may contribute significantly to understand their nature and properties as well as to optimize the preparation of titanosilicates having desired structural topology.

Layered titanosilicate JDF-L1 is synthesized in a Na_2O-TiO_2-SiO_2-H_2O system, applying a rapid procedure without using organic additives in the synthesis mixture. The thermal evolution of the JDF-L1 structure is studied by thermogravimetric-differential thermal analysis, powder X-ray diffraction and Raman spectroscopy. It is shown that on heating JDF-L1 undergoes a sequence of structural rearrangements causing formation of non-crystalline alkali titanate-titanosilicate phases, defect-rich microcrystalline silica and crystalline titanosilicate narsarsukite, the latter becoming the dominant phase at high temperatures. Upon thermal treatment JDF-L1 undergoes reconstructive phase transitions involving order-disorder-order processes and resulting in formation of narsarsukite as a final dominant phase. According to XRD and Raman spectroscopic data, the thermal evolution of the JDF-L1 structure consists of three main stages:

(i) below 550 °C - a gradual decrease in the interlayer spacing caused by H_2O removal, accompanied by topological changes in the Si-O entities and layer undulation;

(ii) near 580 °C - total collapse of the JDF-L1 structure, providing material for formation of X-ray amorphous Ti-rich phases, microcrystalline silica of opal CT-type, and nucleation of narsarsukite;

(iii) above 600 °C - atomic rearrangements in the matrix of non-crystalline alkali titanate-titanosilicate phases and opaline silica resulting in enhanced crystallization of narsarsukite.