Phase transformation in B*m* form of the stearic-acid crystal in low-temperature using combined DSC, Synchroton X-ray diffraction and polarized Raman spectroscopy

João Roberto S. Miranda, Francisco F. Sousa
Universidade Federal do Pará, Brazil

Fatty acids are organic compounds belonging to groups of carboxylic acids formed up of carbon atoms and the most common of them have chains with 10 to 22 carbons. They are the second energy source of the animal body and are also part of the chemical composition of several vegetable oils, likely those who are from the Amazon region. Among naturally occurring saturated fatty acids, the most commonly found in animal body are the palmitic and stearic acids, which are also identified in great quantity in Amazon vegetable oils, like in buriti oil and in cupuaçu butter. Stearic acid (SA) is a long chain saturated fatty acid with 18 carbon atoms. In the solid state the molecules are packed as dimers through O-H*...*O hydrogen bonds SA crystal can be obtained by low evaporation method from the organic solvent, through which obtain one a variety polymorphic forms. The diversity of possible crystalline forms depends on the chain length, parity, and number of unsaturated carbons in the alkyl chain. With regard to the carbon-chain parity, the forms A1, A2, A3, A*super*, C, B*o/m*, and E*o/m* were observed for even normal fatty acids and the forms A*'*, B'. C', C'' and D' were observed for odd ones. The B*m* and E*o* forms exhibit two polytypes, one being monoclinic (*P*21*/a*) and the other being orthorhombic (*Pbca*). In this work, the low-temperature phase transformation of SA crystal in B*m* form are reported. With aim of evaluating their thermodynamic stability, the follows diferential scanning calorimetry (DSC), *Synchroton* X-ray diffraction and polarized Raman spectroscopy techniques were used. Raman spectra measurements were carried out 20-3000 cm-1 range from the room temperature up to 10 K for two scattering geometries [Z(YY)Z and Z(XX)Z]. The behavior of Raman bands was investigated and some changes were observed in lower-frequency region, as well as in wavenumber values up to 1700 cm-*1*, principally in the regions corresponding to motions of the CH2 groups and C=O stretching vibration modes. Such changes were associated with a phase transition undergone by the crystal occurred at about 120-130 K that can be connected with conformational changes in the molecular structure. Diffraction results showed slight changes in the lattice parameters that corroborate the Raman data. Furthermore, an evidence of these phase transformation was provided by the DSC, which identified an enthalpic anomaly in same temperature range.