

Structural resolution of inorganic nanotubes with complex stoichiometry

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Determination of the atomic structure of inorganic single-walled nanotubes with complex stoichiometry remains elusive due to the too many atomic coordinates to be fitted with respect to X-ray diffractograms inherently exhibiting rather broad features. Here we introduce a new approach which enables resolution of their structure [1]. It is based, first, on the use of helical symmetries allowing one to consider the smallest unit cell and then on semi-empirical energy minimization leading to a reduction of the number of structural parameters to be fitted.

We applied this method to recently synthesized methylated alumino-silicate and alumino-germanate imogolite nanotubes of nominal composition $(\text{OH})_3\text{Al}_2\text{O}_3\text{Si}(\text{Ge})\text{CH}_3$ [2]. Thanks to their chemical versatility, imogolite nanotubes (INT) are promising candidates for applications in molecular storage, recognition and separation [3]. Fit of wide-angle X-ray scattering (WAXS) diagrams of methylated INTs enabled us to determine their atomic structure. Unlike their (N,0) zigzag hydroxylated analog, methylated INTs roll up into a (N,N) armchair structure (Figure).

The transferability of the approach opens up for improved understanding of structure-property relationships of inorganic nanotubes, to the benefit of fundamental and applicative research in these systems.

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[1] G. Monet et al., submitted (2018).

[2] I. Bottero et al., Synthesis and Characterization of Hybrid Organic/Inorganic Nanotubes of the Imogolite Type and Their Behaviour Towards Methane Adsorption, *Phys Chem Chem Phys* **13**, 744–750 (2011).

[3] D.-Y. Kang et al., Direct Synthesis of Single-Walled Aminoaluminosilicate Nanotubes with Enhanced Molecular Adsorption Selectivity, *Nat Commun* **5**, 3342 (2014).

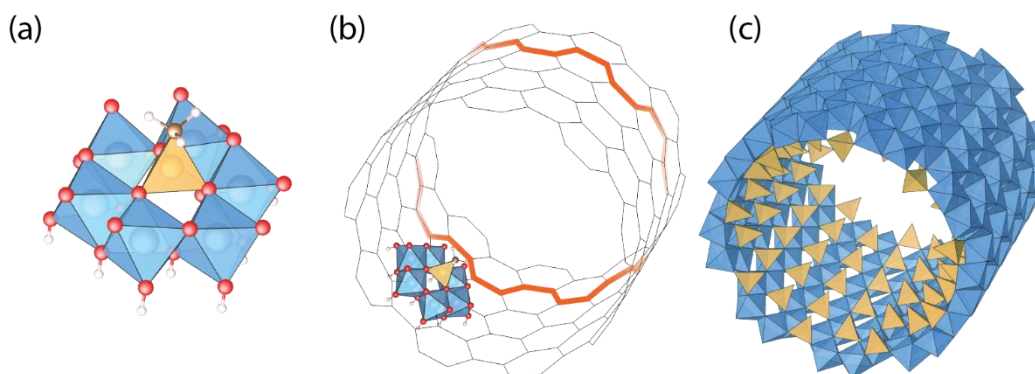


Figure: (a) An octahedral gibbsite-like layer (in blue) with isolated $(\text{Si}(\text{Ge})\text{O}_3)\text{CH}_3$ tetrahedron units (in yellow). (b) and (c) The methylated INT structure. Its armchair character is highlighted by the thick orange line in (b).