ROYAL SOCIETY OF CHEMISTRY
Nanoscale

Highly luminescent scintillating Tunable Microwave-Assisted Surface Functionalization of Biocompatible Magnesium Silicate Nanotubes for Advanced Nanomedicine Applications

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DOI: 10.1039/d5nr02385h
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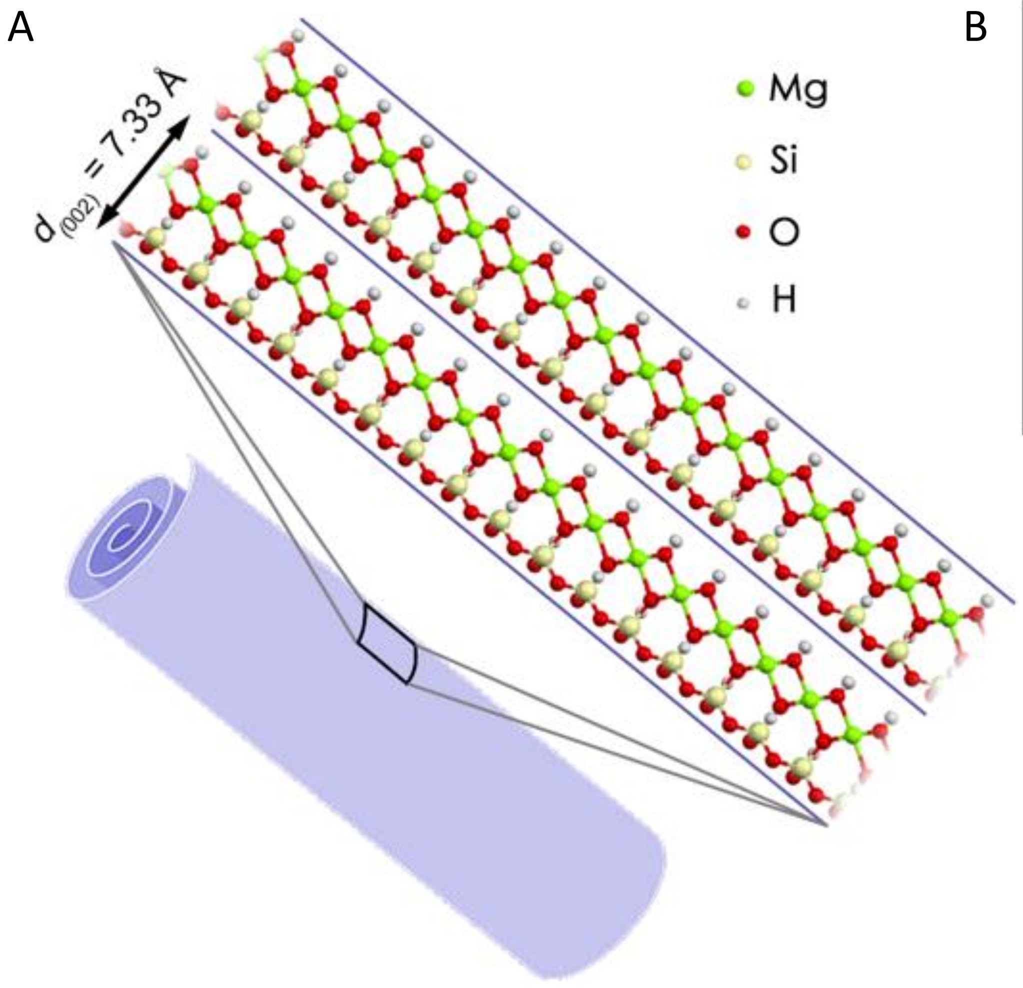
Magnesium silicate nanotubes (NTs) have recently emerged as a versatile class of biocompatible inorganic nanomaterials for biomedical applications, owing to their unique tubular morphology, high surface area, and ability to accommodate multiple functionalities. Developing reliable strategies for controlled surface modification is therefore essential to expand their potential as multifunctional platforms for diagnostics and therapy. Here, we present a rapid and tunable microwave-assisted silanization protocol for the covalent functionalization of synthetic NTs using 3-aminopropyltrimethoxysilane (3-APTMS). Microwave irradiation enables efficient functionalization under mild conditions (70 ° C, 1 h), significantly reducing reaction times compared with conventional approaches. By systematically varying the silane concentration and reaction time, surface coverage can be finely tuned, providing a versatile platform for the subsequent covalent attachment of functional molecules, including photosensitizers, targeting ligands, imaging probes, and therapeutic agents.
The structural integrity of the NTs after functionalization was confirmed by complementary characterization techniques, including TEM, powder XRD, Raman spectroscopy, XPS, solid-state NMR, elemental analysis, TGA, and ninhydrin assays. These analyses reveal that silanization proceeds through surface silanol groups, forming predominantly bi- and tri-dentate Si-O-Si linkages and achieving nearly complete surface coverage while preserving the NTs morphology and crystalline structure.
Overall, these results establish microwave-assisted silanization as a robust and reproducible strategy for engineering magnesium silicate NTs, paving the way for their integration into next-generation nanomedicine platforms.

A



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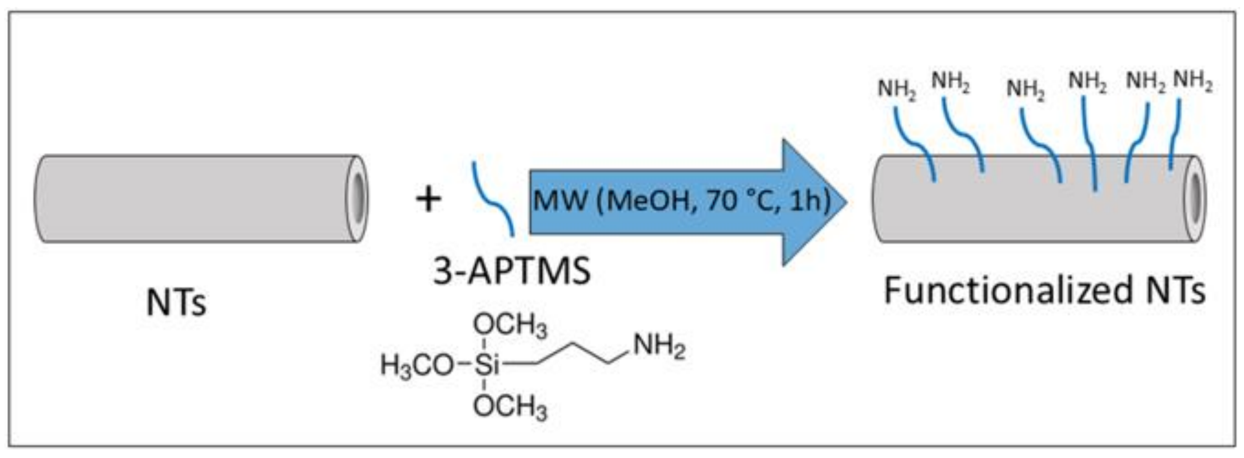
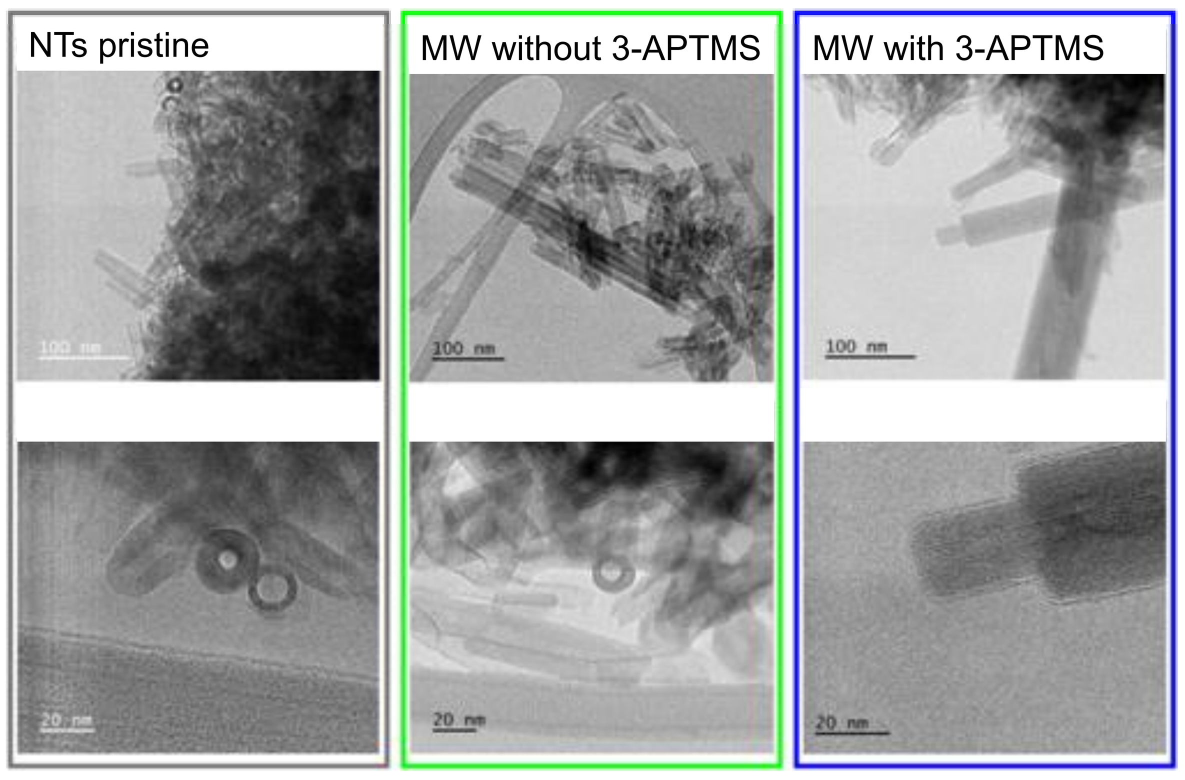


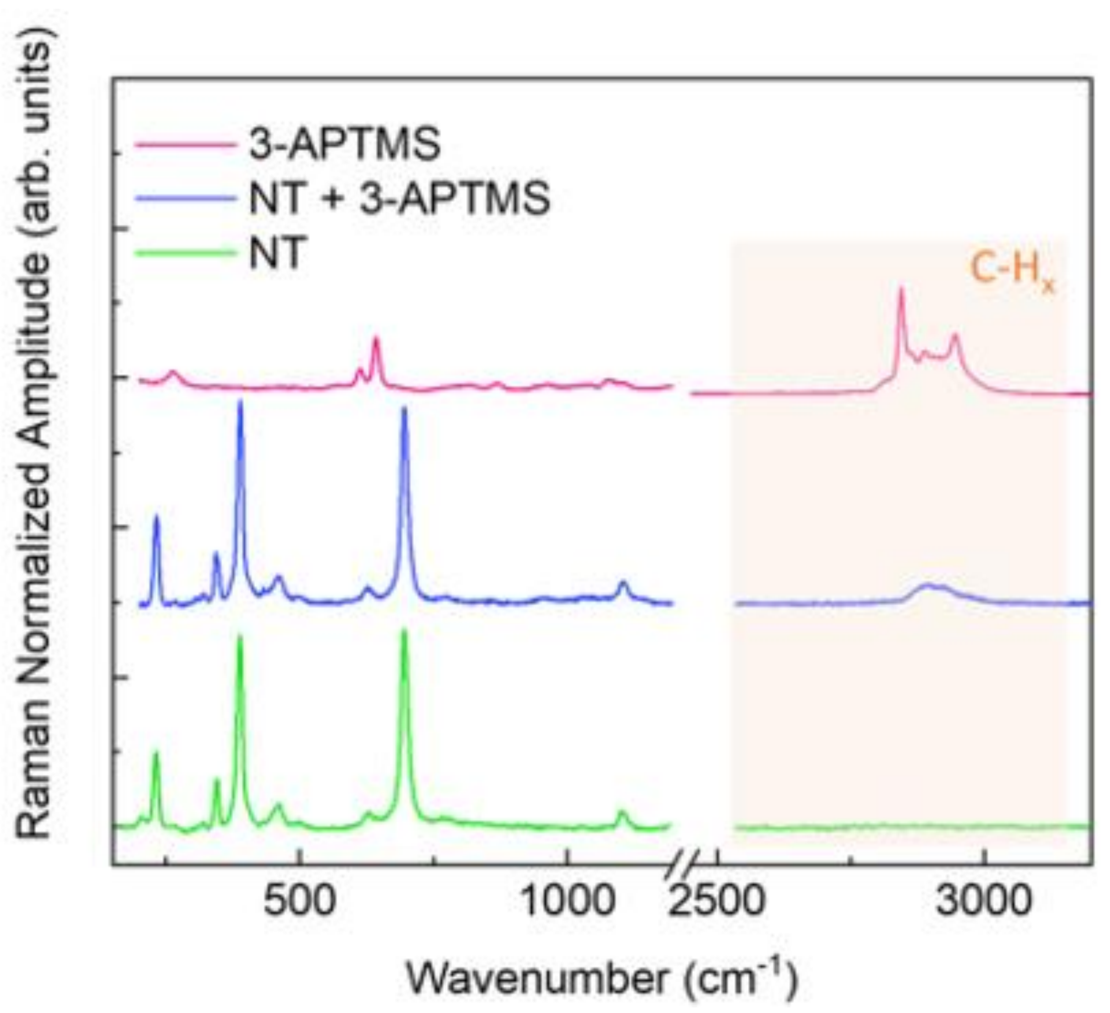
A. Chrysotile-like $Mg_3Si_2O_5(OH)_4$ nanotubes are hollow, biocompatible mineral nanostructures with a versatile outer surface. Hydrothermal synthesis: 250 °C for 16 h; washing and drying at 110 °C.

B. Surface silanization. NTs and 3-APTMS in MeOH: 0–1.0 wt%. MW heating to 70 °C followed by controlled dwelling time. Covalent grafting can improve colloidal stability and enable subsequent functionalization. 3-APTMS introduces surface $-NH_2$ groups for modular chemistry.

C

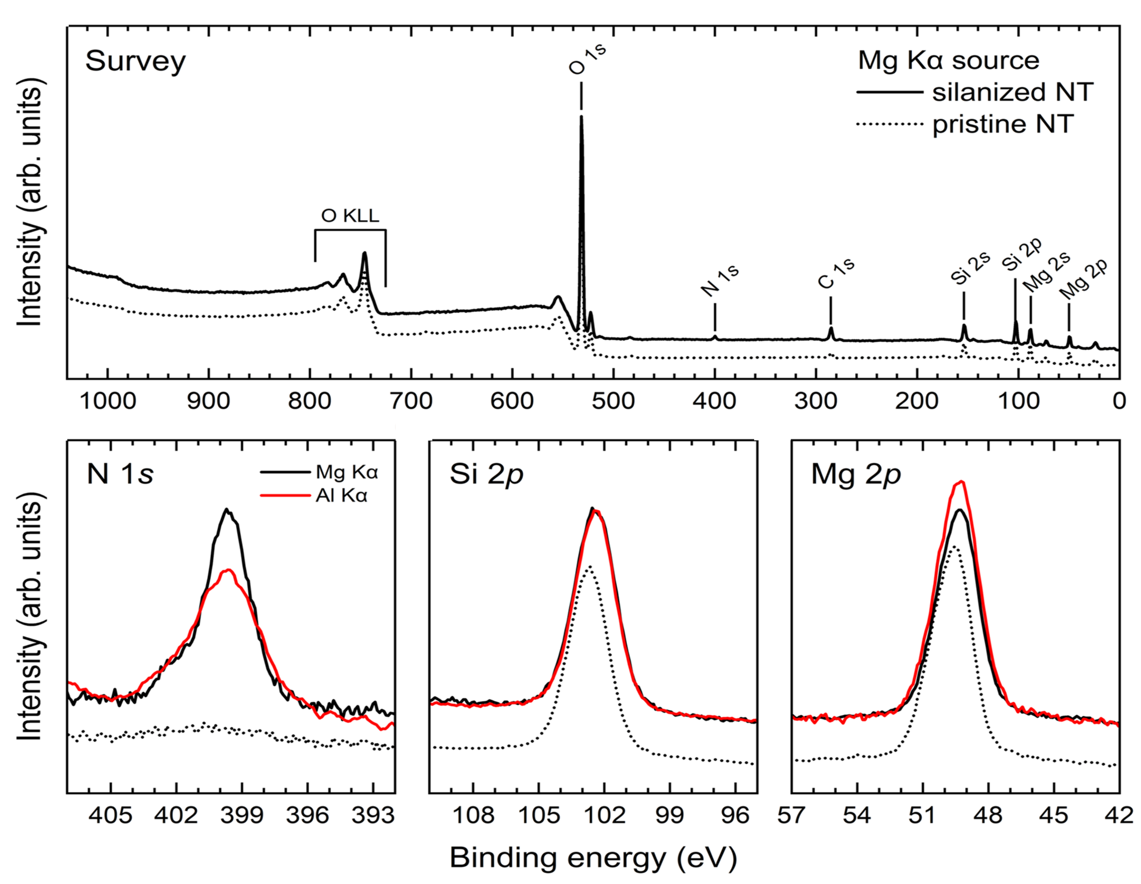


C. TEM images confirms tubular morphology before and after microwave treatment, without or with 3-APTMS. Size: 6–11 nm inner diameter, 15–25 nm outer diameter and 50–200 nm length.



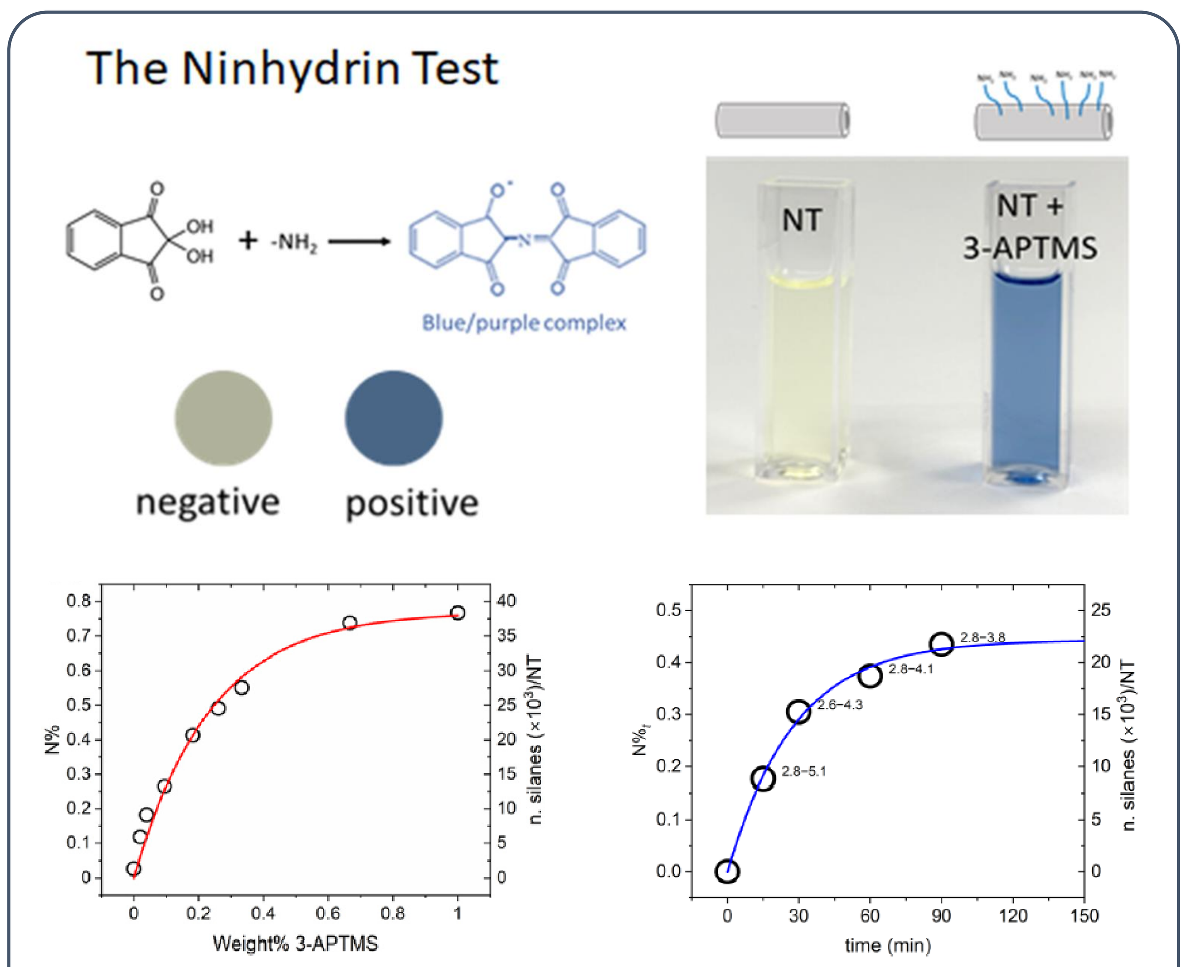
Raman spectra of NTs (green), silanized NTs (blue) and 3-APTMS molecule as reference (pink).

- Raman bands characteristic of NTs are retained after MeOH treatment and silanization.
- New C–H stretching features at 2800–3000 cm^{-1} evidence the organic silane.



Photoelectron spectroscopy results from pristine NTs (dotted line) and silane-treated NTs continuos line).

XPS detects nitrogen from 3-APTMS. The excitation depth comparison indicates that the silane is concentrated at the outermost nanotube surface.



The ninhydrin assay: a blue/violet color indicates the presence of NH_2 groups, N% in NTs and estimation of the number of silane molecules per NT **as** a function of the **silane concentration (**adopted in MW treatments at 70 °C for 1 h) or the **dwelling time** (under MW irradiation at 70 °C with silane concentration of 0.33%).

TAKE-HOME MESSAGE: A rapid, mild and quantitative route to functionalize biocompatible magnesium silicate nanotubes with 3-APTMS.

- **Coverage tunable with ~5–10% precision**
- **Near-saturation coverage under 70 °C / ~1 h conditions**
- **Up to 2.2 silane molecules nm^{-2} after 1 h**
- **Preserved chrysotile-like structure and tubular morphology**
- **Platform for future multifunctional nanomedicine**

Acknowledgements

A.E. thanks grant no. H45F2100343000. V.S. acknowledge the contributions from the University of Milano-Bicocca Starting Grant 2024-ATESP-0005. V.S. and A.M. acknowledge contribution from project no. PNC0000003. M.C. acknowledges contributions from The University of Milano – Bicocca and Cariplo Foundation through grants no. 2024-ATEQC-0017 and 2020-0977.