Report on the outcomes of a Short-Term Scientific Mission[1]

Action number: CA21126

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Details of the STSM

Title: **Interfacial Structure and Spectroscopic Signatures of Graphene Oxide-Containing Hybrid Nanostructures: Laboratory Models for Carbonaceous Nano-Environments**

Description of the work carried out during the STSM

The STSM was carried out at the University of Calabria within the framework of COST Action **CA21126 - NanoSpace**. The mission aimed to investigate graphene oxide-containing hybrid nanostructures as laboratory model systems for carbonaceous nano-environments by establishing correlations between their structural organization, interfacial interactions, and spectroscopic response. Two model systems were investigated: Fe_3O_4/chitosan, used as the reference material, and Fe_3O_4/chitosan/graphene oxide (GO), representing the carbon-containing hybrid nanostructure.

Prior to the STSM, both nanosystems were synthesized at the home institution using a co-precipitation method followed by chitosan coating and graphene oxide incorporation. During the mobility period, a comprehensive physicochemical characterization was performed using complementary analytical techniques available at the host institution.

Transmission Electron Microscopy (TEM) was employed to investigate the morphology and spatial organization of the hybrid nanostructures. The Fe_3O_4/chitosan reference system exhibited compact agglomerates resulting from magnetic dipole–dipole interactions between iron oxide nanoparticles. In contrast, incorporation of graphene oxide induced a pronounced reorganization of the hybrid architecture, where Fe_3O_4 nanoparticles became preferentially distributed over the graphene oxide sheets, leading to improved particle dispersion and the formation of extended carbon-rich interfacial domains (Figure 1).

[1] This report is submitted by the grantee to the Action MC for approval and for claiming payment of the awarded grant. The Grant Awarding Coordinator coordinates the evaluation of this report on behalf of the Action MC and instructs the Grant Holder Manager to verify the grant documents compliance and proceed with the payment.

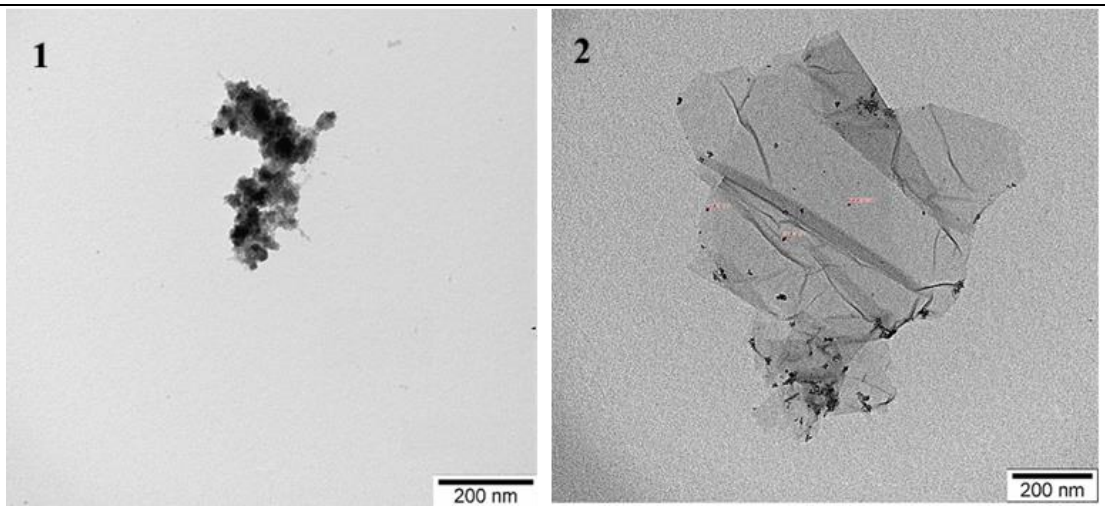


Figure 1. Morphological organization of the investigated hybrid nanostructures

(1) TEM image of the Fe_3O_4/chitosan reference system showing compact magnetic nanoparticle agglomerates formed predominantly through magnetic dipole-dipole interactions.
(2) TEM image of the Fe_3O_4/chitosan/graphene oxide hybrid system showing Fe_3O_4 nanoparticles preferentially immobilized on graphene oxide nanosheets, resulting in improved particle dispersion and formation of extended carbon-rich interfacial domains.

Fourier Transform Infrared (FTIR) spectroscopy was used to identify the functional groups participating in the hybrid interface. The spectra confirmed the presence of characteristic Fe-O vibrations (~524-612 cm^{-1}), C-O/C-O-C stretching bands (1069-1169 cm^{-1}), C-H deformation bands (1373-1453 cm^{-1}), N-H/C=C related vibrations (~1620 cm^{-1}), aliphatic C-H stretching bands (2850-2922 cm^{-1}), and broad O-H/N-H stretching vibrations centered at approximately 3445 cm^{-1}. Comparison of the reference and GO-containing systems revealed spectral changes consistent with hydrogen bonding and other non-covalent interactions between graphene oxide and chitosan.

X-ray diffraction (XRD) analysis confirmed the preservation of the characteristic inverse spinel structure of magnetite after hybrid formation, while maintaining the principal diffraction reflections corresponding to the (220), (311), (400), (422), (511), and (440) crystallographic planes. UV-Visible spectroscopy revealed the characteristic electronic absorption behavior of graphene oxide-containing materials, including the $\pi \rightarrow \pi^*$ transition of aromatic sp^2 carbon domains and the $n \rightarrow \pi^*$ transition associated with oxygen-containing functional groups, confirming successful hybrid formation. Thermogravimetric analysis (TGA/DTG) further demonstrated the thermal behavior of the hybrid systems, allowing evaluation of the contribution of chitosan and graphene oxide to the thermal stability of the composites.

The experimental work closely followed the original STSM work plan. Minor adjustments were limited to optimization of the analytical sequence and experimental scheduling according to instrument availability at the host institution. These modifications did not affect the scientific objectives or expected outcomes of the mission.

Description of the STSM main achievements and planned follow-up activities

The STSM successfully achieved its planned scientific objectives by generating a comprehensive experimental dataset describing the structural, morphological, spectroscopic, thermal, and optical properties of graphene oxide-containing hybrid nanostructures. The combined application of TEM, FTIR, XRD, UV-Visible spectroscopy, and TGA enabled a systematic investigation of the relationship between interfacial organization and the physicochemical behavior of carbon-containing hybrid systems.

TEM analysis demonstrated that graphene oxide actively reorganized the morphology of the hybrid nanostructure by serving as a two-dimensional interfacial scaffold for Fe_3O_4 nanoparticles. Compared with the Fe_3O_4/chitosan reference system, the GO-containing material exhibited reduced magnetic aggregation and a more homogeneous nanoparticle distribution. FTIR spectroscopy provided complementary molecular-level evidence of this structural

reorganization through modifications of O-H, N-H, C-O, and C-O-C vibrational bands, indicating hydrogen bonding and other interfacial interactions between graphene oxide and chitosan.

The structural integrity of the magnetite phase after graphene oxide incorporation was verified by XRD, while UV-Visible spectroscopy confirmed modifications in the electronic absorption characteristics associated with the formation of the hybrid interface. TGA/DTG analysis demonstrated the successful integration of the individual components and revealed thermal behavior consistent with the formation of stable hybrid nanostructures. Together, these complementary characterization techniques established coherent structure-property relationships and provided experimentally validated spectroscopic signatures of carbon-functional interfaces.

The obtained results directly contribute to the objectives of COST Action NanoSpace by providing laboratory-scale model systems for investigating carbonaceous nano-environments under well-controlled experimental conditions. The generated multi-technique dataset improves the understanding of how carbon-rich interfaces regulate nanoparticle organization, surface interactions, and physicochemical properties, thereby supporting the Action's objective of strengthening experimental research on carbon-based nanostructures and their interfacial behavior.

The STSM significantly strengthened scientific collaboration between Baku State University and the University of Calabria through joint experimental work, exchange of expertise, and interpretation of advanced characterization data. The results obtained during the mission constitute the basis of a joint scientific publication currently in preparation. Future collaboration will focus on extending the physicochemical investigation of graphene oxide-containing hybrid nanostructures, and participating in future COST Action activities, thereby reinforcing the long-term interdisciplinary research network established within NanoSpace.