

# Revealing the Atomic Structure and Ion Exchange Effects of One-Dimensional Lepidocrocite Nanofilament

Fatemeh Karimi<sup>1,\*</sup>, Francisco Lagunas<sup>1</sup>, and Robert Klie<sup>1</sup>

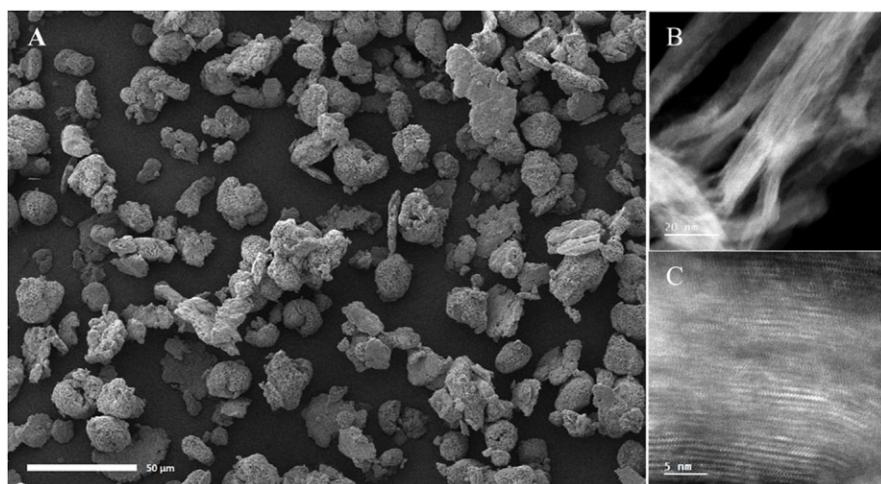
<sup>1</sup>Department of Physics, University of Illinois at Chicago, Chicago, Illinois, United States

\*Corresponding author: [Fkarim4@uic.edu](mailto:Fkarim4@uic.edu)

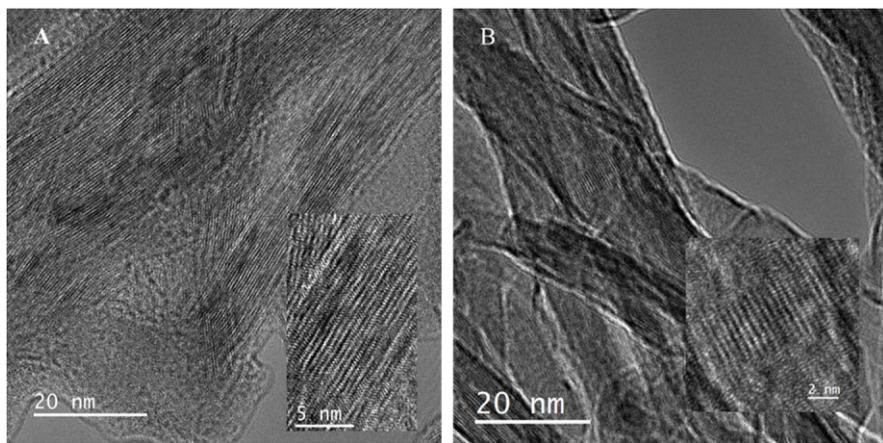
Low dimensional materials offer several advantages over bulk counterparts, such as high surface-to-volume ratios, quantum size effects, and tunable surface properties, etc. Nanostructured (NS) titania ( $\text{TiO}_2$ ) continues to attract considerable research interest due to its unique physical and chemical properties, along with its potential applications in diverse fields such as paint pigments, catalysis, photocatalysis, photoluminescence, gas sensors, and solar and fuel cells. In 2021, Badr et al. reported the synthesis of  $\text{TiO}_2$ -based one-dimensional (1D) Nano bundles, using a one pot, solution-reprecipitation for producing cost-effective, earth abundant materials in a highly scalable process [1].

In our recent study [2], we analyzed 1D  $\text{TiO}_2$  material using atomic resolution scanning transmission electron microscopy (STEM). Our findings reveal that the fundamental components of this material consist of 1D lepidocrocite nanofilament (NFs), which exhibit alignment and organization into diverse heterostructures. When (1DL NFs) are dried in ethanol without dispersion in water, they aggregate to form spherical, mesoporous particles (MPPs). These MPPs, comprising 1DL NFs, exhibit a highly ion-exchangeable space between the NFs. Consequently, the cations initially present can be readily replaced by  $\text{Li}^+$ , or  $\text{Na}^+$  ions following the reaction stage. Utilizing scanning/transmission electron microscopy (S/TEM) techniques, we examined these nanostructures at both cryogenic temperature (96 K) and room temperature (295 K). Our study also highlights the exciting prospect that the fundamental properties of these 1D  $\text{TiO}_2$ -based materials can be altered through ion exchange, showcasing the potential for manipulation with a wide range of other ions.

Atomic-resolution cryo-S/TEM analysis was conducted using an aberration-corrected cold field-emission JEOL ARM200CF operated at 200 kV primary electron energy with the emission current at 15  $\mu\text{A}$  together with the Gatan double-tilt  $\text{LN}_2$  stage. The electron probe semi-convergence angle was set to 17.8 mrad at 200 kV and the inner and outer detector angles for annular bright-field (ABF), low-angle annular dark-field (LAADF), and high-angle annular dark-field (HAADF) imaging were 11 mrad - 23 mrad, 30 mrad - 120 mrad and 68 mrad - 280 mrad, respectively. The beam current for imaging was 19 nA [3].



**Fig. 1.** (A) Low mag SEM images of 1DL-based mesoporous particles, (B and C) LAADF STEM micrographs—at various magnifications—of bundles of 1DL NFs oriented along the fiber axis and two Ti-atoms with a zigzag pattern.



**Fig. 2.** (A) TEM image showing a collection of TiO<sub>2</sub>-Na bundles, (A) at room temperature (B) at cryogenic temperature.

## References

1. HO Badr *et al.*, *Materials Today* **54** (2022), p. 8. <https://doi.org/10.1016/j.mattod.2021.10.033>
2. HO Badr *et al.*, *Matter* **6** 10 (2023), p. 3538. <https://doi.org/10.1016/j.matt.2023.07.022>
3. The authors gratefully acknowledge the support and assistance provided by collaborators at Drexel University, particularly Prof. Michel W. Barsoum and Dr. Hussein O. Badr from the Department of Materials Science & Engineering. Their expertise and guidance were instrumental in the success of this research project. We also extend our appreciation to the entire research team at Drexel University for their valuable contributions and support throughout the course of this study. This work is supported by a grant from the National Science Foundation (DMR-2309396). The acquisition of UIC JEOL JEM ARM200CF is supported by an MRI-R2 grant from the National Science Foundation (Grant No. DMR-0959470).