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## Supplementary information

# Extreme biomimetic approach: Hydrothermal synthesis of $\beta$ -chitin/ZnO nanostructured composites

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## Materials and methods

### Raman spectroscopy

The excitation of Raman scattering was obtained with a diode laser emitting at a wavelength of 785  
25 nm, propagated to the microscope with a 100  $\mu\text{m}$  optical fibre and focused on the samples by means of  
a 50x/0.75 microscope objective, leading to a focal spot of about 20  $\mu\text{m}$ . The Raman signal was  
collected in reflection configuration and sent to the f/1.8 holographic imaging spectrograph by using a  
62.5  $\mu\text{m}$  core optical fiber. The spectral resolution in the range 150-3250  $\text{cm}^{-1}$  was 4  $\text{cm}^{-1}$ . Raman  
spectra were punctually recorded, using an integration time of 2 s and averaging several spectra in  
30 order to improve the signal to noise ratio. Spectroscopic data were analyzed with MATLAB toolboxes  
(MathWorks Inc., Natick, USA). The baseline of Raman spectra was estimated within multiple  
windows of 150  $\text{cm}^{-1}$  width, shifted with 150  $\text{cm}^{-1}$  step; a linear interpolation method was chosen.  
Subsequent normalization of the set of spectra was obtained by standardizing the area under the curve  
to the group median value. Finally the data with high noise were smoothed using a Savitzky-Golay  
35 filter.

### Thermogravimetric analysis

The thermogravimetric analyzer (TG, model Jupiter STA 449F3, Netzsch) was used to investigate the thermal decomposition behavior of the samples. Measurements were carried out under flowing nitrogen (10 cm<sup>3</sup>/min) at a heating rate of 10°C/min over a temperature range of 25–1000°C, with an initial sample weight of approximately 5 mg.

### HRTEM

TEM images and electron diffraction were recorded by a FEI Tecnai 10 equipped with LaB6-source and at 100 kV acceleration voltage. Micrographs were recorded by means of TEM camera F224HD 2k x 2k (TVIPS company, Gauting, Germany) with an active area of 49 mm x 49 mm and a dynamic range of 25 000:1. For electron microscopy, a drop of the water suspension containing nanoparticulated sample was placed on the electron microscopy grid (Plano GmbH, Wetzlar, Germany) covered with a perforated carbon film. The sample thereafter was dried in air.

TEM was used to retrieve different microstructure parameters, namely crystallinity, crystallite size and shape, (local) preferred orientation of ZnO crystallites in the local regions of ZnO reference samples obtained without organic template.

In the selected area electron diffraction (SAED), high resolution TEM (HRTEM) and energy dispersive X-ray (EDX) methods were applied at 200 kV acceleration voltage by a transmission electron microscope JEM2200FS of JEOL equipped with C<sub>s</sub>-corrected illumination system, ultra-high resolution (UHR) objective lens (C<sub>s</sub>=0.5) and in-column filter. The corresponding micrographs and spectra were recorded by using of a 2k x 2k CCD-camera of Gatan Inc. and energy dispersive X-ray analyzer of JEOL, respectively. The evaluation of diffraction pattern and Fast Fourier Transformed (FFT) HRTEM-images was done by using of DigitalMicrograph software of Gatan Inc. taking into account the inverse interlattice plane distances and angles between lattice planes to obtain the orientation local regions of the sample.

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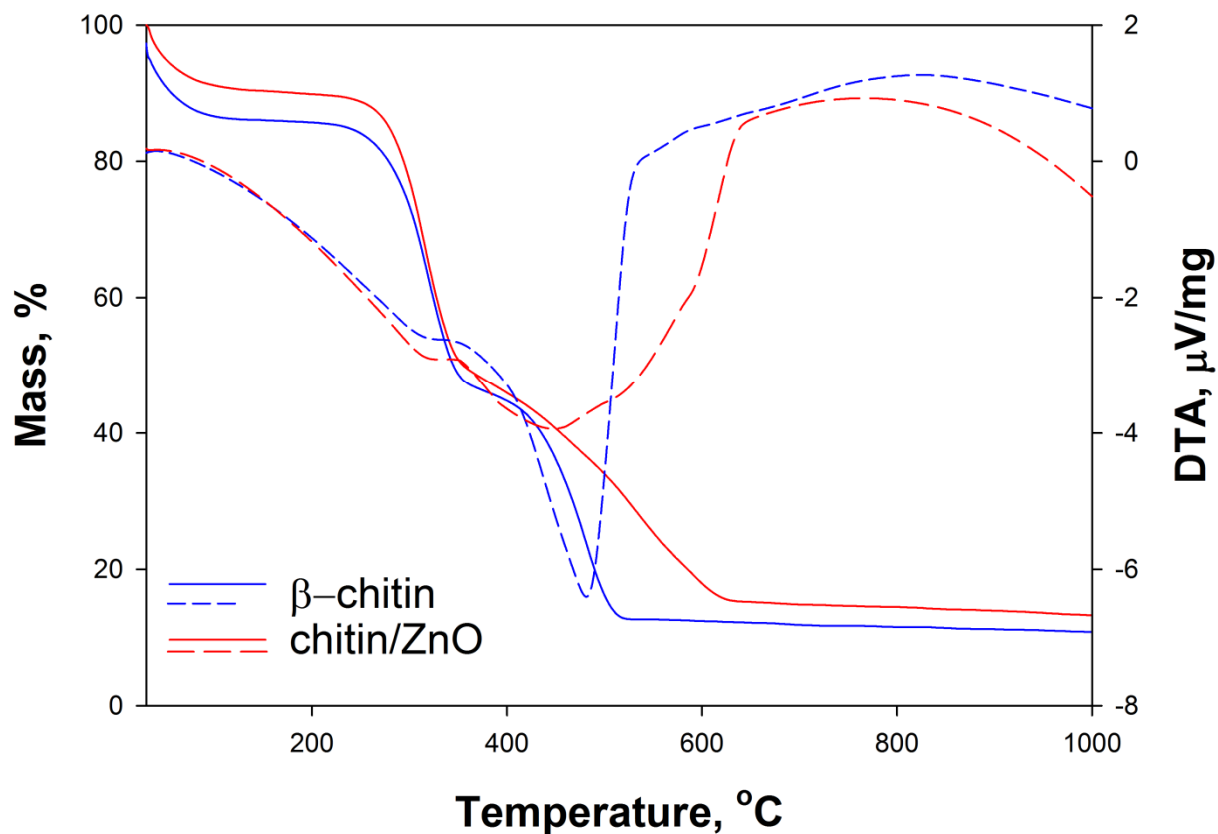


Fig. S1 TG/ DTA curves of  $\beta$ -chitin (blue line) and  $\beta$ -chitin/ZnO composite (red line)

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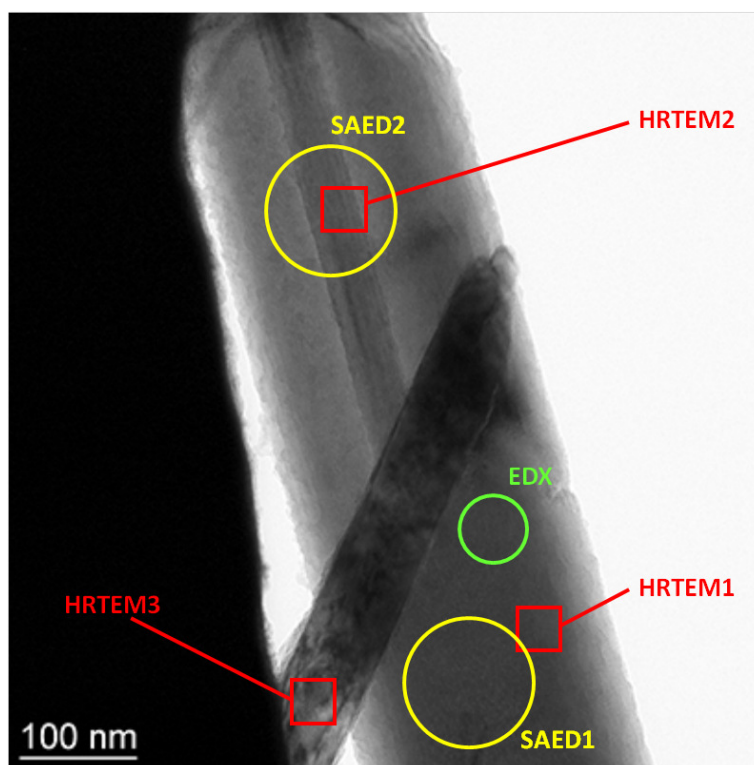


Fig. S2 Bright field image of investigated areas of ZnO crystallites.

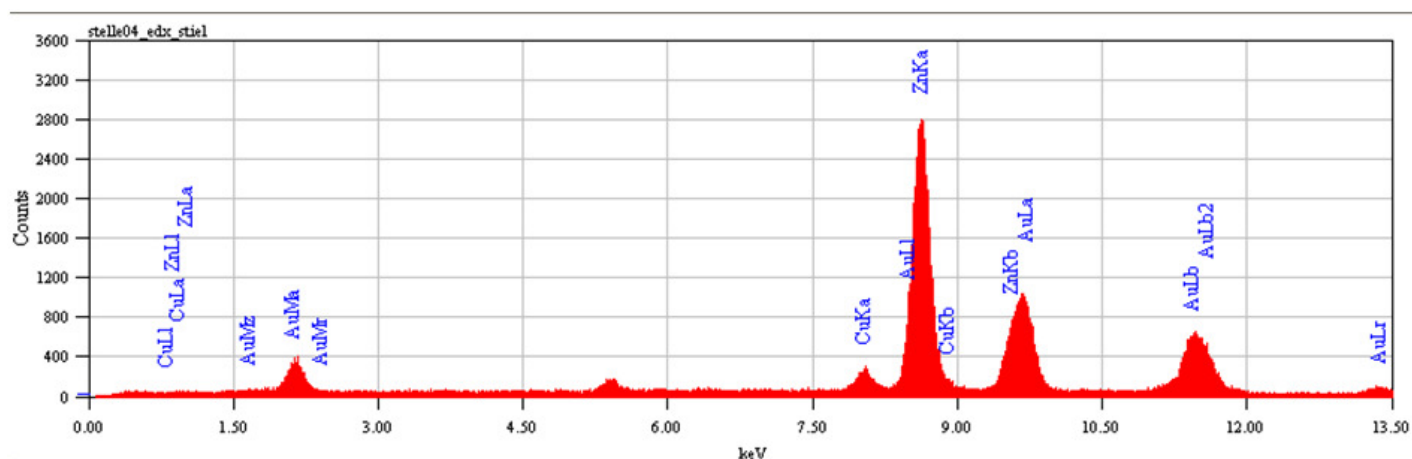
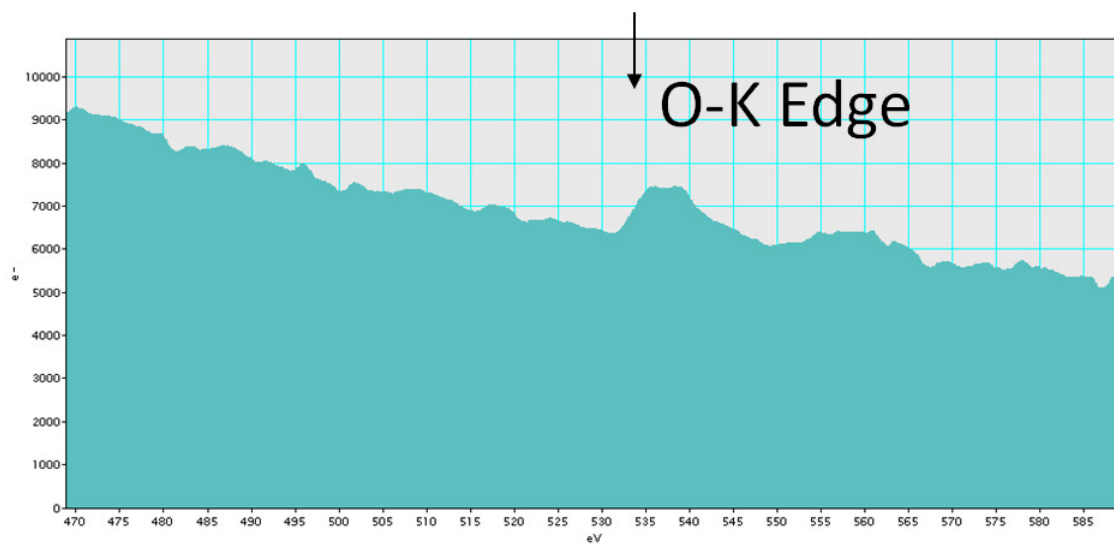
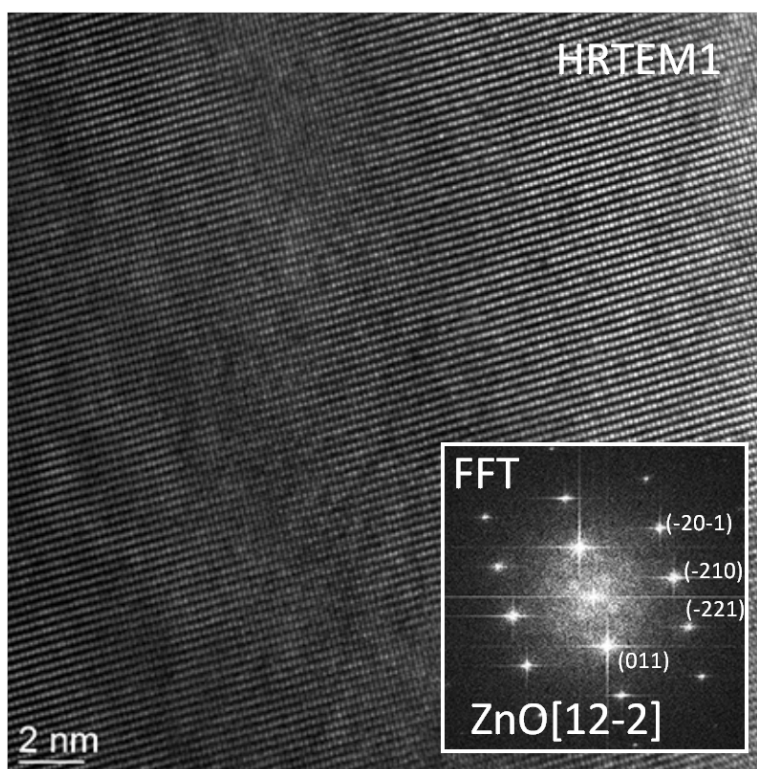


Fig. S3 EDX analysis of ZnO crystallites

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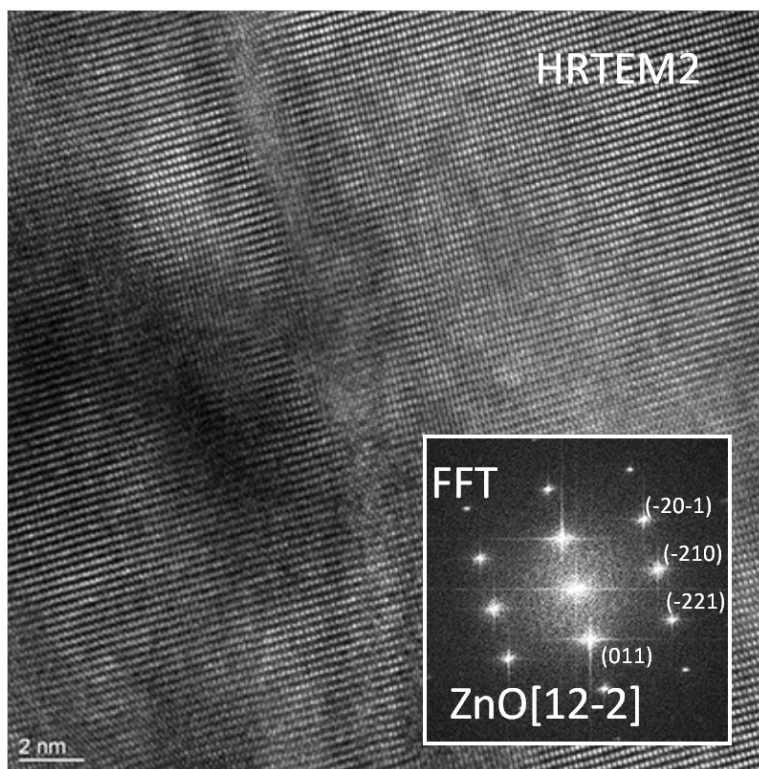


**Fig. S4** EELS spectra with O-K edge

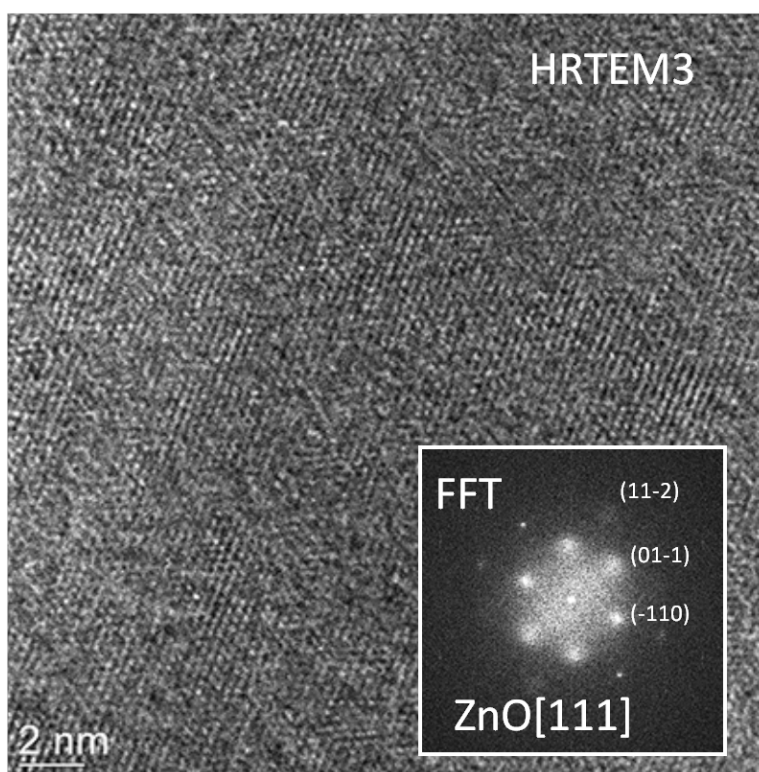


**Fig. S5** HRTEM image and corresponding indexed FFT of hexagonal ZnO with [12-2] orientation



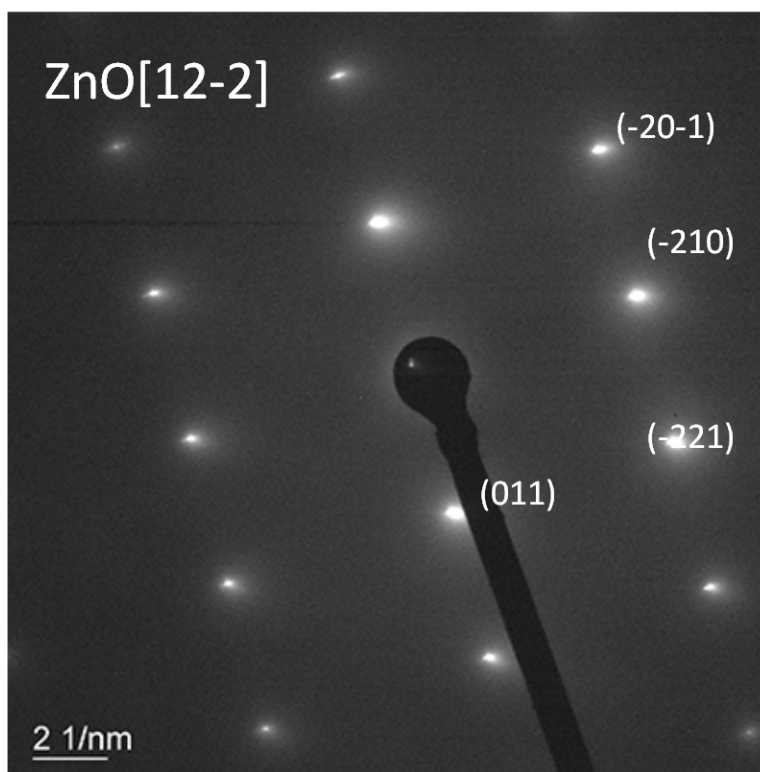


**Fig. S6** HRTEM image and corresponding indexed FFT of hexagonal ZnO with [12-2] orientation

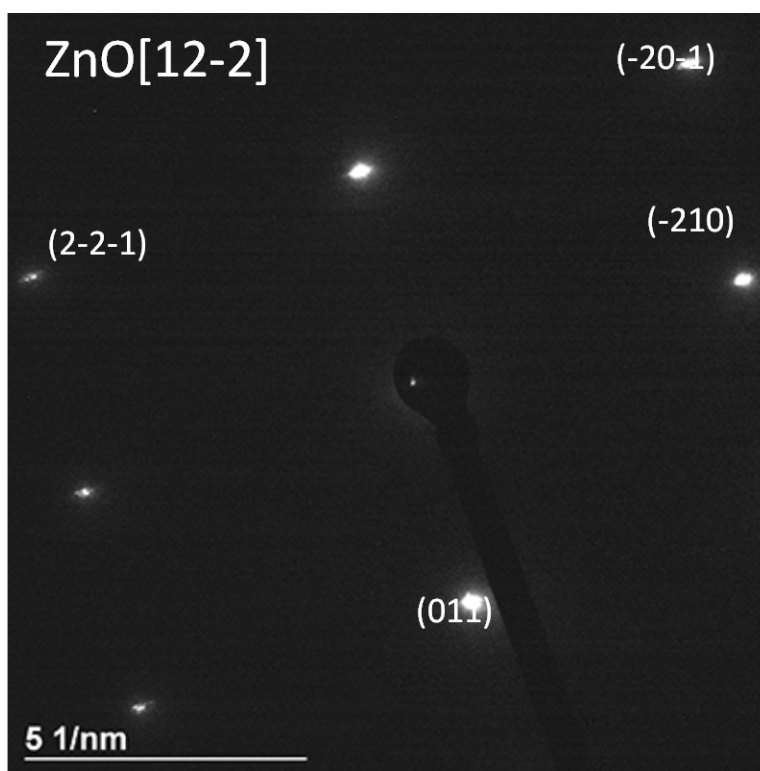


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**Fig. S7** HRTEM image and corresponding indexed FFT of other hexagonal ZnO crystal with [111] orientation



**Fig. S8** SAED from the bottom of the ZnO crystallite (camera length 40cm)



**Fig. S9** SAED from the upper region of the ZnO crystallite (camera length 60cm)