

Composition-dependent mechanism of formation of γ -Fe₂O₃/carboxymethylcellulose nanocomposites

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Materials

CMC sodium salt (CMC-Na) with molecular mass of 90,000 (a molar content of carboxylic groups equal to 0.7) (Sigma-Aldrich, USA), and Mohr salt (NH₄)₂Fe(SO₄)₂·5H₂O (Merck, USA), NaH₂PO₂ (VK Labor und Feinchemikalien, Germany), NaOH (Chemapol, Czech Republic) were used as received.

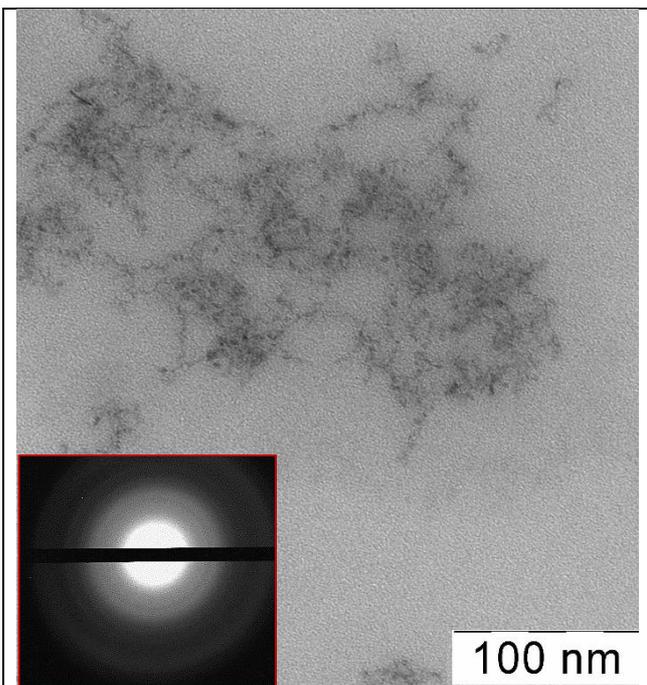
The iron-containing nanocomposite was synthesized as follows. A weighted amount of Mohr salt was dissolved in 1 mL of distilled water and mixed with 5 mL of 2% aqueous solution of CMC-Na and 1 mL of 2 mM NaOH aqueous solution. The resulting three-component system was vigorously intermixed for 2 min and then added to 1 mL of NaH₂PO₂ solution whose concentration ensured a molar ratio (NH₄)₂Fe(SO₄)₂ × 5H₂O/NaH₂PO₂ = 1/1. The reaction was completed when a color of the mixture changed from green to dark red. The resulting solution was purified from inorganic salts by dialysis.

Table S1 Composition of the reagents in reaction mixture.

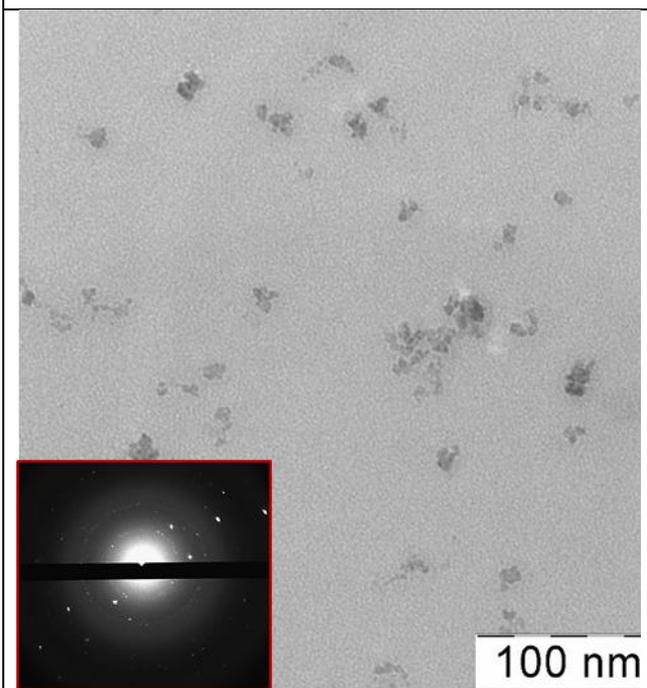
Sample	Molar ratio Fe:COOH
Composite I	0.04
Composite II	0.25
Composite III	1.0

Methods

Structural studies were carried out using transmission electron microscopy (TEM) with a JEM-100 B unit (JEML, Japan) equipped with an attachment for X-ray phase analysis. Samples were examined without preliminary contrasting. Nanoparticles sizes were established by means of statistical analysis of TEM images using MicroCal Origin software.



Composite I



Composite II

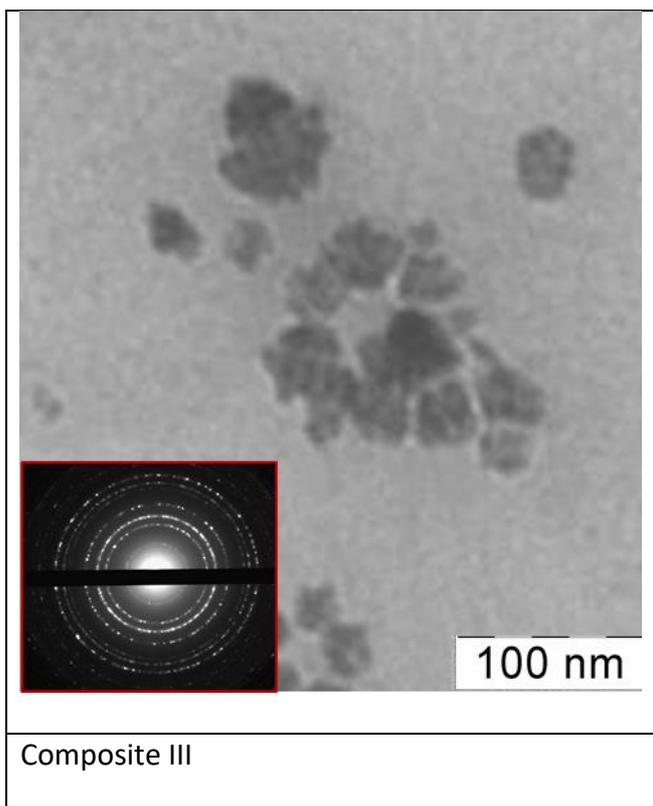


Figure S1 TEM images of the nanocomposites, the electron diffraction patterns of the nanocomposites are presented in inserts.

IR spectra were recorded on a Tensor 27 Bruker IR spectrometer (Germany) equipped with a liquid nitrogen cooled MCT detector with a Huber thermostat (USA). The measurements were carried out in a thermostatic cell of the disturbed total internal reflection (ATR, BioATR-II, Bruker, Germany) using a single-reflection crystal ZnSe at 22 ° C and a constant speed of purging the system with dry air using the Jun-Air apparatus (Germany). An aliquot (50 μ l) of the sample was applied to the crystal of the ATR cell, the spectrum was recorded three times in the range from 4000 to 950 cm^{-1} , with a resolution of 1 cm^{-1} ; performed 70-fold scanning and averaging. Background was recorded similarly.

The spectra were analyzed using the Origin 7.0 software.