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Supplementary Materials

Structural, Optical, and Catalytic Properties of $MgCr_2O_4$ Spinel-Type Nanostructures Synthesized by Sol–Gel Auto-Combustion Method

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Experimental Details

IR spectroscopy. To monitor the formation of the spinel structure and for monitor the absence of organic phases, FTIR spectroscopy has been used. The IR spectra were collected at room temperature by using a PerkinElmer Spectrum Two™ spectrometer in attenuated total reflectance (ATR) mode.

XRD study. The X-ray powder diffractograms (XRD) were recorded by using a Bruker D8 Advance diffractometer equipped with a Cu anode (λ = 0.15406 nm). The measurements were recorded at ambient condition for the samples, previously treated at 750 °C during 8 hours.

Scanning electron microscopy (SEM). SEM images were carried out with a Hitachi SU-70 Field Emission Scanning Electron Microscope (FE-SEM)

Elemental analysis were recorded with an Oxford Instrument EDX-detector equipped to Field Emission Scanning Electron Microscope.

The UV-Vis spectra were recorded by using an Ocean Optics USB2000 spectrophotometer and colloidal solutions of $MgCr_2O_4$ nanoparticles suspended in water in the presence of citric acid by.

The dielectric analysis was performed by using a CONCEPT 40 Broadband Dielectric Spectrometer (Novocontrol GmbH) equipped with an Alpha-A high performance frequency analyzer, in a frequency range from 0.1 Hz to 1 MHz).

Morphological Analysis

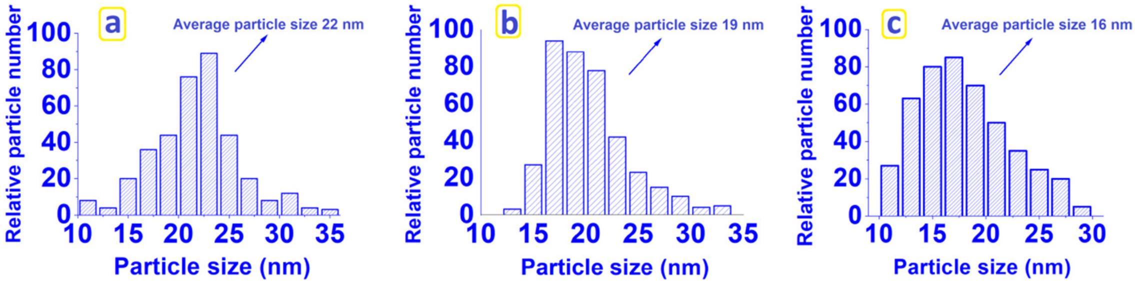

Figure S1. Measured average size of $MgCr_2O_4$_HMTA – (a), $MgCr_2O_4$_FS – (b) and, $MgCr_2O_4$_TA – (c) nanoparticles.

Table S1. Average particle size and size distribution of $MgCr_2O_4$ nanoparticles synthesized by hexamethylenetetramine, tartaric acid and fructose.

Series of nanoparticles	Size of nanoparticles, nm	Size distribution, (%)	Average particle size, nm
$MgCr_2O_4$_HMTA	10 – 19	18,2%	22
	19 – 25	69.0%	
	25 – 35	12.8%	
$MgCr_2O_4$_FS	10 – 17	7.6%	19
	17 – 22	66.7%	
	22 – 35	25.7%	
$MgCr_2O_4$_TA	10 – 13	5,8%	16
	13 – 19	64,8%	
	19 – 35	29,4%	

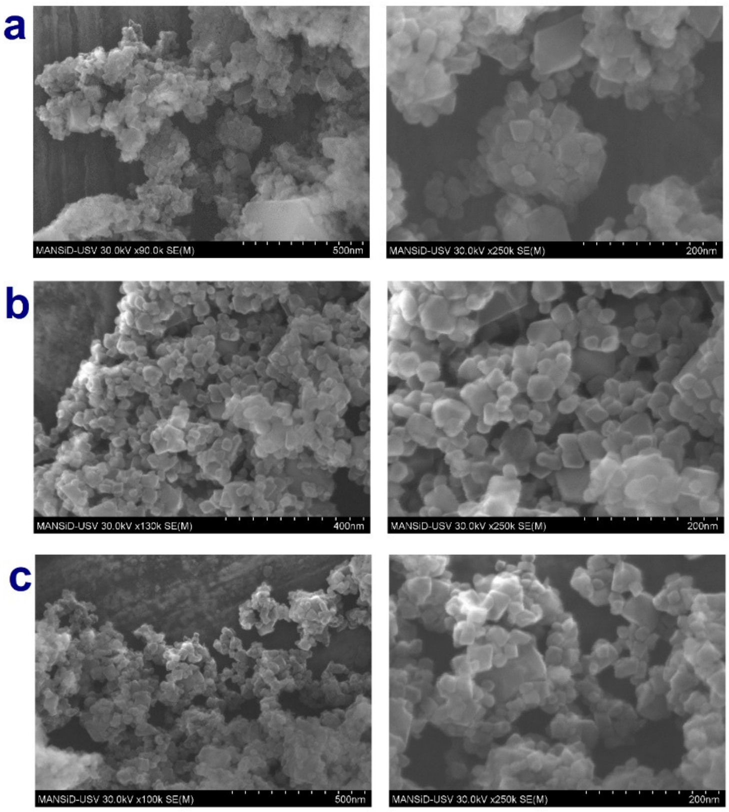

Figure S2. Set of SEM images used to determine the size histograms.

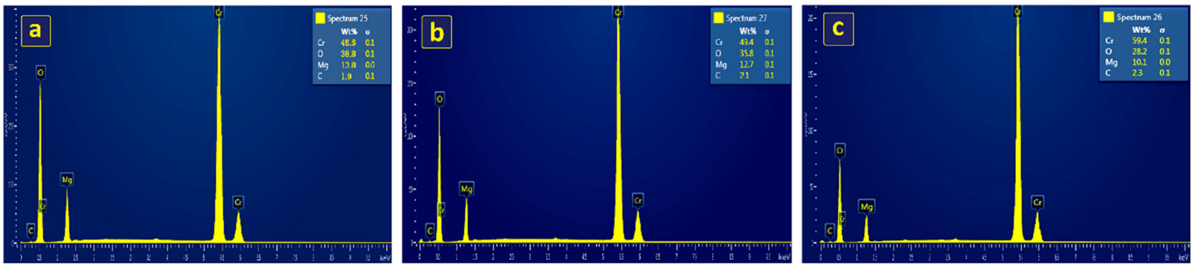

Figure S3. EDX elemental analysis of $MgCr_2O_4$ nanoparticles synthesized by hexamethylenetetramine (HMTA) - (**a**), fructose (FS) - (**b**) and tartaric acid (TA) - (**c**).

Broad-Band Dielectric Spectroscopopy Analysis

The dynamic analysis of the charge transport properties has been also investigated. Figure S4 shows the frequency dependence of the real part of the AC conductivity and of the dielectric constant, recorded in the 1 Hz - 1 MHz frequency range, at room temperature. As expected, the $MgCr_2O_4$_HMTA nanoparticles, characterized by a higher diameter, are the most conductive and have the highest value of the dielectric permittivity among the three samples (ca. 21 at 1Hz). On the other hand, the $MgCr_2O_4$_TA particles, which have the smallest diameter, are less conductive and have a dielectric constant of ca. 10 at 1Hz. An important Maxwell-Wagner relaxation due to space charge effect at the interface between nanoparticles is observed in all studied samples [1, 2].

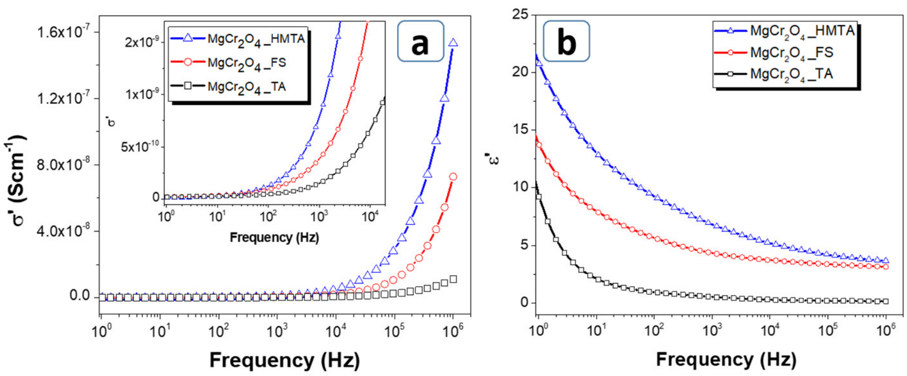

Figure S4. Frequency dependence of the real part of the AC electrical conductivity (**a**) and of the real part of dielectric permittivity (**b**) recorded at room temperature, on the three series of spinel $MgCr_2O_4$ nanoparticles.

Catalytic properties were simply analyzed. Volume of the produced oxygen during the H_2O_2 decomposition was measured as a function of water volume (see Figure S5).

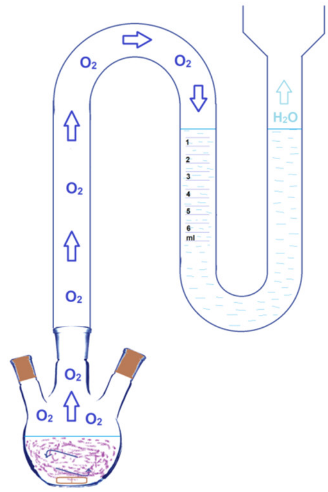

Figure S5. Schematic representation of experimental setup used in the evaluation of the catalytic properties.

References

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2. Wagner, K.W. Erklärung der dielecktrischen nachwirkungsvorgänge auf grund Maxwellscher vorstellungen. *Arch. Electrotech.* **1914**, *2*, 371–387.