Study of ZnO nanoparticle generation for long lasting exposure experiments

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Keywords: externally heated tube reactor, chemical composition, chemical vapor synthesis, nanoparticle generation.

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Generally, nanoparticles (NPs) exhibit unique chemical, optical, electrical, and even toxic properties due to their strong size and shape dependence. Despite increasing applications of ZnO NPs as UV emitting diodes to gas sensors, data about potential toxic properties are still rather rare and contradictory (Heckenberg *et al.* 2011). In this work we tested the long term generation of ZnO NPs for potential use in exposure experiments with laboratory animals.

Experiments were performed in an externally heated work tube, made from impervious aluminous porcelain, with an i.d. of 25 mm and a 1 m-long heated zone. A boat with zinc shot was inserted into the middle of the heating section. Nitrogen carrier gas, saturated to some extent by Zn vapours, was mixed in the outlet part of the work tube (diluter) with a stream of air, where oxidation of Zn vapours was carried out and ZnO NPs formation via gas-to-particle process occurred. Particle production was monitored using SMPS (TSI model 3936L75). Samples for NPs characterization were deposited onto TEM grids using a Nanometer Aerosol Sampler (TSI model 3089) and on cellulose and Sterlitech Ag filters. NPs characteristics were studied with high resolution TEM (*JEOL 3010*), energy dispersive spectroscopy (EDS, *INCA/Oxford*), inductively coupled plasma – optical emission spectrometry (ICP-OES, *IRIS Intrepid II XDL*), X-ray diffraction (XRD, *Philips X'Pert PW3020*), and X-ray photoelectron spectrometry (XPS, *Axis Supra, Kratos Analytical Ltd.*).

Two experimental campaigns, with total duration 102 hours, were performed. The particle production by chemical vapor synthesis method proceeded satisfactorily and could be controlled by adjusting experimental conditions: reactor (work tube) temperature T_R=520-530 °C, reactor flow rate Q_R=600-1000 cm^3/min, and flow rate of diluting air Q_{Dil}=1800-2200 cm^3/min.

Morphology of NPs was studied by HRTEM and an example of a TEM image is shown in Figure 1. Primary particles are mostly spherical, agglomerated into clusters of various sizes, and the size of primary particles varies from 10 to 20 nm. EDS analyses showed an almost stoichiometric ratio of Zn to O for ZnO. ICP-OES analyses of the samples deposited on cellulose filters determined the content of Zn in the samples to be 71 wt. %, which corresponds to the value 89 wt. % of ZnO. XPS analysis of the surface layer of NPs determined the presence of Zn in the bonds Zn-O. XRD analysis of four samples detected hexagonal ZnO crystalline phase in all samples, typically ICDD Pdf 89-0510. Mean crystallite size, calculated from Scherrer equation, varied between 12.5 and 13.7 nm. Results obtained by XRD were confirmed by selected area electron diffraction method.

In conclusion, the method of evaporation of metallic zinc and subsequent oxidation of Zn vapours by a stream of diluting air can generate NPs of a desired size and number concentration for a sufficiently long time. The method can be applied for long lasting exposure experiments with laboratory animals.

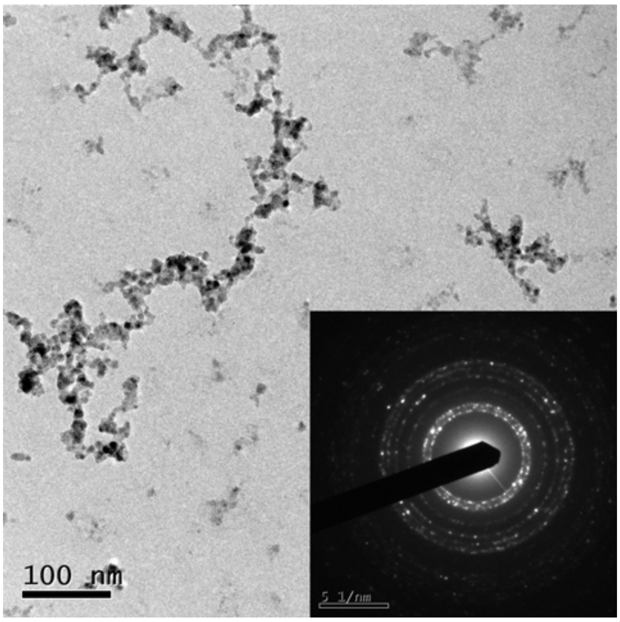

Figure 1. TEM image and SAED pattern of the sample synthesized at T_R=530 °C, Q_R=800 cm^3/min and Q_{Dil}=1800 cm^3/min.

This work was supported by the Czech Science Foundation under grant P503/12/G147. ICP-OES analyses were performed by Dr. Šárka Matoušková, Geological Institute of the CAS, v.v.i., XRD analyses by Mgr. Anna Kallistová, Geological Institute of the CAS, v.v.i., and XPS analyses by Dr. Josef Zemek, Institute of Physics of the CAS, v.v.i.

Hackenberg, S., Scherzed, A., Technau, A., *et al.* (2011) *Toxicology in vitro.* **25**, 657-663.