

Laboratory of Nanostructures
INSTITUTE OF HIGH PRESSURE PHYSICS
Polish Academy of Sciences

Nanoparticles with precisely regulated size

Collaboration offer for the European Nanomedicine Technology Platform Partners

- Application of nanomaterials
- Delivering of nanoparticles for modelling studies
- Studying nanoparticle size dependent properties
- Developing of new nanomaterials
- Scaling up nanomaterials
- High quality nanomaterials characterisation
- Joint projects

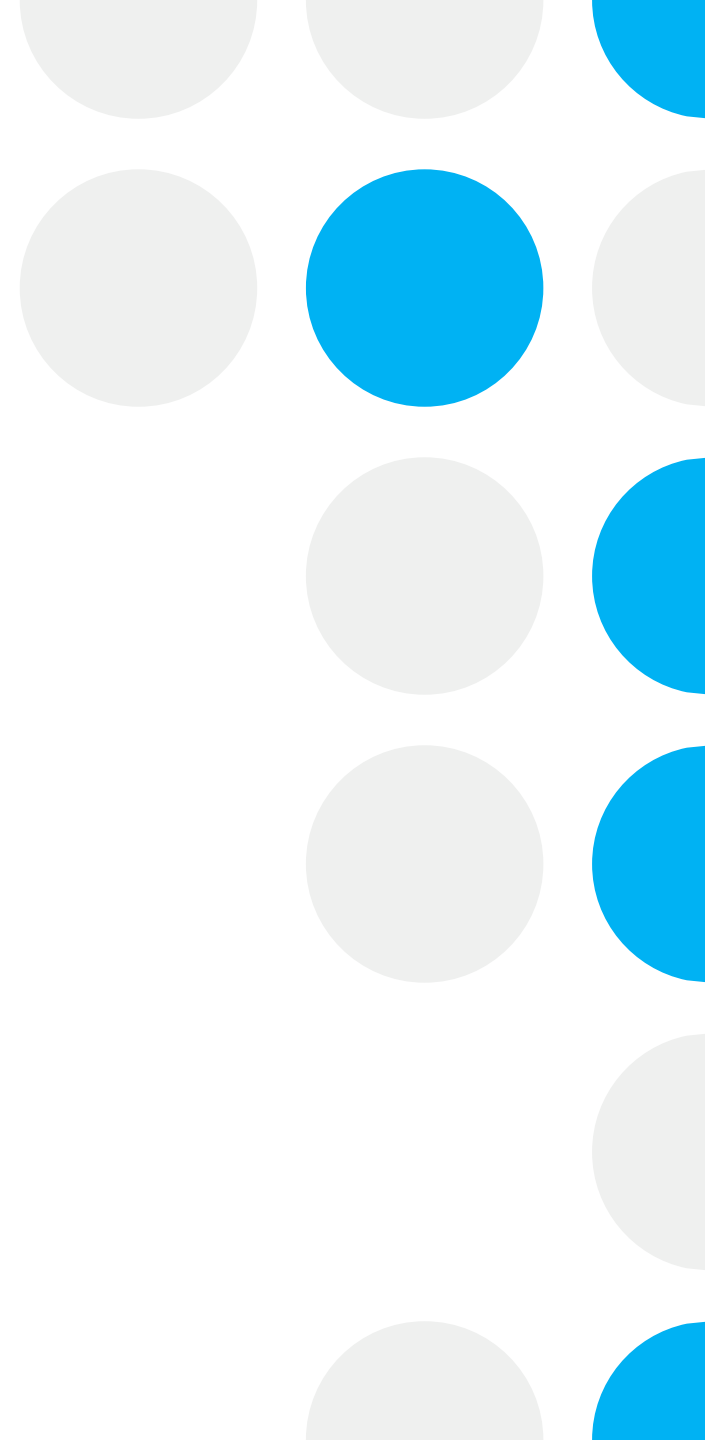
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Nanoparticles for research on size effects in nanoscale

Precise regulation of crystallites size permits to:

- compare real properties with these from modelling experiments
- measure size dependent properties





HAP with particle size from 8 to 39 nm

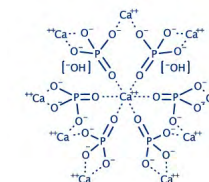
Crystallite (particle) size of hydroxyapatite nanoparticles (GoHAP™)
produced in the Laboratory of Nanostructures and Nanomedicine IHPP PAS

GoHAP™ type	SSA ¹ [m ² /g]	Density [g/cm ³]	Grain size, from SSA ² [nm]	Grain size, from XRD ³ d _i ; d _w ⁴ [nm]	Grain size, from TEM, [nm] ⁵
GoHAP™ 1	258 ± 25	2.86 ± 0.02	8 ± 1	19 ± 9; 6 ± 2	6,5 ± 0.5
GoHAP™ 2	211 ± 20	2.92 ± 0.02	10 ± 1	24 ± 9; 7 ± 5	7.3 ± 0.3
GoHAP™ 3	149 ± 14	2.95 ± 0.01	14 ± 1	28 ± 12; 14 ± 6	11.7 ± 0.03
GoHAP™ 4	85 ± 14	3.00 ± 0.01	23 ± 2	38 ± 17; 23 ± 6	18.4 ± 0.6
GoHAP™ 5	61 ± 6	3.03 ± 0.01	32 ± 3	50 ± 20; 30 ± 9	26.9 v 0.6
GoHAP™ 6	51 ± 5	3.04 ± 0.01	39 ± 4	60 ± 20; 33 ± 9	34.8 ± 0.9

¹ Specific Surface Area; ² Calculated from SSA; ³ Calculated from XRD patterns using Scherrer's Formula; ⁴ ZnO crystallites have a hexagonal structure. d_a – size along the a axis; d_c – size along the c axis; ⁵ Measured by means of TEM – Transmission Electron Microscopy.

Source: Influence of hydrothermal synthesis parameters on the properties of hydroxyapatite nanoparticles
Sylvia Kuśnieruk et al., *Beilstein J. Nanotechnol.* 2016, 7, 1586–1601. doi:10.3762/bjnano.7.153

Hydroxyapatite
or Hydroxylapatite
HAP
 $\text{Ca}_{10}(\text{PO}_4)_6(\text{OH})_2$



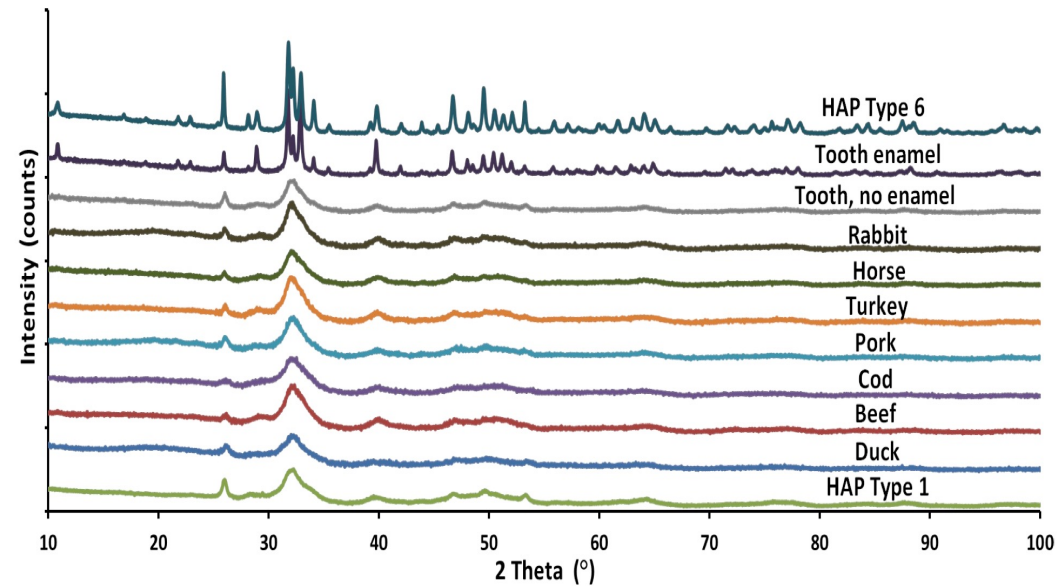
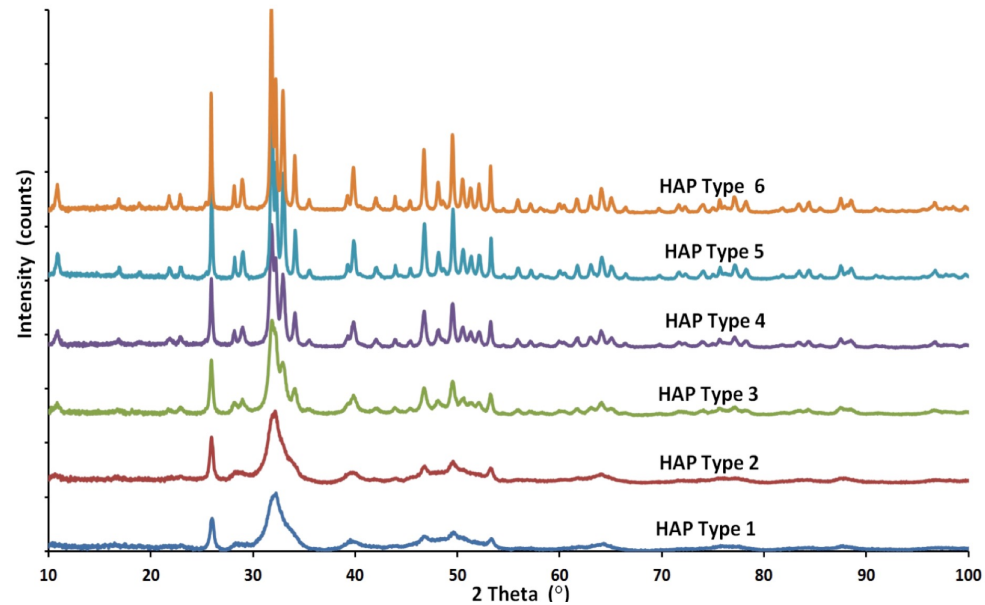
Source : <https://www.chromospheres.com/nano-hydroxyapatite-powder/>

GoHAP™ is a synthetic nanomaterial mimicking Hydroxyapatite (HAP) in human bone. It was successfully used for bone regeneration in many veterinary operations.





XRD patterns of synthetic GoHAP™ 1-6 and natural HAP



GoHAP™ 1 = natural bone. GoHAP™ 6 = tooth enamel



ZnO with particle size regulated from 16 to 43 nm

Crystallite (grain) size of ZnO nanoparticles produced
in the Laboratory of Nanostructures and Nanomedicine IHPP PAS

ZnO type	SSA ¹ [m ² /g]	Density [g/cm ³]	Grain size, from SSA ² , [nm]	Grain size, from XRD ³ d _a ; d _c ⁴ [nm]	Grain size, TEM, [nm] ⁵
ZnO 1	74.7	5.12 ± 0.03	16 ± 1	16 _a ; 20 _c	
ZnO 2	44.9	5.32 ± 0.03	25 ± 1	26 _a ; 37 _c	
ZnO 3	28.9	5.40 ± 0.02	29 ± 1	34 _a ; 51 _c	
ZnO 4	26.5	5.42 ± 0.04	43 ± 1	35 _a ; 51 _c	
ZnO 5	25.9	5.42 ± 0.03	43 ± 1	35 _a ; 53 _c	39.5 ± 0.5

¹ Specific Surface Area; ² Calculated from SSA; ³ Calculated from XRD patterns using Scherrer's Formula; ⁴ ZnO crystallites have a hexagonal structure. d_a – size along the a axis; d_c – size along the c axis; ⁵ Measured by means of TEM – Transmission Electron Microscopy.

Source: *Size control mechanism of ZnO nanoparticles obtained in microwave solvothermal synthesis*, Jacek Wojnarowicz *et al* 2018 *Nanotechnology* **29** 065601



Up to 115 nm achievable



ZnO doped with Co^{2+} up to 10 wt%

Crystallite (particle) size of $\text{ZnO}_{0.9}\text{Co}_{0.1}$: nanoparticles produced in the Laboratory of Nanostructures and Nanomedicine IHPP PAS.

ZnO type	SSA ¹ [m ² /g]	Density [g/cm ³]	Grain size, from SSA ² , [nm]	Grain size, from XRD ³ d _a ; d _c ⁴ [nm]	Grain size, TEM ⁵ , [nm]
ZnO 1	42.6 ± 0.1	5.05 ± 0.04	28 ± 2	23 _a ; 26 _c	23 ± 1
ZnO 2	37.3 ± 0.1	5.13 ± 0.03	31 ± 3	27 _a ; 27 _c	31 ± 1
ZnO 3	31.7 ± 0.1	5.26 ± 0.03	36 ± 3	28 _a ; 33 _c	34 ± 1
ZnO 4	28.8 ± 0.1	5.30 ± 0.03	39 ± 3	30 _a ; 37 _c	38 ± 1
ZnO 5	21.2 ± 0.1	5.35 ± 0.02	53 ± 3	36 _a ; 50 _c	52 ± 3

¹ Specific Surface Area; ² Calculated from SSA; ³ Calculated from XRD patterns using Scherrer's Formula; ⁴ ZnO crystallites have a hexagonal structure. d_a – size along the a axis; d_c – size along the c axis; ⁵ Measured by means of TEM – Transmission Electron Microscopy.

Source: *Size Control of Cobalt-Doped ZnO Nanoparticles Obtained in Microwave Solvothermal Synthesis*; Jacek Wojnarowicz et al. 2018 *Crystals* **8**, 179; doi:10.3390/cryst8040179

Mn^{2+} as well as
 Mn^{2+} & Co^{2+} cooping
Available



Antibacterial ZnO: 2 wt% Ag nanoparticles with grain size from 22 to 38 nm

Crystallite (particle) size of ZnO: 2 wt% Ag nanoparticles produced in the Laboratory of Nanostructures and Nanomedicine IHPP PAS.

ZnO type	SSA ¹ [m ² /g]	Density [g/cm ³]	Grain size, SSA ² , [nm]	Grain size, XRD ³ d _a ; d _c ; d _{Ag} ⁴ [nm]
ZnO 1	50.7 ± 0.1	5.32 ± 0.01	22 ± 2	21 _a ; 25 _c 28-57 _{Ag}
ZnO 2	38.4 ± 0.1	5.40 ± 0.03	30 ± 3	25 _a ; 38 25-57 _{Ag}
ZnO 3	31.8 ± 0.1	5.40 ± 0.03	35 ± 3	31 _a ; 45 _b 25-57 _{Ag}
ZnO 4	29.0 ± 0.1	5.43 ± 0.01	38 ± 3	34 _a ; 52 _b 25-57 _{Ag}

¹ Specific Surface Area; ² Calculated from XRD patterns using Scherrer's Formula. ³ ZnO crystallites have a hexagonal structure. d_a – size along the a axis; d_c – size along the c axis; d_{Ag} – size of the Ag nanocrystals
Source: Internal report of Laboratory of Nanostructures, IHPP PAS, author J. Wojnarowicz

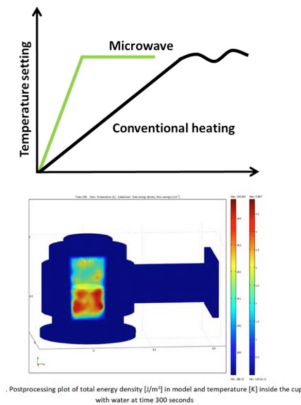


Particle size control and high yield production using MSS Reactors for Microwave Solvothematic Synthesis

MSS-4 reactor

MSS -2 reactor

- $T_{\max} = 250^{\circ}\text{C}$, $P_{\max} = 6 \text{ MPa}$,
- Teflon vessels volume 0,3 l
- Heating rate up to 1,8 K/s
- Process duration is controlled with high accuracy
- High power density delivered in the reagents: approximately 10 W/ml
- Developed in collaboration with Lukasiewicz – Institute of Sustainable Technologies



The production process is clean, in a rigid technological regime, reproducible and scalable



Nanomaterials characterisation services offered by Labnano

- Particle size distribution, by several methods
 - Specific surface, surface energy, nano and micro porosity
 - Zeta potential
 - Suspensions stability
 - Chemical composition, Phase composition
 - TEM, FE SEM, EDS, XRD
 - TG+DSC+FTIR+MS
 - Selected optical properties
 - Other
-





Collaboration offer

Bilateral or in joint EU projects.

- Delivering of nanoparticles for modelling studies and size dependent properties
- Developing of new nanomaterials
- Investigation of size dependent properties
- Scaling the nanomaterials rial production to industrial scale
- Joint projects on nanomaterials application
- High quality characterisation of nanoparticles