Electronic supplementary material

Use of alkaline or enzymatic sample pre-treatment prior to characterization of silver nanoparticles in human tissue by single particle ICP-MS

Janja Vidmar,[a,b] Tina Buerki-Thurnherr,[c] Katrin Loeschner*[d]

[a]*Department of Environmental Sciences, Jožef Stefan Institute, Jamova 39, 1000 Ljubljana, Slovenia*

[b]*Jožef Stefan International Postgraduate School, Jamova 39, 1000 Ljubljana, Slovenia*

[c]*Particles-Biology Interactions, Empa, Swiss Federal Laboratories for Materials Science and Technology, Lerchenfeldstrasse 5, 9014 St. Gallen, Switzerland*

[d]*Division for Food Technology, National Food Institute, Technical University of Denmark, Kemitorvet 201, DK-2800 Kgs. Lyngby, Denmark*

*Corresponding author:
Katrin Loeschner
Phone: +45 35887029
Fax: +45 3588 7448
Email: kals@food.dtu.dk

Table S1 spICP-MS settings

Parameter (unit)	Value
RF power (W)	1550
Plasma gas flow rate (L/min)	15
Nebulizer gas flow rate (mL/min)	1.03
Cell gas (flow rate) (mL/min)	n/a
Sample uptake flow rate (mL/min)	0.30 – 0.33[a]
Monitored isotopes (*m/z*)	^{107}Ag, ^{197}Au[b]
Dwell time (ms)	3
Analysis time (s)	180 - 600
Nebulizer type	MicroMist (Agilent G3266-65003; borosilicate glass)
Spray chamber type	Scott double-pass, Peltier-cooled (PFA)

[a] Determined on a daily basis (corresponding peristaltic pump speed was 0.1 rounds/s)
[b] For determination of transport efficiency

Determination of the Ag concentration in tissues by conventional ICP-MS

The Ag mass concentrations in the acid digested tissues were determined by conventional ICP-MS analysis. 2 mL of HNO_3 (65%) were added to approximately 0.7 g of tissue, followed by microwave-assisted digestion (12 min heating up to 250°C, 8 min at 250°C, cooling down to RT). The digests were filled up to 10 mL with UPW, and to 1 mL aliquot 4 mL of HNO_3 (65%) and 4 mL of HCl (30%) were added. The so-prepared samples were left overnight at the RT and filled up to 10 mL with UPW the next day. All digestions were performed in duplicate. For evaluation of the accuracy of the analytical method (digestion procedure + ICP-MS analysis), 250 mg of CRM for tuna fish (ERM-CE464) was spiked with 150 µL of 100 µg/mL ionic Ag solution to a concentration of 60 µg/g. Spiked samples were digested in exactly the same way as the tissues. For spiked CRM, recoveries of 24.9% ± 9.7% (N=3) were achieved. Ag concentrations determined in the digested tissue samples were corrected with the average recovery of the spiked CRM samples.

Prior to the quantification of the Ag mass concentration by ICP-MS against an external calibration curve and with the use of In as internal standard, the digests were 5-times diluted with UPW. Calibration standard solutions of Ag were prepared from Ag stock solution (1000 µg Ag/mL in 2-3% HNO_3) and diluted in 5.2% HNO3 and 2.4% HCl. Experimental conditions for the 7900 ICP-MS instrument are summarised in Table S2.

Table S2 ICP-MS settings for the determination of Ag mass concentration in the tissues

Parameter	**Value**
Plasma power	1,550 W
Plasma gas flow rate	15 L/min
Carrier gas flow rate	1.05 L/min
Sample uptake flow rate	0.1 rounds/sec
Isotopes monitored	^{107}Ag, ^{115}In
Integration time per isotope	300 ms
Nebulizer type	MicroMist
Spray chamber type	Scott chamber, Peltier-cooled

Table S3 Comparison of Ag concentrations (in nanograms of Ag per gram of placental tissue) in placental tissues collected before the start of perfusion, obtained by spICP-MS after enzymatic treatment and by conventional ICP-MS after digestion with acids. The given values represent the mean ± STD of two determinations for each sample

Perfusion experiment	Enzymatic digestion (spICP-MS)	Acid digestion (ICP-MS)
AgPEG NPs	17.8 ± 1.2	<36.6
AgCOONa NPs	14.2 ± 1.6	<15.6
Ag +	15.5 ± 1.0	<15.6
control	27.3	24.5

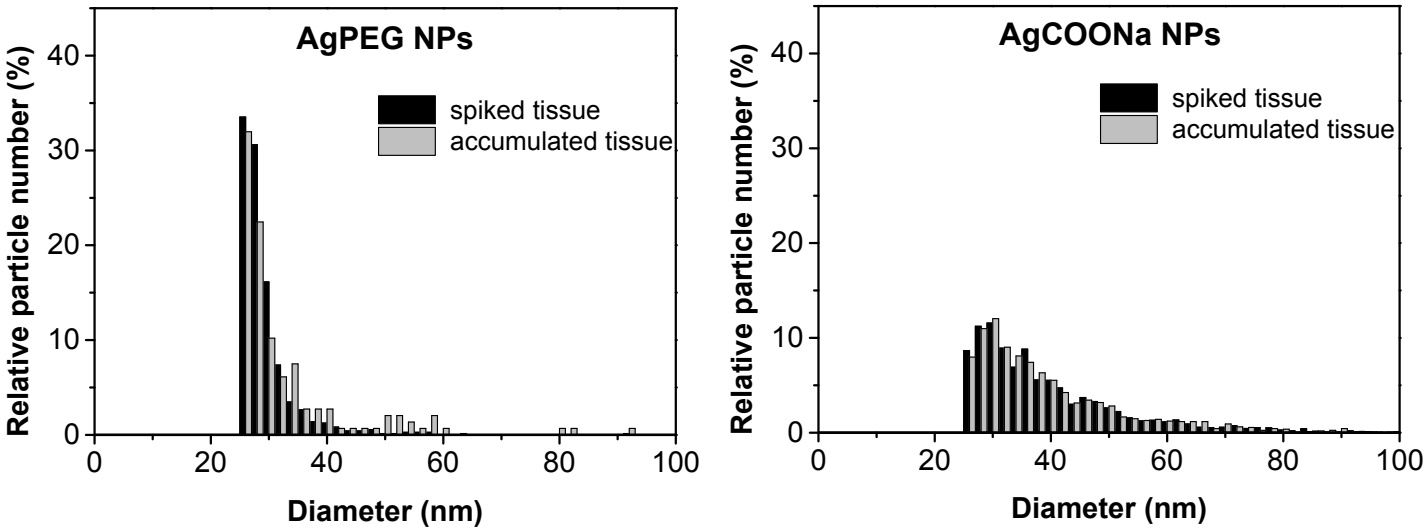

Fig. S1 Particle size distribution (bin size 2 nm) obtained by spICPMS for AgPEG NPs and AgCOONa NPs in spiked placental tissue (black histogram) and placental tissue in which AgNPs accumulated after 6h of perfusion (gray histogram). All placental tissues were enzymatically treated.