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Electronic Supplementary Information (ESI)

Silver nanoparticles as solid sorbent in ultrasound-assisted dispersive micro solid-phase extraction for the atomic absorption spectrometric determination of mercury in water samples

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1. Instrumentation

Analytik Jena ContrAA 700 spectrometer comprises a compact high-resolution double echelle monochromator and a charge-coupled device (CCD) array detector with a resolution of about 2 pm per pixel in the far-ultraviolet range. In this research, a transversely heated graphite atomizer (ET) with pyrolytically coated graphite tubes was employed for atomization of analyte.

The operating parameters of the HR-CS ETAAS instrument during mercury determination after AgNPs USA DMSPE are summarized in Table S1.

For SPE procedure the AgNPs were weighed using an M2P microanalytical balance (Sartorius, Gottingen, Germany) with a resolution of 1 μg (electronic weighing range up to 2 g). The pH values were measured with a pH-meter (pH 211 Microprocessor, Hanna Instruments, Kehl, Germany) supplied with a glass-combined electrode. In the first stage of the study, the samples with AgNPs were shaking using a Genius 3 vortex mixing machine (IKA, Staufen, Germany). A Sonopuls HD 70 ultrasonic cell disruptor/homogenizer (70 W, 20 kHz, Bandelin, Germany) equipped with a 2-mm titanium microtip was used for dispersive extraction processes. Ultrasonic energy was fixed at any desired level using a power setting in the 10-65 W for 2-mm titanium microtip. Additionally, a centrifuge (Model EBA 20, Hettich, Germany) was employed for phase separation after extraction procedure.

Table S1
Optimized experimental conditions for ultrasound-assisted dispersive micro solid-phase extraction (USA DMSPE) with AgNPs as sorbent material prior to AAS determination of mercury (parameters for HR-CS ETAAS are also presented).

USA DMSPE with AgNPs	
Sample volume (mL)	10
Amount of AgNPs (mg)	10
pH of sample solution	3.5
Sonication time (min)	0.5
Centrifugation time (min)	1
Solvent for solid phase / concentration (mol L^{-1}) / vol. (μL)	HNO_3 / 7 / 200
HR-CS ET AAS detection	
Wavelength (nm)	253.6519
Lamp current (A)	9
Spectral range (pixel)	200
Dispersion (pm $pixel^{-1}$)	2
Read time (s)	5
Delay time (s)	0
Measurement mode	peak height
Sample volume (μL)	20
Modifier	$Pd(NO_3)_2/Mg(NO_3)_2$
Modifier concentration (μg μL^{-1})	2
Modifier volume (μL)	5
Furnace program steps	
Drying	80 °C, ramp 6 °C s^{-1}, hold 20 s
Drying	90 °C, ramp 3 °C s^{-1}, hold 20 s
Drying	120 °C, ramp 5 °C s^{-1}, hold 10 s
Pyrolysis	300 °C, ramp 50 °C s^{-1}, hold 20 s
Atomization	1300 °C, ramp 2000 °C s^{-1}, hold 3 s
Cleanout	1600 °C, ramp 500 °C s^{-1}, hold 4 s

2. Preconcentration and AAS determination procedures

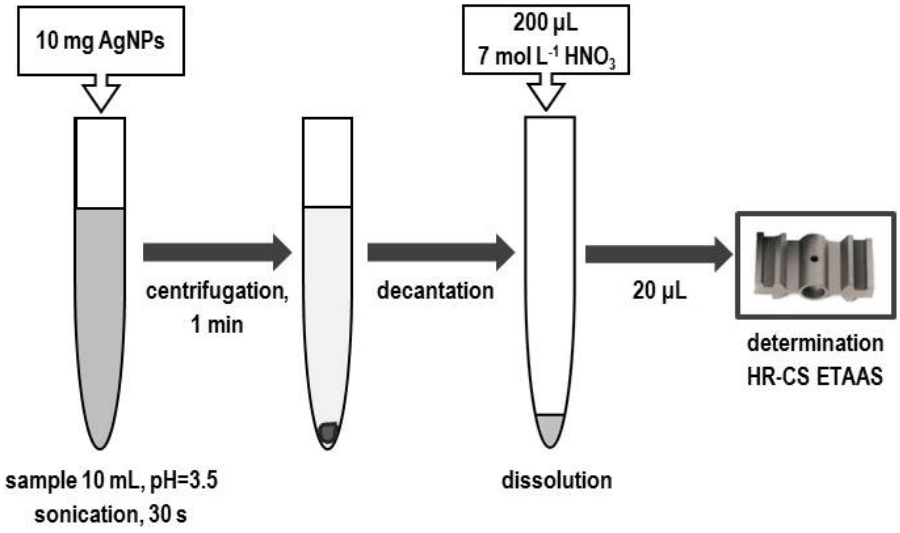

Fig. S1. Schematic diagram for AgNPs ultrasound-assisted micro dispersive SPE procedure combined with HR-CS ETAAS for the determination of mercury.

3. Results and Discussion

3.1 Optimization of AgNPs USA MDSPE conditions

3.1.1. Particles amount

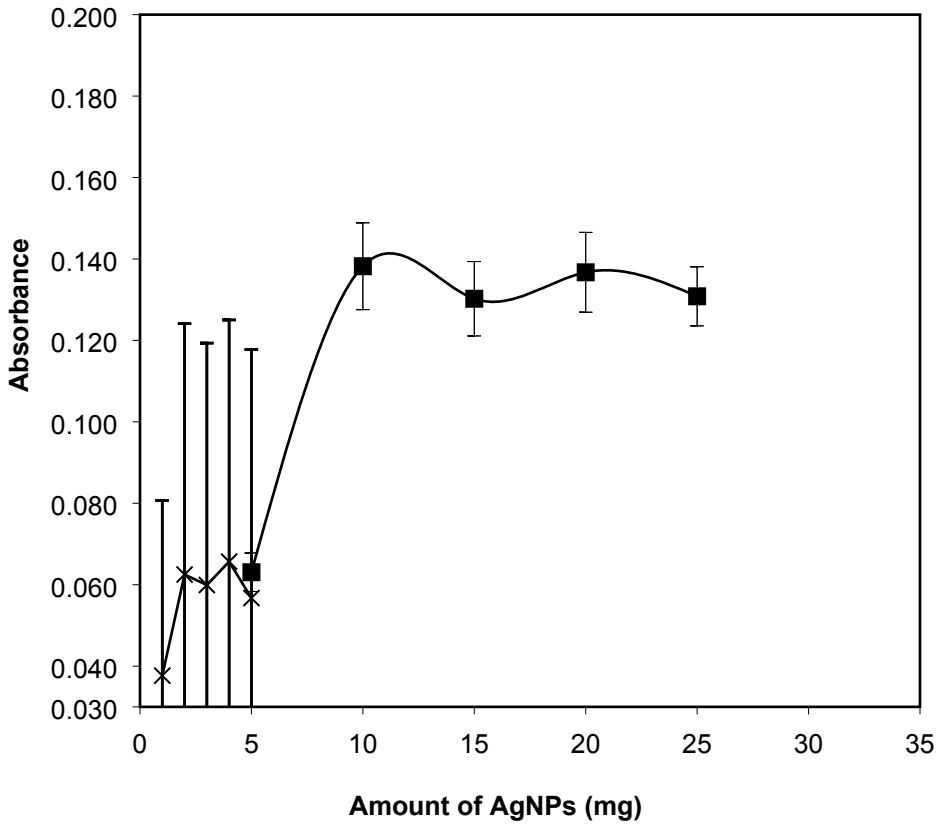

Fig. S2. Effect of amount of AgNPs on the determination of Hg^{2+} with the use of USA MDSPE procedure. Conditions: pH=3.5, sample volume 10 mL, sonication time 40 s, centrifugation 1 min, dissolution of AgNPs after extraction in 250 μL of 7 mol L^{-1} HNO_3. The error bar is the standard deviation (SD, n= 3).

3.1.2. Sample pH

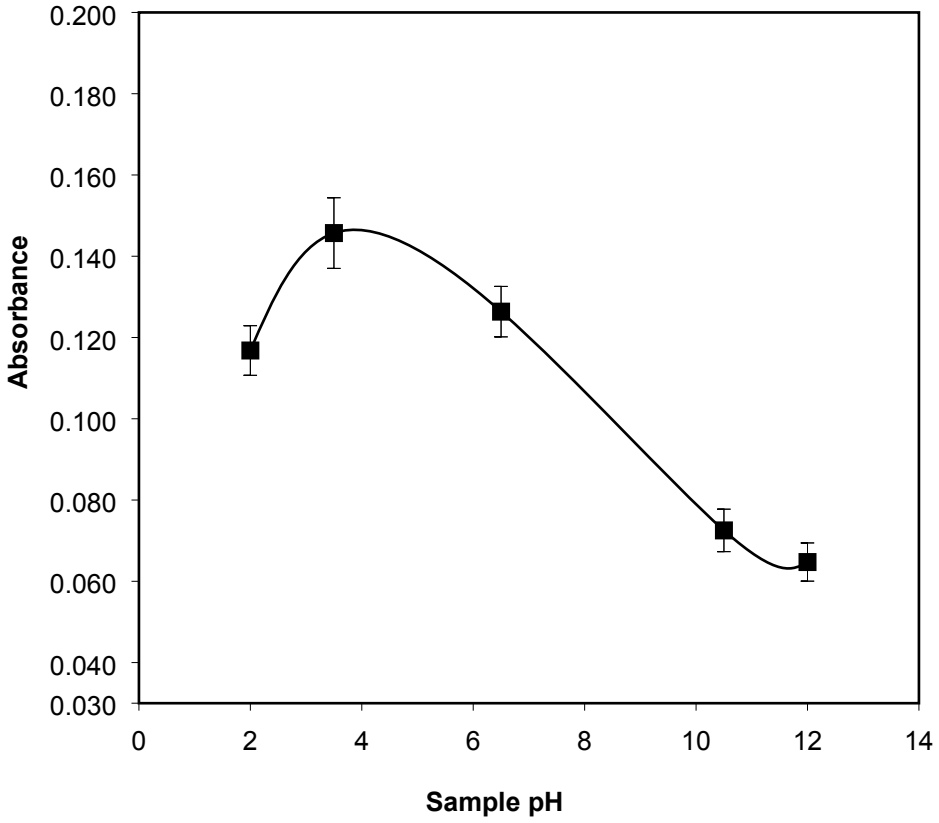

Fig. S3. Effect of sample pH on the determination of Hg^{2+} with the use of USA MDSPE procedure. Conditions: sample volume 10 mL, 10 mg of AgNPs, sonication time 30 s centrifugation 1 min, dissolution of AgNPs after extraction in 250 µL of 7 mol L^{-1} HNO_3. The error bar is the standard deviation (SD, n= 3).

3.1.3. Sonication and centrifugation time

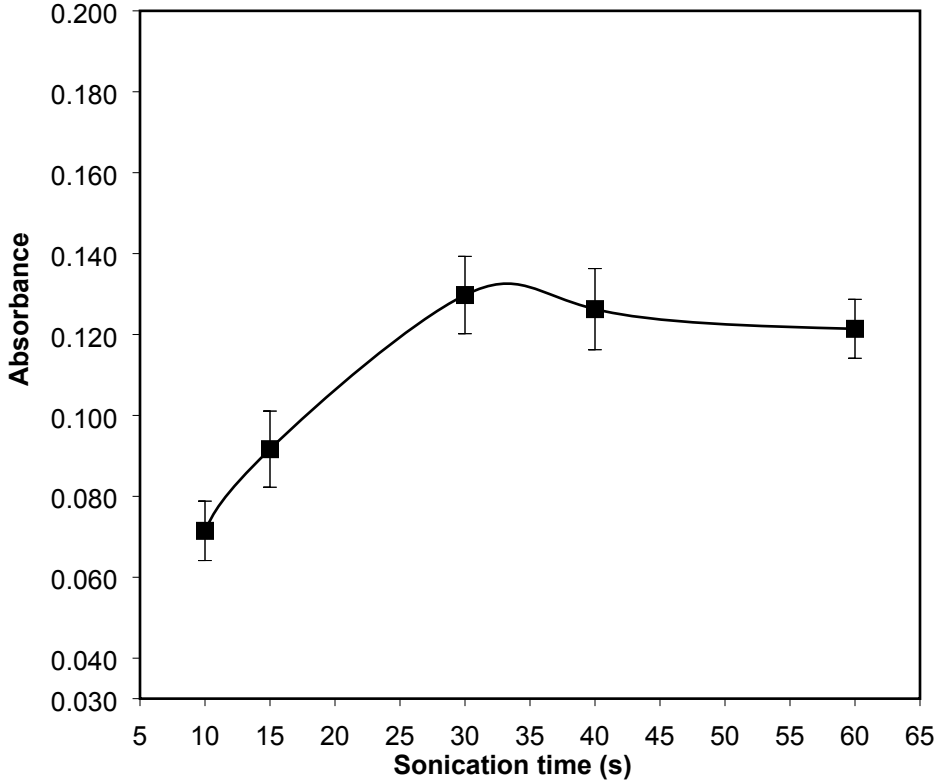

Fig. S4. Effect of sonication time on the determination of Hg^{2+} with the use of USA MDSPE procedure. Conditions: pH=3.5, sample volume 10 mL, 10 mg of AgNPs, centrifugation 1 min, dissolution of AgNPs after extraction in 250 μL of 7 mol L^{-1} HNO_3. The error bar is the standard deviation (SD, n= 3).

3.1.4. Dissolution of solid phase after extraction step

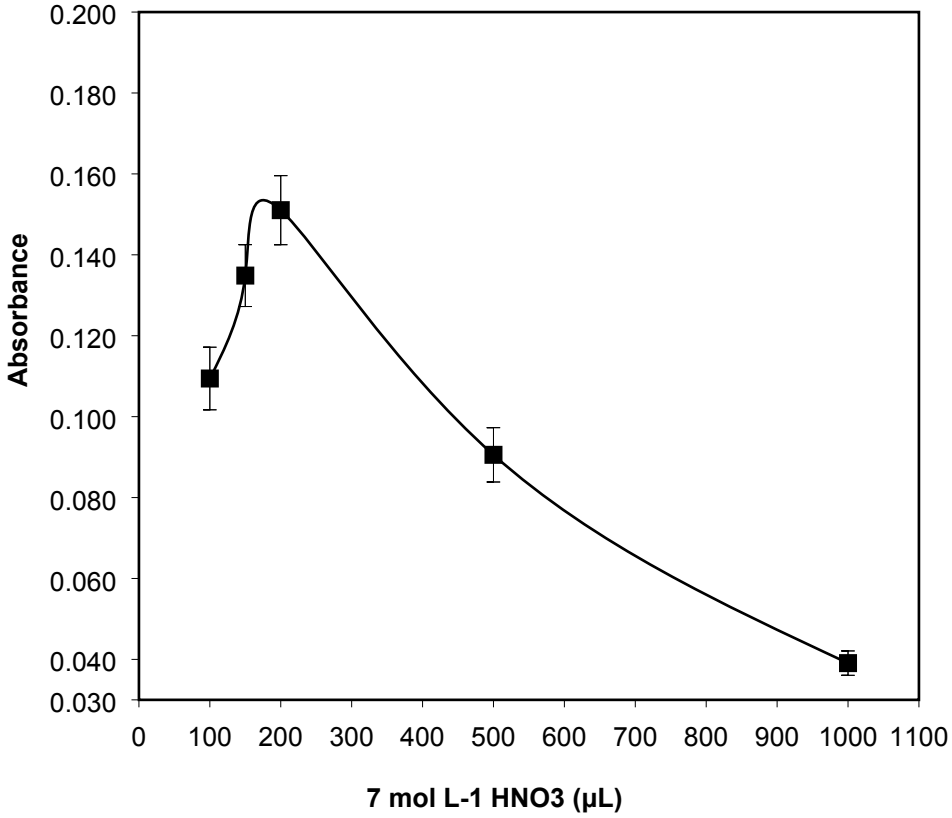

Fig. S5. Effect of volume of 7 mol L^{-1} HNO_3 (used as dissolving solution for solid phase after extraction) on the determination of Hg^{2+} with the use of USA MDSPE procedure. Conditions: pH=3.5, sample volume 10 mL, 10 mg of AgNPs, sonication time 30 s, centrifugation 1 min. The error bar is the standard deviation (SD, n= 3).