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Ultrasound assisted enzymatic hydrolysis for isolating titanium dioxide nanoparticles from bivalve mollusk before sp-ICP-MS

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Abstract

Applicability of single-particle inductively coupled plasma mass spectrometry (sp-ICP-MS) using dwell times equal to or shorter than 100 μs has been tested for assessing titanium dioxide nanoparticles (TiO_2 NPs) in bivalve mollusks. TiO_2 NPs isolation from fresh mollusk tissues was achieved by ultrasound assisted enzymatic hydrolysis procedure using a pancreatin/lipase mixture. Optimum extraction conditions imply ultrasonication (60% amplitude) for 10 min, and 7.5 mL of a solution containing 3.0 g $L^{-1}$of pancreatin and lipase (pH 7.4). The developed method was found to be repeatable (repeatability of 17% for the over-all procedure, TiO_2 NPs concentration of $5.33\times10^7 \pm 8.89\times10^6$, n=11), showing a limit of detection of 5.28×10^6 NPs g^{-1}**,** and a limit of detection in size of 24.4-30.4 nm, based on the 3σ criteria, and on the 3σ/5 σ criteria, respectively. The analytical recovery within the 90-99% range (use of TiO_2 NPs standards of 50 nm at 7 and 14 μg L^{-1} as Ti). Several bivalve mollusks (clams, cockles, mussels, razor clams, oysters and variegated scallops) were analyzed for total titanium (ICP-MS after microwave assisted acid digestion), and for TiO_2 NPs by the proposed method. TiO_2 NPs concentrations were within the 2.36×10^7-1.25×10^8 NPs g^{-1} range, and the most frequent

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