

## Supporting Information

### Detection and Characterization of SiO<sub>2</sub> and TiO<sub>2</sub> Nanostructures in Dietary Supplements

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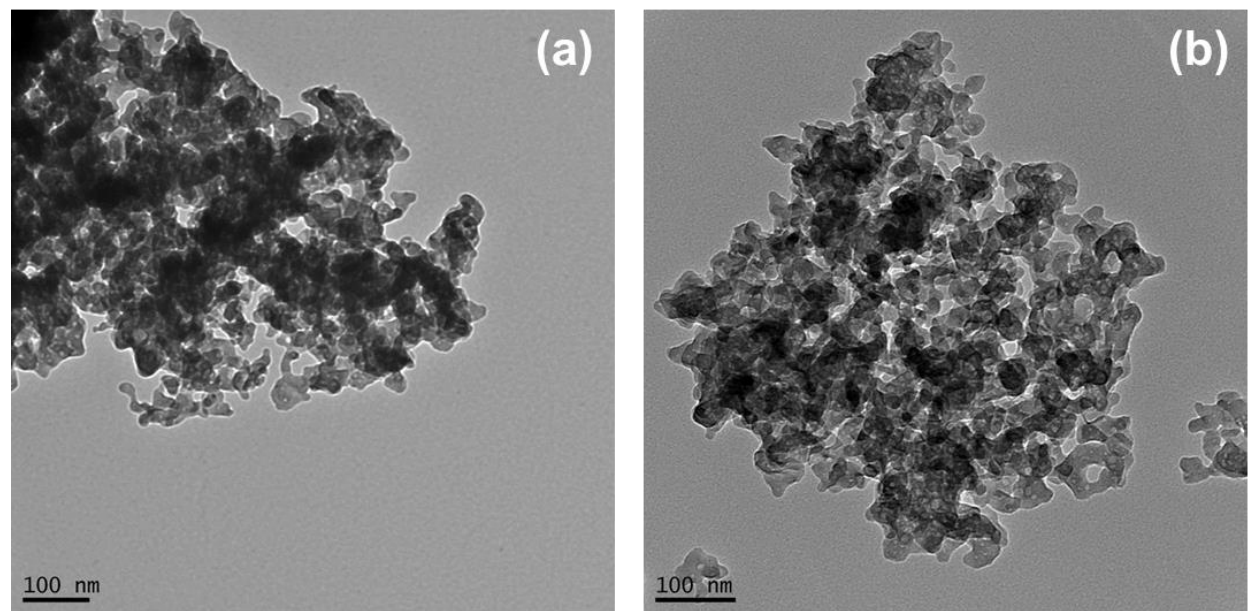
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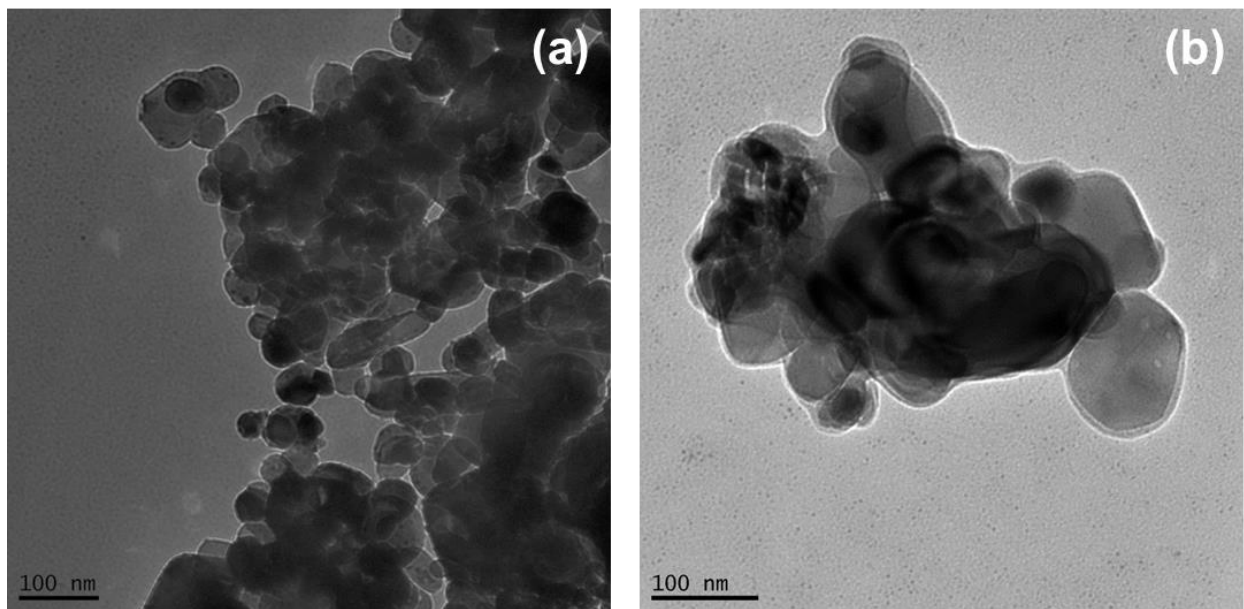
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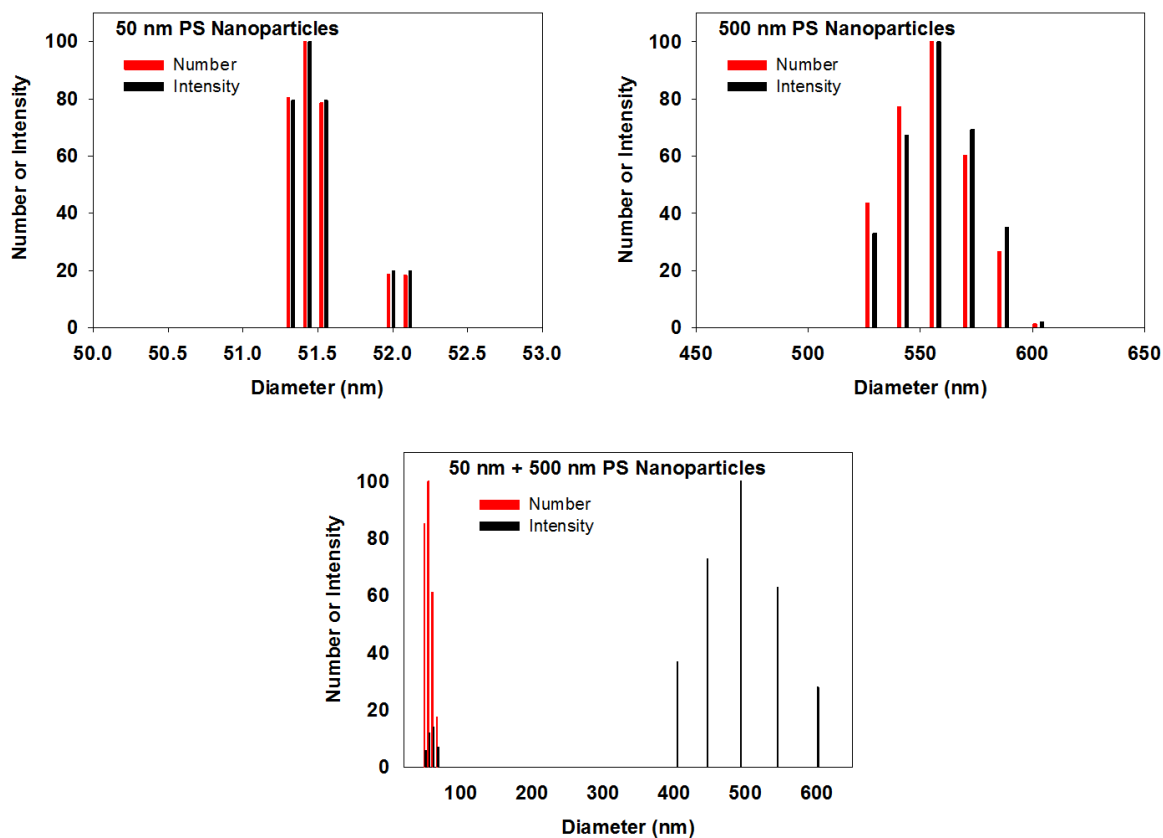
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**Figure SI1.** TEM images of commercial SiO<sub>2</sub> nanoparticles: (a) before digestion and (b) after digestion in a mixture of H<sub>2</sub>O<sub>2</sub> and HNO<sub>3</sub> at 120 °C.



**Figure SI2.** TEM images of commercial  $\text{TiO}_2$  nanoparticles: (a) before digestion and (b) after digestion in a mixture of  $\text{H}_2\text{O}_2$  and  $\text{HNO}_3$  at  $120\text{ }^\circ\text{C}$ .



	PS 50 nm	PS 500 nm	PS 50 nm + 500 nm
Number based particle size	51.5 nm	554.8 nm	55.5 nm
Intensity based particle size	51.5 nm	557.6 nm	442.0 nm
Smallest particle size	51.3 nm	528.0 nm	49.8 nm
Polydispersity	0.01	0.05	0.34

**Figure SI3.** DLS data of polystyrene (PS) standard nanoparticles with diameter of 50 nm, 500 nm, and mixture of 50 nm and 500 nm (1:1 volume ratio).

63 **Table SII.** Experimentally determined Si and Ti contents per capsule or tablet.

Sample	Si (mg/ per capsule or tablet)	Ti (mg/ per capsule or tablet)
DS-1	8.94	5.25
DS-2	12.1	2.95
DS-3	3.08	0
DS-4	0.266	0.228
DS-5	5.02	0.606
DS-6	2.40	0
DS-7	1.60	0
DS-8	0	0.468
DS-9	4.51	0
DS-10	3.26	1.55
DS-11	2.93	0
DS-12	12.6	5.63

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65 The concentration of Si and Ti of each product was experimentally determined by ICP-MS. Nitric acid

66 (HNO<sub>3</sub>, Optima 67-70%) and hydrofluoric acid (HF, Optima 47-51%) were purchased from Fisher

67 Scientific (Houston, TX, USA). 10 ppm silicon (Si), titanium (Ti), and scandium (Sc) standard solutions

68 were purchased from Inorganic Venture (Christiansburg, VA, USA), and used to prepare standard

69 calibration solutions for ICP-MS measurements, where Sc was used as an internal standard. Si and Ti

70 standard solutions (1000 ppm) used to prepare an initial calibration verification (ICV) sample were

71 acquired from Ultra Scientific (N. Kingstown, RI, USA) and Spex CertiPre group (Metuchen, NG, USA),

72 respectively. To determine a total Si and Ti concentration, dietary supplements were first digested using a

73 CEM (Matthews, NC, USA) MARS-Xpress microwave digester (maximum power: 1600 W). The tablets

74 or capsules for each sample were suspended in 20 mL of 2.5% HNO<sub>3</sub> solution and allowed to dissolve

overnight. 100  $\mu$ L of suspended material, 2 mL  $\text{HNO}_3$  and 200  $\mu$ L HF were transferred into a digestion vessel. Samples were then digested at 180°C for 30 minutes. After microwave digestion, the sample solutions were transferred into 15 mL tubes and each digestion tube was rinsed in triplicate with ultra-pure water. The final volume of each sample was 10 mL including the internal standard (Sc, 100 ppb). The samples were analyzed using an Agilent (Santa Clara, CA, USA) 7700X ICP-MS with a PFA inert kit and nebulizer. The concentrations of Si (mass: 28) and Ti (mass: 47) were analyzed in triplicate using  $\text{H}_2$  and He gas modes, respectively. Reagent blanks containing 2 mL  $\text{HNO}_3$  and 200  $\mu$ L HF were prepared simultaneously and analyzed along with the sample. No contamination was found in the reagent blank. To validate the digestion and ICP-MS methodologies, a series of recovery experiments were performed using commercially purchased  $\text{SiO}_2$  and  $\text{TiO}_2$  nanoparticles. For this study, the  $\text{SiO}_2$  and  $\text{TiO}_2$  (0.5-1 mg) standards were digested using the previously defined experimental conditions. The final recovery rates of both Si and Ti were in excess of 90% ( $n = 3$ ). (*Caution: all concentrated acids should be handled in fume hood. Microwave acid digestion procedures are followed the manufacturer safety recommendations.*) Based on the product labeling, unexpected Si and Ti compounds are included in DS-1, DS-2, DS-4, and DS-5. As we know, commercial products contain many other ingredients. It was not clearly determined where the Si came from. However, in our study,  $\text{SiO}_2$  nanoparticles in DS-1 and DS-5 were not found.