

# Chemical Engineering Doctoral Defense

## Tiered Approach to Detect Nanomaterials in Food and Environmental Matrices

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### abstract

Nanomaterials (NMs), implemented into a plethora of consumer products, are a new class of pollutants with unknown hazards to the environment. Exposure assessment is necessary for hazard assessment, life cycle analysis, and environmental monitoring. Current nanomaterial detection techniques on complex matrices (heterogeneous samples with low weight content of nanomaterials) are expensive and time intensive, requiring weeks of sample preparation and detection by specialized equipment, limiting the feasibility of large-scale monitoring of NMs. Because of the tedious lead times, a need exists to develop a rapid pre-screening technique to detect, within minutes, nanomaterials in complex matrices.

The goal of this dissertation is to develop a tiered process to detect and characterize nanomaterials that uses a rapid detection technique to first pre-screen samples. The approach is accomplished through a two tier rapid screening process followed by a three tier characterization process. A sample passes each of the two rapid prescreening tiers if an element (e.g. Si, Ti) suspected to be present as a nanomaterial (e.g. SiO<sub>2</sub> and TiO<sub>2</sub>) is identified. If the element is absent, the sample is deemed free of nanomaterials and the process is stopped at that tier, eliminating the need to use specialized characterization equipment on that sample.

The five step tiered process as follows: 1) screen for elements in the sample by laser induced breakdown spectroscopy (LIBS) and X-ray fluorescence (XRF), 2) extract nanomaterials from the sample and screen for extracted elements by LIBS and XRF, 3) confirm presence and elemental composition of nanomaterials by transmission electron microscopy paired with energy dispersive X-ray spectroscopy, 4) quantify the elemental composition of the sample by inductively coupled plasma – mass spectrometry, and 5) identify mineral phase of crystalline material by X-ray diffraction.

The tiered approach focuses on nanomaterials (e.g. TiO<sub>2</sub>, SiO<sub>2</sub>, and calcium phosphate) dispersed in food products, products that have direct human contact and are a major source of NM release into the environment. Development of tier one and two to prescreen and eliminate products free of nanomaterials results in time and cost savings compared to other conventional techniques (e.g. TEM).