



Consumer use effects on nanoparticle release from commercially available ceramic cookware

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ABSTRACT

Food preparation and storage materials are often subjected to stressful conditions such as scraping and cleaning with abrasive pads throughout their lifecycle. In general, understanding potential nanoparticle migration when in contact with food is important in assessing their safety. A ceramic-coated fry pan and a ceramic sauce pot (both commercially available) were evaluated for nanoparticle migration under three consumer use conditions. Washing, scouring, and scratching conditions were simulated by linear abrasion using scrubbing pads, steel wool and tungsten carbide burr attachments, respectively. Migration of titanium (Ti) and silicon (Si) was evaluated using 3% acetic acid as a food simulant. Ti and Si concentrations in simulant were generally higher under the consumer use scenarios than in fresh/unused pans. Titanium dioxide (TiO₂) and silicon dioxide (SiO₂) nanoparticles were detected in the simulant under the most aggressive use scenario simulated by abrasion with the tungsten carbide burr attachment. TiO₂ and SiO₂ particle number concentrations were on the order of 10⁸ and 10⁷ particles dm⁻², with median diameters of 250 nm and 460 nm, respectively. The aluminum (Al) concentration migrating from the sauce pot was also higher under the consumer use scenarios than in fresh/unused pots, but without any detectable nanoparticle migration.

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1. Introduction

The use of nanomaterials in consumer products has grown rapidly in recent years because of their potential to provide improvements in mechanical, chemical, and thermal properties (Addo Ntim & Noonan, 2017; Chaudhry et al., 2008; Duncan, 2011; Mihindukulasuriya & Lim, 2014). There is increased interest in the commercial application of nanotechnology in food-related products, especially in products which incorporate nanomaterials into food contact materials (FCMs) used for cookware, food preparation tools, food packaging materials, and food storage products (Hatzigrigoriou & Pappaspyrides, 2011; Lagarón & Busolo, 2012; Mihindukulasuriya & Lim, 2014). Worldwide, commercial

applications primarily involve the use of nanoparticles incorporated into polymer matrices to form nanocomposites (Lagarón & Busolo, 2012; Lagarón et al., 2005; López-Carballo, Gómez-Estaca, Catalá, Hernández-Muñoz, & Gavaara, 2012; Paul & Robeson, 2008; Peelman et al., 2013; Plackett & Siró, 2012).

A relatively newer class of nano-enabled products have emerged that incorporate ceramics in whole products or as coatings for nonstick cooking surfaces. These products are mainly fry pans and sauce pots for stove top and oven cooking. Some of the ceramic materials used in making this class of consumer products include silica nanoparticles (SiO₂), titanium dioxide nanoparticles (TiO₂), and nanoclay. These nano-enabled nonstick cookware have been marketed to consumers as an alternative to polytetrafluoroethylene (PTFE)-coated nonstick products. Although there has been considerable focus on evaluating nanoparticle migration from food-related polymer nanocomposites (PNCs) (Addo Ntim, Thomas, Begley, & Noonan, 2015; Bott, Störmer & Franz, 2014a,b; Echegoyen & Nerín, 2013; von Goetz et al., 2013), there are very

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little to no data in the literature regarding potential migration from nanoceramic cookware.

The US Food and Drug Administration (US FDA) and the Consumer Product Safety Commission (CPSC) are interested in developing integrated knowledge about the current applications of nanotechnology in consumer products that come in contact with food, to better monitor these products, and to further understand and predict their safe use. The mechanism, forms and amounts of nanocomponents migrating from these consumer products are poorly understood and cannot be predicted based on the product type and physicochemical properties of the FCMs and the nanomaterials incorporated. There is, therefore, a need for method development and data generation to characterize these FCMs and the potential for nanomaterials to migrate from them under intended use conditions.

This project characterized commercially available ceramic and ceramic-coated cookware and investigated the migration of nanoparticles into food simulants under both normal and stressful use conditions. Nanoparticle migration was evaluated from the fresh/unused FCMs and after being subjected to linear abrasion using attachments such as a scrubbing pad, steel wool and a tungsten carbide burr to simulate washing, scouring, and scratching, respectively. Migration simulants were analyzed by single-particle inductively coupled plasma mass spectrometry (*sp*-ICP-MS) and electron microscopy for the presence of nanomaterials.

2. Materials and methods

2.1. Materials

Glacial acetic acid (Optima grade), nitric acid (HNO₃, Optima™), hydrofluoric acid (HF, Optima™), hydrochloric acid (HCl, Optima™) were purchased from Fisher scientific (Pittsburgh PA, USA). Water (18 MΩ.cm) was obtained from an Aqua Solutions (Jasper, GA) water purification system. The sauce pot was purchased online and the nonstick fry pan was purchased at a retail store in the United States. The products were selected based on advertising claims. The fry pan and the sauce pot were initially advertised as being coated with nanomaterials, both claims have since been removed from the manufacturers website likely based on the assertion that although nanosized particles may be precursors for the coatings, the normal sintering process causes the particles to fuse together and the coating that results is a micrometer thick layer, and not simply nanoparticles loosely bound to a bulk matrix. Our interest was therefore in the potential impact of use on the integrity of the coating and the potential for nanoparticle release.

2.2. Product characterization

The FCMs were characterized prior to use and migration evaluation. High-resolution imaging and elemental analysis of the FCMs was performed on an FEI Helios NanoLab 660 focused ion beam scanning electron microscope (FIB SEM) (Hillsboro, OR, USA) equipped with an EDAX Octane Plus energy dispersive x-ray spectrometer (EDS) and TEAM analysis software (Mahwah, NJ, USA).

Total Ti and Si in the fry pan coating and Al concentration in the sauce pot were determined using microwave assisted acid digestion and inductively coupled plasma-mass spectrometry (ICP-MS) (Weir, Westerhoff, Fabricius, Hristovski, & von Goetz, 2012). The fry pan coating was released by dissolving the aluminum base with concentrated hydrochloric acid (HCl). Weighed amounts of material from the fry pan coating and the sauce pot (5 mg–60 mg) were placed into perfluoroalkoxy alkane (PFA) vessels and filled with 2 mL–4 mL of nitric acid (Fisher Optima), 1 mL–2 mL hydrofluoric acid (Fisher Optima), and 0.5 mL of hydrochloric acid and digested

in a high pressure microwave reactor (Milestone Ultraclave, Shelton, CT, USA). The reaction temperature was ramped from room temperature to 200 °C in 30 min and held at 200 °C for an additional 60 min. After cooling to room temperature, 3 mL of 4% boric acid was added to the vessels and digested a second time. The temperature profile for the second step of digestion involved ramping from room temperature to 200 °C in 15 min and holding at 200 °C for 30 min. Method blanks, matrix spikes, and certified reference materials were included in each batch of digestion for performance verification. The digestates were diluted with 50 mL 18 ΩM.cm water. Al concentration was determined by an Agilent 7700 ICP-MS (Agilent technologies, Santa Clara, CA, USA) in helium mode. The ICP-MS sample introduction system was replaced with an HF resistant PFA system prior to analysis. Ti and Si concentrations were determined by an Agilent 8800 QQQ ICP-MS (Agilent technologies, Santa Clara, CA, USA) in hydrogen mode. Q1/Q2 masses of the determined isotopes for Si and Ti were 28/28 and 48/48, respectively.

2.3. Simulating consumer use scenarios

Ceramic FCMs are used under harsh conditions of high temperature stove top frying and oven baking, among others. Cooking utensils also have the potential to cause some damage to the FCMs. Additionally, the FCMs may be subjected to stringent cleaning to remove baked on food. To simulate the different mechanical wear scenarios normally experienced by cookware, the FCMs were subjected to abrasion using the Taber linear abrader (Taber Industries Model 5750, North Tonawanda, NY) (Fig. 1a and b). Three different abrading attachments (Fig. 1c) were used to simulate three separate mechanical wear scenarios. A piece of scrubbing pad (3 M Final Stripping Pad) wrapped around a rubbing finger was used to simulate washing conditions. A collet stuffed with steel wool (Rhodes American Medium Steel Wool) was used to simulate scouring of cooking surfaces. A tungsten carbide burr (Grobet end-cut carbide burr) attachment was used to simulate scratching of the surfaces using metal utensils.

The samples were secured on the linear abrader sample stage with clamps (Fig. 1b) to prevent movement and vibration during the abrasion process. The number of abrasion cycles and the load applied were designed to produce sufficient and noticeable wear of the sample surface and, therefore, varied depending on the abrasive attachment and the sample type (Table 1). To increase the abraded surface area, abrasion runs at several different locations were performed on each sample. For the purpose of this study, an abrasion run was defined as a predetermined number of cycles of linear abrasive motion. In addition to the intact pots and pans undergoing abrasion for migration experiments, for SEM analysis 1 in x 1 in pieces were cut from the FCMs and their surface was abraded.

High-resolution imaging and elemental analysis of the abraded sample surface were performed on an FEI Helios NanoLab 660 focused ion beam scanning electron microscope (FIB SEM) (Hillsboro, OR) equipped with an EDAX Octane Plus energy dispersive x-ray spectrometer (EDS) and TEAM analysis software (Mahwah, NJ). Loose particles and dust from the abraded surface of each sample were collected using a double-sided carbon tab mounted on an SEM pin stub and analyzed in the SEM without additional coating. The remaining surface debris on the abraded cut sample was removed by wiping with a Texwipe (Kernersville, NC) and isopropyl alcohol. Each sample was coated with roughly 10 nm of amorphous carbon using a Cressington 208 carbon coater prior to SEM analysis to reduce charging artifacts. SEM imaging and EDS analysis were performed at various beam energies ranging from 2 keV to 15 keV and beam currents ranging from 100 pA to 800 pA. Both secondary electron images and back scattered electron images were collected.

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