Microwave Digestion of Mixed Powder Samples Using a Reconstitution Step to Increase Sample Homogeneity

Abstract

Microwave digestion of powdered materials, such as infant formula and dairy powders, have proven to be a difficult task. Powders are inherently non-homogenous and digesting a sample large enough to provide a homogeneous representative sample often leads to excessive venting and incomplete digestion.

Reconstituting the powdered samples in water greatly improves their homogeneity and yields more consistent and complete digestions. This application note will describe the procedure of reconstitution and digestion of four food powder samples using the MARS 6 microwave digestion system.

Introduction

An estimated 1 million infants in the United States are fed formula from birth and, by the time they are three months old, about 2.7 million rely on formula for at least part of their nutrition. Accurate nutritional labeling of food products is a legal requirement in most countries to help consumers make informed healthy choices. This means all label claims need to be supported by accurate scientific measurements. For vulnerable groups such as babies and people requiring clinical nutrition products, infant formula nutrient analyses need to be very specific and able to accurately quantify even very low concentrations of heavy metals such as Pb, As, and Cd. In order to detect the low levels of heavy metal impurities obtaining a large representative sample is critical.

This application note will focus on the preparation of powdered samples including infant formula, cheese powder, milk powder, and powdered butter. Samples will be reconstituted in warm water and an aliquot of each powder slurry will be prepared in the MARS 6 digestion system using the 75 mL MARSXpress vessel.

Instrumentation

A total of forty samples were prepared using a CEM Corporation MARS 6 microwave digestion system equipped with iWave technology. iWave is a novel technology advancement that utilizes Light Emitting Technology™ to accurately measure the temperature of the actual sample solution inside the vessel and does not require an internal probe.

Samples were prepared in CEM 75 mL MARSXpress vessels. This easy to use three-part vessel allows up to 40 samples to be prepared simultaneously and are sealed with a simple hand held torque device. Although the 75 mL MARSXpress vessels were used for the acid digestion for this study, the MARSXpress Plus vessel could be used as an alternative.

Procedure and Method

Reconstitution Step

In a 250 mL beaker with stir bar, 85 g of ASTM Type II or better water was added and heated to 60°C on a hot plate. A 15 g powder sample was added and allowed to stir and heat for 10 minutes until all powder was well incorporated into the slurry. A thermometer was used to confirm the temperature of 60°C +/- 5°C. A 7 g aliquot was weighed into a disposable pipette or syringe. Based upon a 15% (w/w) the slurry will yield a final sample size of about 1.05 g of powder. **Figure 1** shows what the reconstituted powder samples look like prior to analysis.

7 g x 15% (w/w) = 1.05 g

Figure 1: Reconstituted samples on hot plate

Digestion Step

A 7 g aliquot of reconstituted sample was added to each MARSXpress liner along with 10 mL of concentrated HNO_3. 9 vessels for each sample were prepared with the reconstituted sample. Eight of them were spiked with 1 mL of a 10 ppm spike containing Pb, As, and Cd. One vessel was an acid blank. The vessels were capped and placed in the MARS 6 for digestion. The One Touch Method Food was used. The One Touch Technology automatically recognized the vessel type and counted the number of vessels. It then chose the optimized conditions for the acid digestion. The maximum temperature achieved using this method is 200°C. Food samples in general do not require extremely high temperatures to achieve complete digestion.

Table 1

Samples	Slurry Weight	Dry Sample Weight
Powdered Infant Formula	7 g	1.05 g
Non Fat Dry Milk Powder	7 g	1.05 g
Butter Powder	7 g	1.05 g
Parmesan Cheese Powder	7 g	1.05 g

Results and Discussion

The reconstitution step allows for a larger sampling of the powdered material yielding a more homogenous sample. The MARS 6 with 75 mL MARSXpress vessels and iWave Temperature Control simultaneously digested the four different powder samples in a single batch offering a sufficient digestion solution for high throughput food testing labs. For each sample one blank, one unspiked sample, and 8 samples spiked with Cd, Pb, and As were digested in a single run for a total of 40 samples. **Figure 2** illustrates the system automatically adjusts the power to compensate for the varied sample types. **Figure 3** shows the variability in sample temperature during the ramp due to the different sample compositions. During the hold phase all vessel temperatures normalize as all bonds have now been broken and the samples completely digested as seen in **Figure 4**. The before and after samples shown in **Figure 5** demonstrate clear and particulate free digestions.

Figure 2: iWave provides precise power control during run

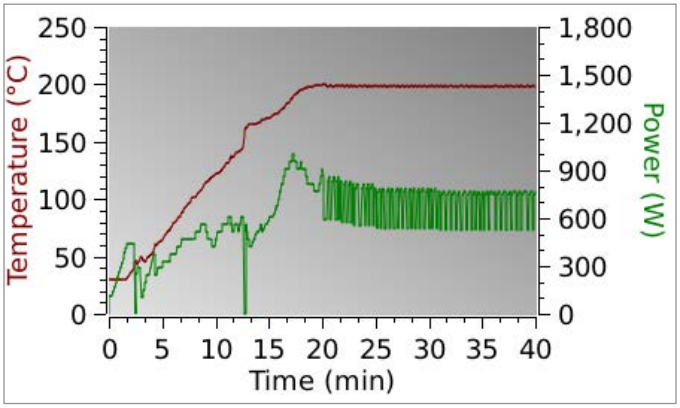

Figure 3: Bar graph showing vessel temperatures during ramp

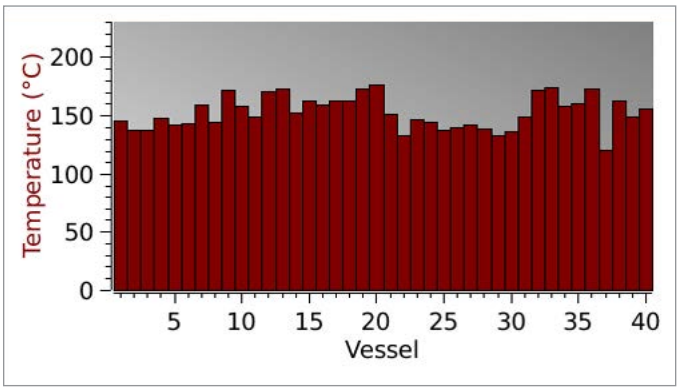

Figure 4: Bar graph at end of run showing consistent temperature across all vessels

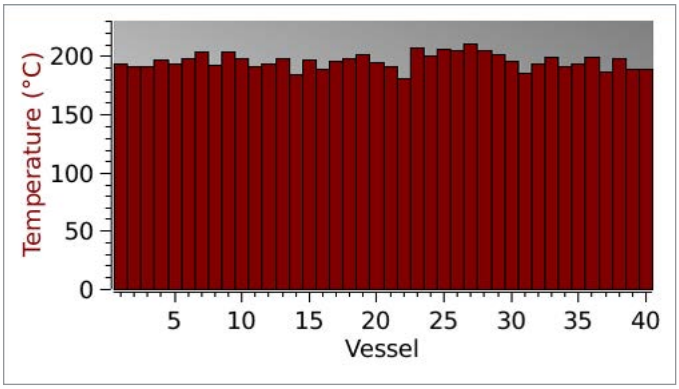

Figure 5: Samples before and after digestion

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