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Technical note: The equivalency of sodium results in cheese digested by either dry ashing or microwave-accelerated digestion

T. C. Schoenfuss,*[1] Z. P. Metz,† A. E. Pataky,* and H. L. Schoenfuss‡
*Department of Food Science and Nutrition, University of Minnesota, Saint Paul 55108
†Chemical Engineering and Materials Science, University of Minnesota, Minneapolis 55455
‡Aquatic Toxicology Laboratory, St. Cloud State University, St. Cloud, MN 56301

ABSTRACT

Analysis of dairy products for minerals such as sodium requires mineralization of the sample, which is typically done by either dry ashing or atmospheric wet ashing; both methods are time consuming and wet ashing requires the repeated handling of hot acid. A rapid method using microwave-accelerated acid digestion before atomic absorption spectrometry to measure sodium was compared with dry ashing in 138 samples of blue cheese (in duplicate) that varied in sodium content and age. Linear regression of the results obtained within different cheese salting treatments and sampling locations over time showed that the methods were equivalent in terms of linearity and the slope of the line. A consistent bias was observed, with lower sodium concentrations being quantified during atomic absorption spectrometry for the microwave-digested samples. Evaluation of this difference by the 2 one-sided test (TOST) procedure showed that the confidence intervals of the percentage difference between the methods fell within the predetermined acceptable percentage difference. We conclude that this rapid microwave digestion procedure of blue cheese yielded equivalent results to dry ashing.
Key words: atomic absorption, sodium in cheese, microwave-accelerated reaction, digestion

Technical Note

Mineral analysis of food products can be a lengthy and time-consuming process. Organic compounds must first be destroyed by wet or dry ashing before analysis by a method such as atomic absorption spectrometry (**AAS**) or inductively coupled plasma-optical emission spectrometry (**ICP-OES**). For measurement of minerals such as sodium in cheese, wet and dry ashing are used (ISO-IDF, 2007). With wet ashing at atmospheric pressure, the sample is boiled in concentrated nitric acid in a flask with a watch glass cover, for 2 h. Dry ashing involves acid soaking of crucibles to remove residual minerals, lighting the cheese weighed into the crucible on fire to carbonize it, followed by ashing in a muffle furnace at 550°C for at least 6 h. As a result, the sample preparation steps for both of these methods consume at least 1 d before analysis by AAS or ICP-OES. These methods also expose the operator to strong acids and high temperatures, thus invoking safety concerns. Microwave-accelerated wet digestion (**MAD**) is a technique where sample and acid are placed in a polytetrafluoroethylene (PTFE) reaction vessel and heated to digest the sample. This is a much faster and self-contained means of digestion and could greatly improve the speed for analyzing products (such as cheese for sodium), reduce the use of concentrated acid, and reduce exposure to hazards for the operator. The International Dairy Federation/International Organization for Standardization method IDF119:2007/ISO 8070:2007 (IDF-ISO, 2007) describes mineral quantification by AAS in dairy products with several digestion options, one being microwave digestion. Pressurized MAD is described in general in this method, but is unspecific for conditions required except in specifying microwave ashing at 150°C for at least 3 h. An AOAC International official method exists for microwave digestion of fortified food products followed by ICP-OES, but cheese was not included and hydrochloric acid is used instead of nitric acid (AOAC International, 2011). One collaborative interlaboratory study included MAD of cheese before sodium analysis by AAS, and microwave digestion was determined to produce accurate results (Julshamn et al., 2005). Multiple microwave oven types were used in the aforementioned study, and digestion temperature, wattage, sample size, and acid amount were not controlled. One cheese was sampled by all laboratories evaluating the method, so variations in sodium concentration or matrix were not evaluated nor were comparisons made to a standard sample preparation procedure.

A microwave-accelerated reaction system produced by CEM Corp. (Matthews, NC) has the ability to monitor the temperature of each vessel (24 per carousel) as they

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[1]Corresponding author: tschoenf@umn.edu

rotate in a carousel and adjust the power to maintain a constant average temperature for all vessels throughout the hold time. We compared this system of MAD to standard dry ashing in 138 samples of blue cheese, in duplicate, with sodium concentrations ranging from approximately 780 to 1,300 mg/100 g of cheese. The effect of variation in sodium concentration and cheese chemistry due to cheese aging on the equivalency of the methods was evaluated.

Surface-salted blue cheese wheels (weighing approximate 3 kg each) were sampled at 2 locations per wheel at monthly intervals during aging to monitor sodium migration. Cheeses were manufactured as part of a sodium reduction study and the treatments were (1) control cheese, (2) 25% reduced sodium, and (3) 25% reduced sodium with KCl. The gross composition at initial sampling is shown in Table 1. The amount of NaCl (g) applied per wheel was calculated as 0.035 ×

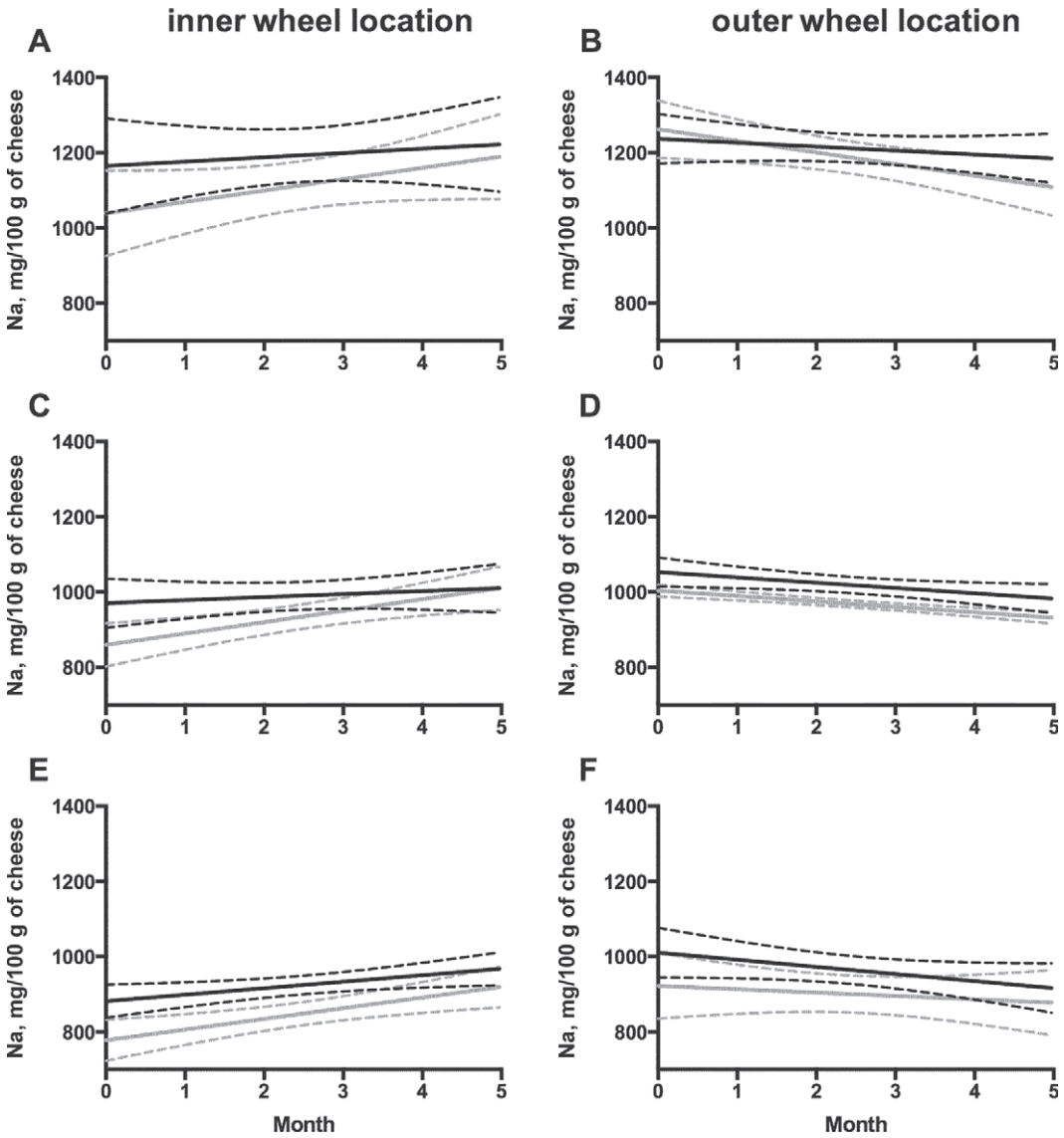

Figure 1. Sodium measured over 5 mo of ripening for blue cheese samples digested by either dry ashing (black solid lines) or microwave digestion (gray solid lines); dashed lines indicate 95% confidence intervals. Panels A, C, and E are measurements obtained from sampling the inner portion of the wheel of blue cheese for control, reduced sodium, and reduced sodium with KCl, respectively. Inner location was cut from the core of the wheel, >5.1 cm from the surface, >1.26 cm from the top. Panels B, D, and F are for cheese samples from the respective treatments sampled at an outer wheel location. Outer location sampled from between 0.63 and 4.4 cm from the surface of the wheel. Treatments consisted of different salting applications. The amount of NaCl (g) applied per wheel was calculated as 0.035 × unsalted wheel weight (g) for the control treatment, and as 0.0245 × unsalted wheel weight (g) for both reduced sodium treatments (KCl was added with the NaCl to the treatment with KCl at 0.0134 × unsalted wheel weight, g).