



Identification of goat milk powder by manufacturer using multiple chemical parameters

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ABSTRACT

Concentrations of multiple elements and ratios of stable isotopes of carbon and nitrogen were measured and combined to create a chemical fingerprint of production batches of goat whole milk powder (WMP) produced by different manufacturers. Our objectives were to determine whether or not differences exist in the chemical fingerprint among samples of goat WMP produced at different sites, and assess temporal changes in the chemical fingerprint in product manufactured at one site. In total, 58 samples of goat WMP were analyzed by inductively coupled plasma-mass spectrometry as well as isotope ratio mass spectrometry and a suite of 13 elements (Li, Na, Mg, K, Ca, Mn, Cu, Zn, Rb, Sr, Mo, Cs, and Ba), $\delta^{13}\text{C}$, and $\delta^{15}\text{N}$ selected to create the chemical fingerprint. Differences in the chemical fingerprint of samples between sites and over time were assessed using principal components analysis and canonical analysis of principal coordinates. Differences in the chemical fingerprints of samples between production sites provided a classification success rate (leave-one-out classification) of 98.1%, providing a basis for using the approach to test the authenticity of product manufactured at a site. Within one site, the chemical fingerprint of samples produced at the beginning of the production season differed from those produced in the middle and late season, driven predominantly by lower concentrations of Na, Mg, K, Mn, and Rb, and higher concentrations of Ba and Cu. This observed temporal variability highlights the importance of obtaining samples from throughout the season to ensure a representative chemical fingerprint is obtained for goat WMP from a single manufacturing site. The reconstitution and spray drying of samples from one manufacturer by the other manufacturer enabled the relative influence of the manufacturing process on the chemical fingerprint to be examined. It was found that such reprocessing

altered the chemical fingerprint, although the degree of alteration varied among samples and individual elements. The findings of this study support the use of trace elements and stable isotope ratios to test the authenticity of goat WMP, which can likely be applied to other dairy goat products. This approach could be used to test to the factory of origin (and potentially batch of origin) of products in the supply chain, thus providing the ability to audit the supply chain and monitor for fraudulent activity.

Key words: dairy goat, whole milk powder, origin, trace elements, stable isotope ratios

INTRODUCTION

A series of recent food safety scares and quality issues has raised the level of consumer awareness (Moore et al., 2012) and placed added pressure on food producers and retailers to demonstrate that they take such issues seriously. Provenance is often valued when the food product originates from a source that has high standards of food safety, quality assurance, and quality control (Kelly et al., 2005; Carcea et al., 2009).

Chemical profiling techniques have been used to verify the origin (a geographic origin or processing plant) and authenticity of food products (Rossmann, 2001; Kelly et al., 2005; Förstel, 2007). To be effective, such a profile needs to differ on a geographic scale or among manufacturers and be difficult to replicate. Rapid verification of the authenticity of a suspect sample can be assisted by comparison to a database containing profiles of historical samples from the purported origin (Le Bot et al., 2011). Of these profiling techniques, quantification of trace element concentrations and stable isotope ratios have proven to be the most useful and scientifically robust over a wide range of food products (Luykx and van Ruth, 2008). Several review articles have been published covering the application of these techniques in sectors including dairy, meat, honey, beverages, and plant products (Rossmann, 2001; Kelly et al., 2005; Förstel, 2007; Carcea et al., 2009; Ye, 2012).

Less focus has been concentrated on applying this approach for complex processed or manufactured prod-

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Table 1. Summary of samples of goat whole milk powder included in this study

Country of origin	Year received	Year manufactured	Number of samples
the Netherlands	2011	2011	14 ^{1,2}
New Zealand	2011	2011	10
Spain	2011	2011	1
the Netherlands	2012	2011	3 ¹
New Zealand	2012	2012	13
New Zealand	2014	2012	1
New Zealand	2014	2013	6
New Zealand	2014	2014	6
France	2014	2012	1
the Netherlands	2014	2014	3
Total			58
Total New Zealand			36
Total the Netherlands			20 ¹
Total Spain			1
Total France			1

¹A total of 4 samples from 2011 and 3 samples from 2012 were reconstituted and spray dried in New Zealand.

²These samples were manufactured each month from July to December.

ucts (Scampicchio et al., 2012), where the addition of multiple ingredients and physical contact with the processing facility is likely to contribute trace metals, thus further influencing the chemical fingerprint of a finished product. This leads to the question of whether processed products coming out of different processing plants maintain distinctive fingerprints that could be used as the basis for testing product authenticity.

The current study examined differences in the stable isotope ratios and trace element concentrations—the chemical fingerprint—between goat whole milk powders (WMP) produced by a factory in New Zealand with those produced by a factory in the Netherlands. The hypothesis was that a distinct fingerprint could be determined for product produced by each factory that enables the manufacturer of origin to be investigated. Our objectives were (1) to investigate differences in the trace elemental and light isotope composition of samples of goat WMP among samples from multiple global sources; (2) to investigate temporal changes in the trace elemental and light isotope composition of samples of goat WMP produced by a factory in northern New Zealand; and (3) to examine the relative influence of the manufacturing process on the fingerprint of product.

MATERIALS AND METHODS

Sample Collection and Preparation

Samples of WMP processed by the New Zealand factory were collected from approximately the middle of individual manufacturing runs. Samples of WMP were sourced from single manufacturing sites in the Netherlands, Spain, and France. The samples from Spain and France were included to provide a point of reference only, as small sample sizes prevented any further infer-

ence being drawn. Samples were packaged in foil-lined sachets before subsampling. A total of 58 samples of WMP were sourced between 2011 and 2014, from New Zealand ($n = 36$), the Netherlands ($n = 20$), Spain ($n = 1$), and France ($n = 1$; Table 1). None of the WMP samples originating from the New Zealand factory were reprocessed. New Zealand samples were taken over the course of the milking season, beginning in spring and finishing in autumn. Subsampling was conducted in a manner to avoid trace element contamination.

Elemental Analysis

Subsamples of ~500 mg of WMP were placed in teflon vessels with 10 mL of quartz-distilled nitric acid. Ten percent of samples were analyzed in triplicate to assess repeatability. For every 20 samples analyzed, the following quality control samples were included: 1× blank, 1× matrix matched control, 1× duplicate sample, and 1× certified reference material (Skim Milk Powder BCR-063R from the Institute for Reference Materials and Measurements, Geel, Belgium).

Samples were digested using a microwave-accelerated reaction system (MARS6), which employed closed vessel acid digestions (CEM Corporation, Matthews, NC). The heating program increased temperature to 200°C over 25 min and held this for 15 min; maximum available power was 1,800 Watts. Once the acid digestion was complete, the samples were quantitatively transferred to 50-mL polypropylene tubes and dried for 8 h at 104°C. The resulting residues were redissolved by adding 10 mL of 2% quartz-distilled nitric acid, placing lids on, and heating to 60°C for 60 min. Trace element concentrations were quantified using inductively coupled plasma-mass spectrometry (Agilent 7500cs, Agilent Technologies, Santa Clara, CA). Calibration

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