Milk powder: Contamination- free automatic sample preparation for ED-XRF analysis

Abstract

Here, we present a sample preparation method for automatic pelletizing of milk powder into steel rings for analysis by X-ray fluorescence spectrometry. The material was pressed using the HP-PA automatic pelletizer and analyzed in an EDX spectrometer. We carried out a cross-contamination test with two significantly different sample types. After each sample, the HP-PA was automatically cleaned by compressed air and rotating brushes. The results demonstrate excellent repeatability of the chemical analysis without any cross-contamination. This shows that the applied method meets the high analytical standards in the sample preparation of biomaterials.

Key words

• **Automation** • **Pressing** • **Milk powder** • **Bio materials** • **HP-PA** • **EDX**

Introduction

Milk powder is mainly used for the production of cheese, yoghurt and chocolate. In 2018, the global milk powder market reached a volume of 10.4 Million tons with a CAGR of 4 % for 2019 to 2024.

Quality control of powdered milk includes but is not limited to analysis by XRF spectrometry. While automation of laboratories in the primary industry became very common, milk powder analysis is mostly performed manual yet. Due to the high fat content in milk powder avoiding cross-contamination in automatic sample preparation equipment can be challenging. Common automatic cleaning techniques include air and brush cleaning of the pressing tool. Moreover, the selection of suitable preparation parameters can also reduce the extent of cross-contamination.

Methods

We used an automatic HP-PA pellet press (Figure 1) to press the material into 51.5 mm steel rings. A pressure of 20 kN was applied for 10 s to inhibit the leakage of fat from the powder. Figure 2 shows the preparation parameters used. After 5 s of ramp up time, the force was held for 5 s and released immediately afterwards. The pellets were characterized by a smooth surface and showed high stability. After each preparation cycle, pipes and pressing tool

were automatically cleaned by compressed air and brushes.

Figure 1: *Automatic pellet press HP-PA used for sample preparation.*

First, we prepared 5 pellets of sample type A (samples 1-5), then 5 samples of type B (sample 6-10). Eventually another 5 samples of type A (samples 11-15) were pelletized. The alternation between sample types a and B was chosen to force cross-contamination between samples 5 and 6 as well as between samples 10 and 11.

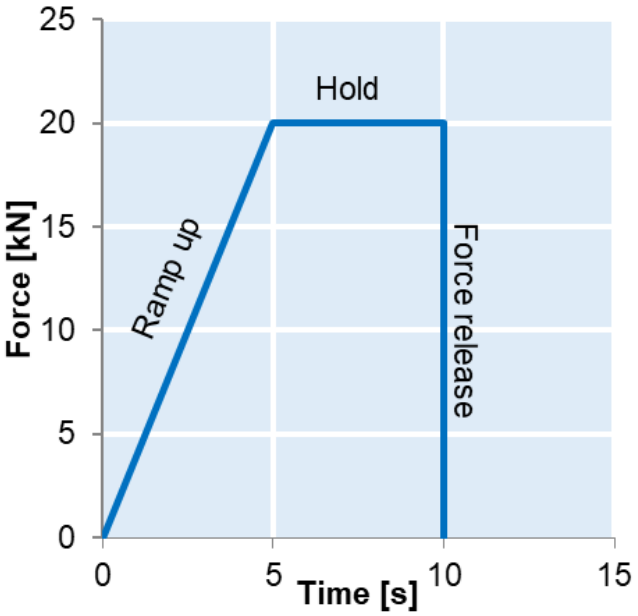

Figure 2: *Sample preparation process. The final pressing force of 20 kN was ramped up for 5 s and released immediately after 5 s of force hold duration.*

The chemical composition of the milk powder pellets was quantified by an energy dispersive XRF spectrometer (model Epsilon 3XL, Malvern Panalytical, Almelo, Netherlands).

We used a standardless method for the XRF analysis. Therefore, reported concentrations might not reflect the exact composition of sample. In fact, concentrations are likely one order of magnitude lower. Nevertheless, the analysis method is sufficient to reflect the sample preparation quality and to exclude significant cross-contamination between samples.

Results

Figure 3 shows the concentrations of calcium and phosphorus of sample type A and B as revealed by series measurements. In comparison to sample type A, sample type B revealed higher mean concentrations for Ca (5.0574 vs. 3.0913 %) and P (0.5638 vs. 0.3587 %). The variance for both sample types were low as shown by the standard deviation of 0.006 and 0.027 for Ca and 0.002 for P.

Furthermore, we did not find any indications for cross-contamination between samples 5 and 6 and samples 10 and 11.

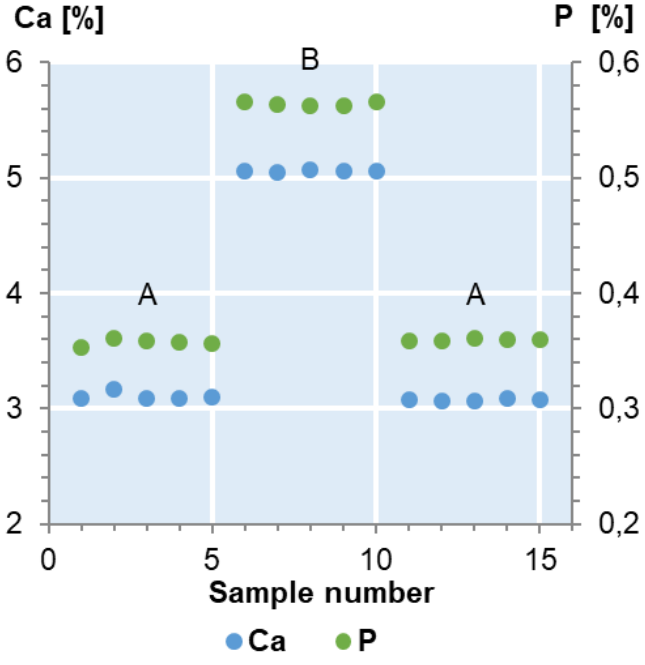

Figure 3: *Results for Ca (blue) and P (green). Five samples of type B were prepared in between samples of type A. Automatic routine cleaning was used in between samples.*

Table 1 shows mean and standard deviation of all elements analyzed. The standard deviation of all elements is low, indicating high repeatability and homogeneity.

	Type A		Type B	
	Mean	**σ**	**Mean**	**σ**
Mg	0.0476	0.001	0.0672	0.001
Si	0.0121	0.002	0.0144	0.001
P	0.3587	0.002	0.5638	0.002
S	0.2584	0.002	0.3318	0.001
Cl	1.4373	0.008	1.3128	0.005
K	3.0220	0.008	3.0636	0.004
Ca	3.0913	0.027	5.0574	0.006
Zn	0.0078	0.000	0.0106	0.000
Br	0.0070	0.000	0.0136	0.000

Table 1: *Mean and standard deviation (σ) of analyzed elements in sample type A and B. The actual concentrations might be one order of magnitude lower.*

Discussion

15 samples of milk powder were automatically prepared using the HP-PA pellet press. The pressing tool was routinely cleaned with compressed air and brushes after each preparation cycle.

In order to avoid fat leakage from the milk powder we applied especially short and low pressure on the material during the pelletizing process. By using these special preparation parameters we could prevent the material from sticking to the surfaces of the machine. At the same time, we were able to produce a stable pressed pellet that can be safely analyzed by an automatic XRF spectrometers.

Standardless obtained EDX results indicated no cross-contamination and furthermore revealed a high repeatability. The results show the possibility of reliably preparing and analyzing milk powder by an automated system without the risk of cross-contamination.

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