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Short communication

Validation and comparable analysis of aluminum in the popular Chinese fried bread youtiao by wavelength dispersive XRF

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ABSTRACT

Aluminum (Al) is an element in alum commonly used as a raising agent for Chinese flour products, especially for a typical fried food youtiao. In the present study, the feasibility of wavelength dispersive X-ray fluorescence spectrometry (WDXRF) for analysis of aluminum levels in youtiao was examined. Youtiao samples spiked with known amounts of aluminum were used for calibration. Linearity, accuracy, precision, and detection and quantification limits were tested, based on three calibration curves. For further validation, test youtiao samples were analyzed by both WDXRF and inductively coupled plasma optical emission spectrometry (ICP-OES). Comparison of the two methods showed that measurement performance was not significantly different. Taken together, these results indicate that WDXRF can form the basis of a rapid and simple methodology for measuring the aluminum content of youtiao, and that it is a good candidate for replacing ICP-OES for analyzing Al-containing flour products.

1. Introduction

Alum, e.g. aluminum sulfate or aluminum potassium sulfate, is commonly used as a kind of raising agent for Chinese flour products. Youtiao is a deep-fried flour product that is widely consumed in China. Its golden-brown color and crispy texture make it a very popular snack with all ages of the population. During production of youtiao, alum is a usual raising agent in the formula. To achieve the crisp and multi-porous texture, relatively large amounts of alum are added, especially in some small factories and roadside stalls. This may be a cause for concern, since many studies have found that chronic exposure to aluminum (Al) can have toxic effects on the nervous, skeletal, hepatic, cardiac, immune and reproductive systems (Costello, Eisen, Brown, Hammond, & Cullen, 2012; Di Paolo, Reverte, Colomina, Domingo, & Gómez, 2014; Li et al., 2011; Mailloux, Lemire, & Appanna, 2011; Zhu, Liu, Liu, & Li, 2013; Zhu et al., 2014). The Food and Agriculture Organization/World Health Organization (FAO/WHO) recommends a provisional tolerable weekly intake (PTWI) of 2 mg/kg (per mass of person; 2011), and the National Standard of China (GB2760-2011) clearly states that the Al remaining in fried food should be less than 100 mg/kg (per dry mass of food).

The commonly used method of inductively coupled plasma optical emission spectrometry (ICP-OES) is recognized as an efficient way to quantify trace elements in food such as tea leaves, mushrooms, maize, tomato, coffee, and dairy products (Bressy, Brito, Barbosa, Teixeira, & Korn, 2013; Durkan, Ugulu, Unver, Dogan, & Baslar, 2011; Duz et al., 2012; Szymczycha-Madeja, Welna, & Pohl, 2014, 2015; Şanal, Güler, & Park, 2011). However, the method requires a pretreatment step (sample digestion) that is time-consuming and ecologically unfriendly (corrosive and toxic reagents such HNO_3, H_2O_2, and HCl are used). X-ray fluorescence spectrometry (XRF), in contrast, entails a simple and rapid sample preparation process, without any chemical treatment of the sample. In addition, the analysis time is short and the whole procedure is automated easily. Further advantages of XRF over conventional analytical methods, including other spectroscopic modalities, such as atomic absorption spectroscopy (AAS) and ICP-OES, are that it is selective and that it allows for semi-quantitative or quantitative analysis of samples, as well as re-analysis if necessary (Marguí, Fontas, Buendía, Hidalgo, & Queralt, 2009).

Recently, XRF has been applied to determine the trace elements in different kinds of food, such as soybean, dairy products, cumin, grain, and bread (Hondrogiannis et al., 2012; Otaka, Hokura, & Nakai, 2014; Paltridge et al., 2012; Pashkova, 2009; Perez & Leon, 2010; Perring, Andrey, Basic-Dvorzak, & Blanc, 2005). However,

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to the best of our knowledge, no previous studies have investigated the application of XRF to determining the Al content of youtiao.

In this study, we examined the applicability and effectiveness of wavelength-dispersive XRF (WDXRF) in determining the Al content of commercial and lab-made youtiao samples. The results were compared to those achieved via the ICP-OES method.

2. Materials and methods

2.1. Preparation of youtiao with known Al contents

The youtiao dough consisted of flour (500 g), water (270 g), salt (8.5 g), and different amounts of aluminum potassium sulfate dodecahydrate and sodium bicarbonate (see Table 1). The amount of aluminum potassium sulfate and sodium bicarbonate was based on traditional formula and usage analyses of commercial products (data not shown). This amount range covered the level in most of youtiao products. Food-grade low-gluten flour and sodium bicarbonate were purchased from Jiangnan Flour Group Co. (Danyang, China) and Yuhua Chemical Industry Co. (Hengyang, China). Water was purified using a Milli-Q water purification system (Millipore Co., Milford, USA). Aluminum potassium sulfate dodecahydrate was of high analytical grade (⩾99%) and purchased from Sinopharm Chemical Co. (Shanghai, China). All ingredients were mixed and kneaded for 5–6 min to form a soft elastic structure with a smooth skin. Then the dough was rested at 23 °C for 4 h. After resting, it was rolled out and cut into pieces of length, 8.0 cm, and width, 2.5 cm. The pieces were then divided into pairs and one of the two was stacked on top of the other (parallel). The stacked pairs were pressed down at the center with a wooden rod, stretched to 25 cm long from both ends, and then deep-fried at 180 °C for 160 s.

Table 1
Ingredients for the youtiao standards.

No.	$KAl(SO_4)_2{\cdot}12H_2O$ (g)	$NaHCO_3$ (g)	Al added (mg/kg)
std1	0	0	0
std2	0.159	0.175	18.52
std3	0.318	0.350	35.64
std4	0.477	0.525	55.35
std5	0.636	0.700	71.68
std6	0.795	0.875	92.81
std7	1.576	1.734	191.93
std8	3.173	3.490	375.22
std9	4.789	5.268	554.33
std10	6.426	7.069	732.81
std11	8.084	8.892	919.42

$KAl(SO_4)_2{\cdot}12H_2O$, aluminum potassium sulfate dodecahydrate; $NaHCO_3$, sodium bicarbonate.

2.2. System calibration

The matrix significantly affects the determination of metals by XRF spectrometry and this fact has to be taken into account when selecting the calibration standards. In our study, calibration was performed with a set of 11 in house synthetic standards, which were prepared as described in Section 2.1 and Al concentrations ranged from 0.00 mg/kg to 919.42 mg/kg. Therefore, the matrixes of tested and standard samples were the same.

2.3. WDXRF analysis

To ensure homogeneity, samples were obtained by the point-centered quarter' sampling method. Each sample (20 g) was ground into powder using a SPEX 6870 Freezer Mill (SPEX SamplePrep, Metuchen, USA) and then dried at 105 °C for 2.5 h. The powder was quickly pressed through a sieve with a diameter of 0.15 mm, and then pressed into disks (30 mm diameter). All sample pellets were stored in a desiccator prior to WDXRF analysis.

A commercial XRF-1800 instrument (SHIMADZU, Kyoto, Japan) equipped with a rhodium target tube and flow proportional counter detector (FPC) was used to measure the Al content of the youtiao samples; measurements took place under vacuum conditions at 40 kV and 95 mA.

2.4. ICP-OES analysis

Reference Al values of all youtiao samples, including standards, were determined via ICP-OES, using validated in-house procedures. According to the National Standard Recommended of China GB/T 23374-2009, sample digestion was performed in closed vessels with an MDS-10 digestion device (SINEO, Shanghai, China). To a weighed (0.4 g) youtiao sample in a digestion vessel, 8 mL of HNO_3 (65–68%) and 1 mL of H_2O_2 (30%) were added. Digestion parameters were as follows: 130 °C for 10 min, 150 °C for 5 min, 180 °C for 10 min, 1000 W of power. The samples were allowed to cool below 100 °C, after which, the HNO_3 was evaporated

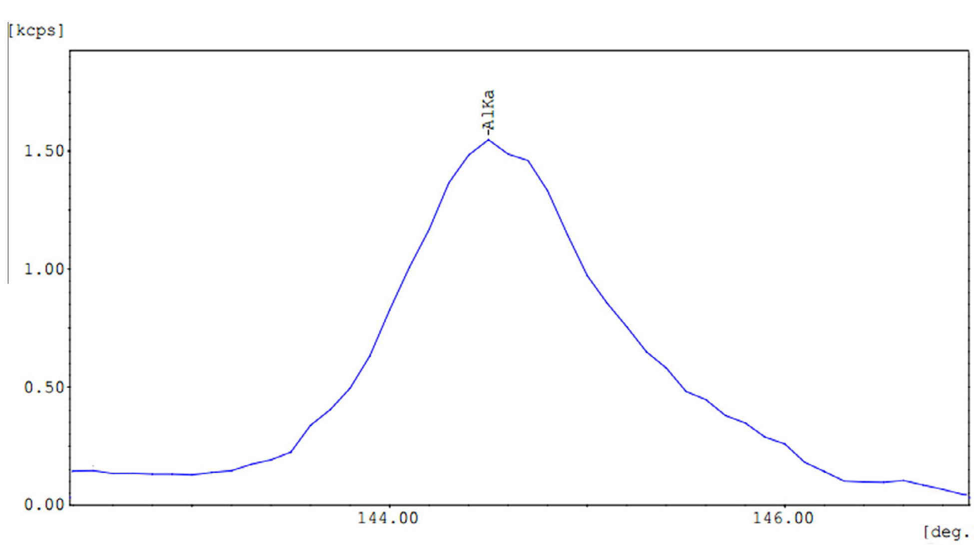

Fig. 1. Aluminum X-ray fluorescence spectrum of the standard sample std11 (40 kV, 318 95 mA, 23 s, pentaerythrite crystal, flow proportional counter detector).