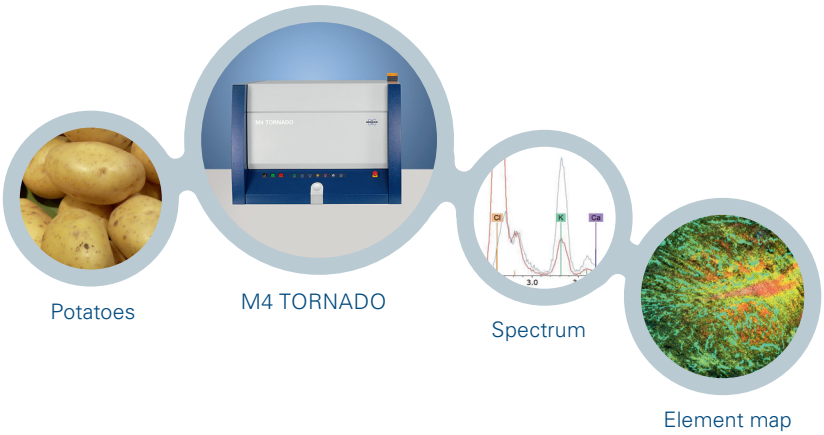


Lab Report XRF 470

Analysis of natural and processed food samples

Every year numerous food recalls are announced due to potential foreign objects found in food items. Foreign objects and contaminants including metal shavings, wood or plastic pieces, and insects may be present in foods because of manufacturing issues, product infestation, or tampered products. Injuries and deaths are reported as a result of the effects of contamination.

Contamination can cost companies hundreds of thousands of dollars in lawsuits and lost merchandise. The US Food and Drug Administration (FDA) publishes strict guidelines to prevent potential hazards to consumers. Regulatory action can be taken against products that do not comply.

While food companies already have some form of screening implemented, Micro-XRF allows for additional analysis of the contaminants providing two additional features above current industry screening practices - material characterization and dimensional measurement. This information can be used to trace the source of the contaminant and to maintain FDA compliance.

The M4 TORNADO Micro-XRF spectrometer is already used for a wide variety of applications from material science to geology, and it has great potential in the food industry. This lab report describes the analysis of different potato samples from a raw potato to the packaged product considering food contamination, patterns of nutrients, and distribution of salt and flavoring.

Instrumentation

The measurements were performed using the Bruker M4 TORNADO, a Micro-XRF spectrometer equipped with an X-ray source with Rh target, a polycapillary X-ray optic with a spot size below 20 microns, and a silicon drift

detector for high spectral resolution and high throughput capabilities (300 kcps). The instrument also has a large vacuum sample chamber and an XYZ-stage for quick sample movement.

The M4 TORNADO can perform point and area analyses and identifies elements from Na to U. The small spot size easily enables the detection of sample inhomogeneities and small particle inclusions within the sample.

The analyses were performed on a raw potato to check for contaminants, on freeze-dried cross sections of several potato breeds to investigate the distribution of nutrients, and on potato chips to understand flavoring and salt content. All measurements were taken as area scans. The measurement conditions are given in Table 1.

Table 1 Samples and measurement conditions

Sample	Voltage (kV)	Current (µA)	Chamber pressure (mbar)	Measure-ment time
Contaminated potato	50	200	20	2 h 30 min
Freeze-dried potato	50	600	20	3 h 17 min
Potato chip	50	200	20	45 min

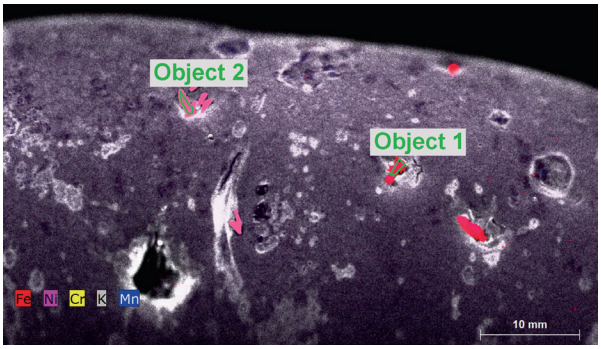
Analysis of a contaminated raw potato

A raw potato with metal contaminants was analyzed with the M4 TORNADO. At the completion of the measurement, several software tools were used for analysis. Objects were drawn around the metal contaminants on the potato surface (Fig. 1a) in order to acquire spectra representative of the specific areas. A spectrum match tool finds similar spectra to the contaminant spectrum from a library of known standards. The library was compiled from alloy standards measured in point mode under the same measurement conditions. As can be seen in Fig. 1b, the stainless steel alloy SS 408 is the closest spectral match for the object 1 surface contamination.

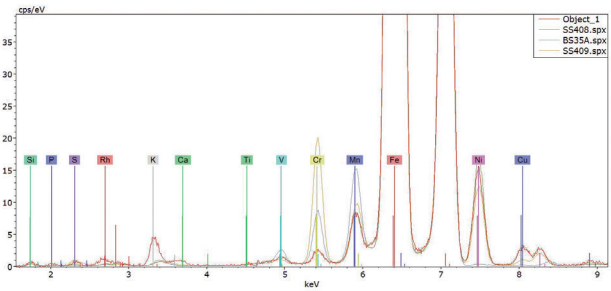
Analysis of potato nutrients in a thin section

For a more in-depth investigation, such as in research and development, some sample preparation can significantly enhance the insight into the product and its properties. Therefore, analyses were performed on thin potato slices, which were freeze-dried after cutting them out from the potato. The benefit of the approach of thin slices is that high spatial resolution can be utilized, whereas in a voluminous

Surface contamination analysis on a raw potato



(a)



(b)

Fig. 1 a) Elemental distribution map of a potato with different metal contaminants; b) Spectrum of Object 1 overlaid with possible alloy database matches.

sample the incoming converging beam will start to diverge as it passes the focal plane (Fig. 2). Depending on the distance between a particle and the focal plane, objects will become increasingly fuzzy. If a particle is beneath the focused surface, the beam already widens again, virtually increasing the size of the object.

In case of a prepared thin section, the source volume is naturally reduced to the cross section of the beam with

Probed volume vs. sample thickness

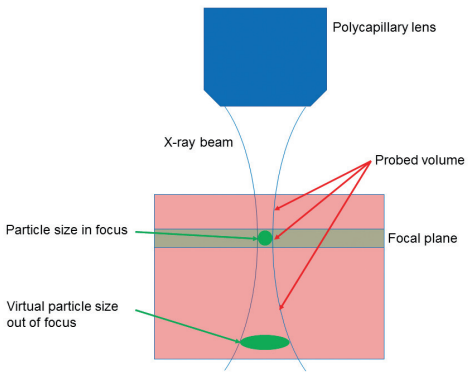


Fig. 2 Schematic of probed volume and resulting particle size. Thin section (green rectangle) with resulting resolution for a particle and volumenous sample (pink rectangle) with resulting, defocussed size of particle.

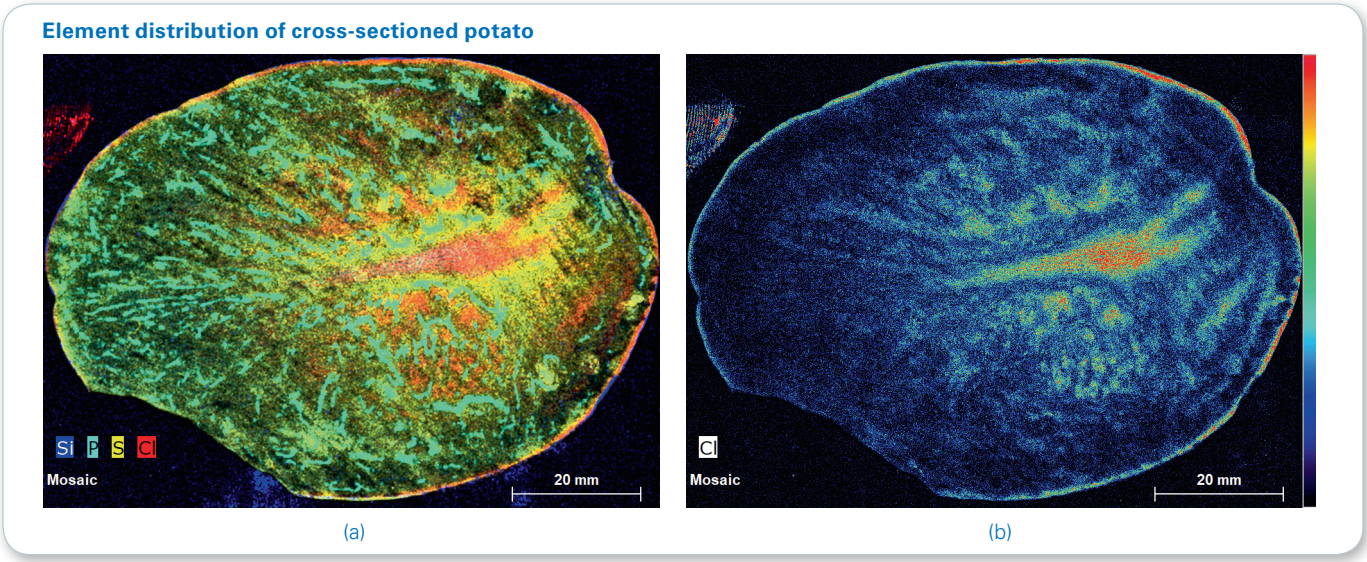


Fig. 3 a) Multi-element map of a freeze-dried slice of a fresh potato, b) Heat map of the normalized Cl intensity (in arbitrary units) across the potato slice sample to visualize the Cl distribution

Table 2 Count rate per element across the whole slice for different potato breeds (in cps).

Breed No.	Mg	Al	Si	P	S	Cl	K	Ca	Ti	Mn	Fe	Cu	Zn	Rh
Breed 1	13	6	6	403	480	534	9461	388	1	49	141	20	50	105
Breed 2	18	7	20	534	680	821	10380	376	5	57	203	22	64	105
Breed 3	16	8	16	524	541	723	10492	442	4	53	555	26	65	120
Breed 4	14	9	22	448	506	686	9620	635	5	54	302	30	76	124
Breed 5	13	11	16	617	555	692	13759	552	3	47	159	30	59	126

the sample. This allows for the highest possible spatial resolution and thus allows to resolve small inhomogeneities down to 20 µm. As the system creates a hyperspectral data cube that contains signals from all visible elements, distribution maps for each element can be extracted.

Fig. 3a displays the multi-element map of a thin freeze-dried slice of a fresh potato. The distributions of individual elements can easily be extracted. Overlaying these element maps allows for understanding the correlation between elements in the specimen and conclusions regarding the chemistry of the specimen, i.e. what type of salt was used. To visualize small concentration variations of Cl in the map, a false color display (Fig. 3b) is used to show full dynamics of the chlorine signal intensity. It is apparent that Cl is concentrated in the center of the potato slice.

If the sample preparation is consistent and similar measurement conditions are applied, it is possible to compare the composition semi-quantitatively. Therefore, integral spectra of similarly sized objects are selected and count rates per element are compared (Table 2). The count rates reflect the changes in the composition between the different breeds. This allows for conclusions on the nutritional value of the various breeds. Rh is an artefact (backscattering) from the excitation, it reflects the density of the probed volume.

Depending on the element analyzed, the limit of detection can be as low as a few ppm (for elements with atomic numbers $22 < Z < 42$). Through XMethod, an additional software package, and the use of suitable standards, an empirically based quantification can easily be built.

Analysis of industrially fabricated potato chips

As shown on the contaminated potato sample (Fig. 1), the M4 TORNADO is a valuable tool to evaluate the product quality control. Its advantages include a small spot size to check for inhomogeneities down to the µm-scale and to acquire detailed elemental maps.

Potato chip analysis

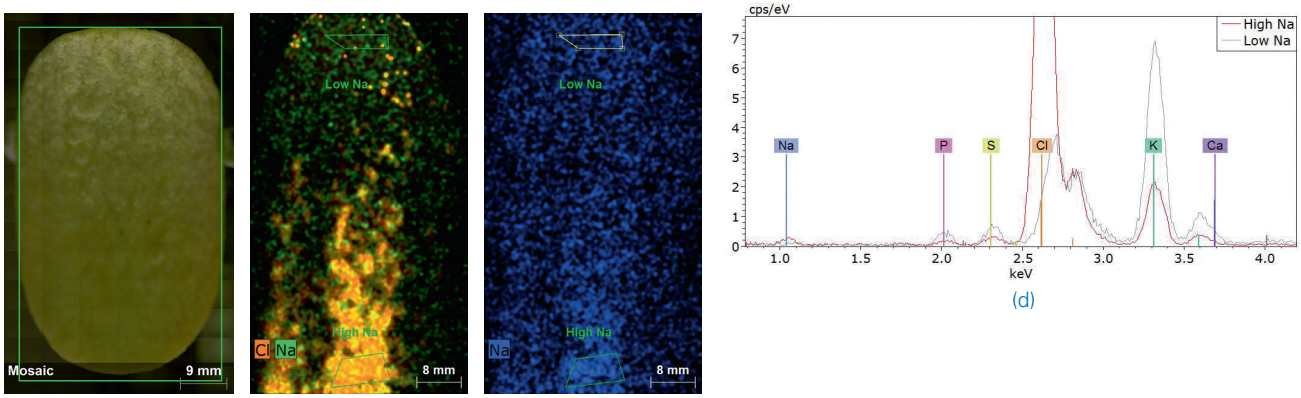


Fig. 4 a) Mosaic image of the potato chip with measurement area, b) element distribution maps for Na and Cl, c) element distribution map of Na with defined objects, d) spectra corresponding to the objects in c).

An industrially fabricated potato chip (Fig. 4a) was analyzed to display its elemental distribution. No sample preparation was required. The potato chip has a noticeable curvature. The highest points of the sample, the edges, were brought into focus for the measurement. Although portions of the sample are not in focus, they are still able to provide elemental distribution maps. Varying heights also create a seemingly 3-dimensional effect to the sample, the portions that are farther away from the focal plane provide less signal (inverse-square law for distance between excitation, sample, and detector).

Conclusion

The M4 TORNADO provides a wide variety of opportunities for analyses within the food industry. These include the distribution of elements as well as the identification and quantification of small inclusions. The Micro-XRF technique is non-destructive and requires minimal or even no sample preparation.

Analyzing contaminants, such as metal inclusions in a raw potato, led to positive identification of alloys. Differences in the nutrients distribution between several potato breeds could be visualized and their absolute content was compared. The variation in the elemental composition of a potato chip could easily be detected, showing a non-uniform application of the flavoring.

Authors

Rebecca Novetsky, Micro-XRF Applications Specialist, Bruker AXS Inc., Madison, USA
Dr. Max Bügler, Micro-XRF Applications Specialist, Bruker Nano GmbH, Berlin, Germany

Bruker Nano GmbH
Berlin · Germany
Phone +49 (30) 670990-0
Fax +49 (30) 670990-30
info.bna@bruker.com

www.bruker.com/m4tornado