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Application News

No. X275

X-Ray Analysis

Quantitative Analysis of Sodium in Food Raw Materials by EDX

Atomic absorption spectrophotometry (hereinafter, AA) is generally used in elemental analysis of the mineral components and salt contained in raw materials for food products. When analyzing solids or powders by AA, chemical pretreatment such as addition of an acid followed by thermal decomposition is necessary. Because X-ray fluorescence spectrometry makes it possible to measure solids, powders, and liquid as-is with only simple pretreatment, this technique has been adopted or studied as a substitute for AA.

AA is used in process control of sodium in powder raw materials for food products. In this article, we conducted a comparative study of quantitative analysis of sodium by AA and a Shimadzu EDX-8100 energy-dispersive X-ray fluorescence spectrometer. Two pretreatment methods were examined, pressure forming (hereinafter, press method) and a powder container method (hereinafter, powder method), and a certain correlation with AA was obtained with both.

T. Nakao, H. Nakamura

Element

Na

Sample

Powder sample of solid raw material for foods

(1) Samples for calibration curve:
2.67%, 11.56%, 20.21% (AA analysis values)
1 sample each, total of 3 levels

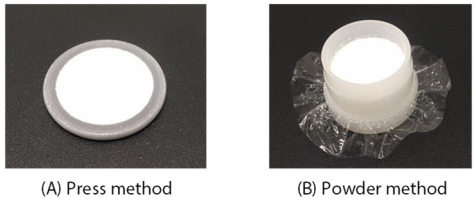
(2) Unknown samples:
Three samples (①, ②, ③) prepared from one type

Sample Pretreatment

Sample pretreatment was done by the following two methods (Fig. 1).

(A) Press method
The sample material was packed in a vinyl chloride ring with an inner diameter of 22 mmφ and pressure formed at 50 kN for 30 s.

(B) Powder method
6 g of the sample material was placed in a sample container lined with a polypropylene film (thickness: 5 µm) and simple compacting was applied.



(A) Press method (B) Powder method

Fig. 1 Examples of Sample Pretreatment

Calibration Curve

Calibration curves were prepared by measuring each of the levels 3 times, for a total of 9 points. Fig. 2 shows the overlaid curves of the calibration curves for the press method and the powder method. The sensitivity of the press method was approximately 3 times higher than that of the powder method. The accuracy of both calibration curves is satisfactory.

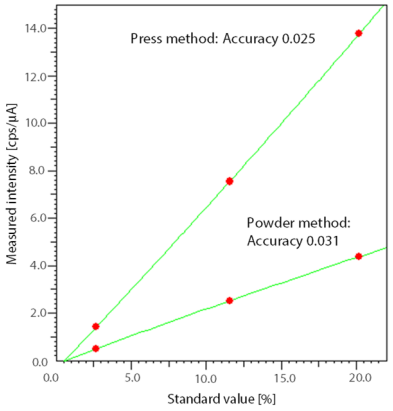


Fig. 2 Calibration Curves

Quantitative Analysis of Unknown Samples

Quantitative analysis of the unknown samples ① to ③ was conducted consecutively 3 times using the calibration curves. Table 1 shows the results, together with the AA analysis values.

As an example, the control value was set to within ±10% of the average of AA values.

Table 1 Comparison of Quantitative Analysis Results of Unknown Samples with AA Values

Unit [%]

Sample/repetition		Press method	Powder method	AA
①	1	6.54	6.33	6.45
	2	6.56	6.34	6.43
	3	6.56	6.36	6.45
②	1	6.42	6.15	6.40
	2	6.43	6.12	6.41
	3	6.40	6.13	6.29
③	1	6.42	6.08	6.33
	2	6.45	6.09	6.39
	3	6.41	6.05	6.37
Average		**6.46**	**6.18**	**6.39**
Standard deviation		0.07	0.12	0.05
RSD [%]		1.05	1.99	0.80
Control value		5.7 - 7.0		

<Discussion>

In comparison with AA, the average values with both the press method and the powder method were in good agreement with the average of AA values, their differences being within 4%.

It can be understood the measurement values were within the control values, even considering measurement error.

Instrument Repeatability

Table 2 shows the results of 10 consecutive measurements of unknown sample ①. Both the press method and the powder method show high repeatability, with relative standard deviation (RSD) of 0.5% or less.

Table 2 Instrument Repeatability Investigation of Unknown Sample ①

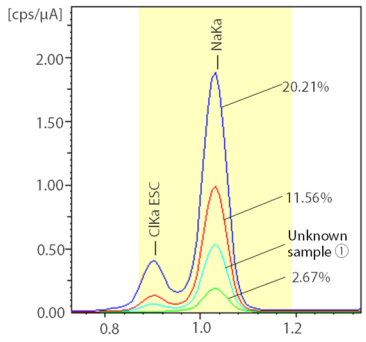
Unit [%]

n	Press method	Powder method
1	6.54	6.33
2	6.56	6.34
3	6.56	6.36
4	6.55	6.36
5	6.58	6.34
6	6.55	6.36
7	6.61	6.35
8	6.57	6.38
9	6.58	6.35
10	6.58	6.34
Average	**6.57**	**6.35**
Standard deviation	0.02	0.01
RSD [%]	0.32	0.23

Spectra

1. Fig. 3 and Fig. 4 show the NaKα analysis line spectra.
   1) 3 levels of calibration curve samples by press method, unknown sample ①
   2) Unknown sample ① by press method and powder method

   All peaks are clear, and intensity is also sufficient.
2. Influence of coexistent elements Cl and intensity calculations
   1) The adjoining ClKα escape peak on the low energy side was separated.
   2) The adjoining ClKβ escape peak on the high energy side was subtracted by spectrum intensity overlap correction.



Calibration curve samples, unknown sample ① (press method)

Fig. 3 Analysis Line Spectra

Conclusion

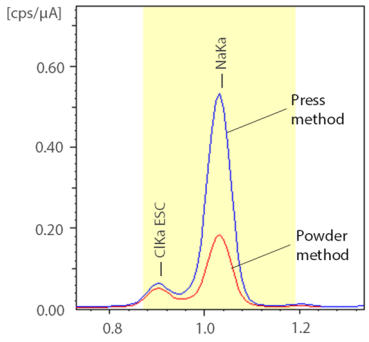
- In a quantitative analysis of sodium in a raw material for food products, a certain correlation with AA was obtained, demonstrating the possibility of using EDX.
- A more appropriate pretreatment method can be selected, corresponding to the content, accuracy, control value, simplicity, and other requirements. Table 3 summarizes the features of the pretreatment methods.
- Use of EDX (as a substitute or in parallel with AA) is considered effective for simplifying control and analysis procedures, and for reducing equipment and costs in production plants.

Table 3 Differences of Press Method and Powder Method

Pretreatment method	Press method	Power method
Simplicity	(Good) Requires press machine and briquetting ring.	(Excellent) Sample is simply placed in a film-lined container.
Peak intensity	(Excellent) No attenuation because X-ray is irradiated directly on the sample.	(Good) In case of light elements, the film lining causes attenuation.
Lower limit of quantitation (reference)	0.035% -	0.067% -

Table 4 Measurement Conditions

Instrument	: EDX-8100 (8000) with 12-sample turret
Element	: Na
Analysis group	: Quantitative
Detector	: SDD
X-ray tube	: Rh target
Voltage	: 15 [kV]
Current	: Auto [μA]
Collimator	: 10 [mmφ]
Primary filter	: None
Atmosphere	: Vacuum
Integration time	: 300 [s]
Dead time	: Max. 30 [%]



Unknown sample ①
(press method and powder method)

Fig. 4 Analysis Line Spectra

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Related Products Some products may be updated to newer models.

› EDX-8100
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