Determination of major, minor and trace elements in rice flour using the 4200 Microwave Plasma-Atomic Emission Spectrometer (MP-AES)

Application note

Food testing

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Introduction

The analysis of foodstuffs, such as rice, is of particular interest for nutrient elements present at high concentrations, and also for toxic elements such as cadmium that can be present at trace levels. The analysis is important to ensure product quality and safety, as well as determining product origin. Food scares related to contamination not only constitute a health risk but also undermine consumer confidence. This can lead to lost earnings through reduced sales and loss of credibility through adverse publicity.

Flame Atomic Absorption Spectroscopy (FAAS) is well established for the analysis of foods, but with lab budgets coming under increasing pressure and current market trends for lower cost of ownership, improved

performance, ease of use, and safety, many FAAS users are looking to transition to another technique to expand their analytical capabilities.

Agilent has expanded its atomic spectroscopy portfolio to include the Microwave Plasma-Atomic Emission Spectrometer. The Agilent 4200 MP-AES is the second generation microwave plasma instrument that features an improved waveguide design that is capable of running samples with high total dissolved solids without compromising detection limits. The 4200 MP-AES significantly reduces running costs through the use of nitrogen as its plasma gas. The use of nitrogen also increases safety, by removal of flammable gases, and allows unattended operation of the instrument. The 4200 MP-AES is easy to use, and is able to achieve lower detection limits than a standard FAAS, as well as being able to determine additional elements such as phosphorus.

This application describes the analysis of rice flour for cadmium and other major, minor and trace elements on the 4200 MP-AES.

Experimental

Instrumentation

The innovative 4200 MP-AES features a second generation waveguide and torch, with mass flow controlled nebulizer gas flow. The 4200 MP-AES has a robust toroidal plasma with a central channel temperature of ~5,000 K which eliminates many of the chemical interferences that are present in FAAS and also expands the concentration working range of the 4200 MP-AES when compared the FAAS. This means that the element specific sample preparation that is commonplace when using FAAS is not necessary when using the 4200 MP-AES, improving ease of use and reducing cost. The 4200 MP-AES also achieves lower detection limits than FAAS, particularly for phosphorus, which enables the analysis of extra elements. By running on nitrogen, the 4200 MP-AES offers reduced operating costs and increased lab safety compared to flame AA, through the avoidance of flammable and costly gases such as acetylene, and nitrous oxide.

The analysis was carried out using a 4200 MP-AES equipped with the standard sample introduction setup consisting of the OneNeb nebulizer and a double pass spray chamber. An SPS 3 autosampler was used to deliver samples to the instrument, allowing the system to be operated unattended.

The MP-AES features continuous wavelength coverage which allows the analyst to select wavelengths that are appropriate for the expected concentration range, and free from spectral interferences. Method conditions for the selected wavelengths are shown in Table 1 and common method conditions are shown in Table 2.

Table 1. Agilent 4200 MP-AES operating parameters

Element	Wavelength	Read time (s)	Nebulizer Flow (L/min)
P	214.915 nm	2	0.55
Cd	228.802 nm	10	0.55
Mg	280.271 nm	1	0.55
Zn	213.857 nm	5	0.55
Mn	403.076 nm	3	0.55
K	766.491 nm	1	0.55
Cu	324.754 nm	2	0.75
Fe	438.354 nm	5	0.75
Ca	422.673 nm	1	1.00

Sample preparation

NIES CRM No.10c Rice Flour (NIES, Japan) was analyzed to validate the analytical method. The rice flour samples were digested using a Milestone Ethos microwave digestion system[1]. Samples were prepared in duplicate with approximately 0.5 g of rice flour CRM accurately weighed into separate TFM vessels. This was followed by the addition of 7 mL of HNO_3 and 1 mL of H_2O_2 and placed in the microwave digestion unit. The samples were digested using the preloaded digestion methods, allowed to cool, and then made up to 25 mL with deionized water. The final solution contained 2% total dissolved solids. No ionization suppressants or matrix modifiers were required for the analysis.

Calibration range

The calibration concentration range of the standard solutions are summarized in Table 3. As the working range of 4200 MP-AES far exceeds that of FAAS (by up to 20x in some instances), only one dilution of the sample is required to measure the complete set of elements. The calibration fit for all wavelengths used was linear.

Results and Discussion

Method detection limits (MDLs)

MDLs were determined from the analysis of 10 digested blank samples. The MDLs (3σ) for the selected analytical wavelengths are listed in Table 4.

Table 2. Common method conditions

Parameter	Value
Replicates	3
Pump rate	15 rpm
Sample uptake delay	30 seconds
Rinse time	60 seconds
Stabilization time	10 seconds
Fast Pump during Uptake and Rinse	On (80 rpm)
Nebulizer	OneNeb
Spray chamber	Double pass cyclonic
Autosampler	Agilent SPS 3
Sample pump tubing	Orange/green
Waste pump tubing	Blue/blue

Table 3. Working concentration range of the 4200 MP-AES standard solutions

Element	Wavelength	Concentration range	
P	214.915 nm	0–100	ppm
Cd	228.802 nm	0–1.0	ppm
Mg	280.271 nm	0–40	ppm
Zn	213.857 nm	0–4.0	ppm
Mn	403.076 nm	0–1.0	ppm
K	766.491 nm	0–100	ppm
Cu	324.754 nm	0–1.0	ppm
Fe	438.354 nm	0–1.0	ppm
Ca	422.673 nm	0–4.0	ppm

Table 4. Agilent 4200 MP-AES element wavelength and MDL (mg/kg in sample)

Element/ Wavelength (nm)	Ca 422.673	Cd 228.802	Cu 324.754	Fe 438.354	K 766.491	Mg 280.271	Mn 403.076	P 214.915	Zn 213.857
MDL (mg/kg)	0.10	0.16	0.05	0.44	3.0	0.06	0.05	13	0.15

Analysis of certified reference material

Results of the analysis of major, minor and trace elements in rice is listed in Table 5. The measured values (average result on two different 4200 MP-AES instruments carried out in duplicate) are in good agreement with the certified values for all CRM samples. The results demonstrate the capability of the 4200 MP-AES to achieve excellent results across a wide concentration range in a sample with 2% dissolved solids.

Long term stability

A digested rice flour sample was repeatedly analyzed under method conditions over 8 hours to test the long term stability of the method. The test was performed under controlled laboratory environmental conditions within the instrument operating specification, with a recalibration every 2 hours. The resulting stability plot is show in Figure 1. Excellent stability of < 3 % RSD for all elements was achieved, demonstrating the capability of the 4200 MP-AES, OneNeb nebulizer and mass flow controlled nebulizer gas flow to handle 2% total dissolved solids.

Table 5. Results of NIES No.10c Rice Flour. All results in mg/kg in the solid sample.

Element/ Wavelength (nm)	Ca 422.673	Cd 228.802	Cu 324.754	Fe 438.354	K 766.491	Mg 280.271	Mn 403.076	P 214.915	Zn 213.857
Mean	96.0	1.96	4.13	11.50	2700	1174	37.35	3139	22.02
SD	2.5	0.11	0.29	1.03	105	23	1.04	92	0.48
Certified value	95	1.82	4.1	11.4	2750	1250	40.1	3350	23.1
2SD certified	2	0.06	0.3	0.8	100	80	2.0	80	0.9
% difference	101.0	107.7	100.8	100.9	98.2	93.9	93.1	93.7	95.3

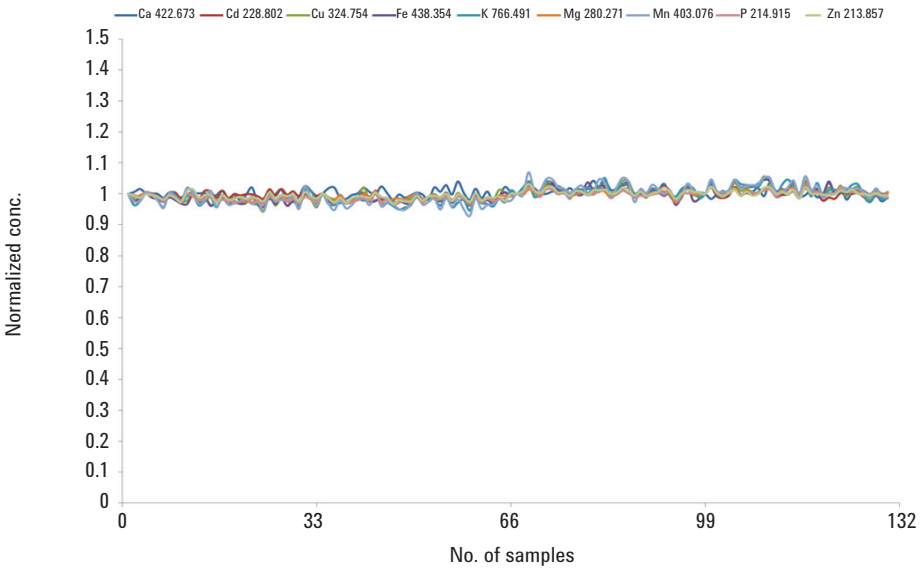

Figure 1. Normalized concentration of elements in a rice flour digest analyzed over an 8 hour time period, with recalibration every 2 hours.

Conclusion

A method for the determination of major, minor and trace elements in rice flour has been described. The next generation 4200 MP-AES achieved recoveries in a rice CRM of +/- 10 % of the assigned value, with MDLs sufficient for the analysis and excellent long term stability.

The excellent analytical performance, including phosphorus which is not practical by FAAS, multi-element unattended operation, improved safety and ease of use make the 4200 MP-AES the ideal alternative for FAAS users looking to transition to a new technique. Furthermore, the sample preparation process can be simplified, with no modifiers or ionization suppressants required due to the higher temperature excitation source of the MP-AES.

Reference

1. Milestone Application Note. Food/Feed. Rice Flour. ID HPR-FO-39. Milestone Ethos with internal temperature sensor, HPR1000/10S high pressure segmented rotor.

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