Feasibility of Microwave-Induced Combustion for Ultra-Trace Determination of Chlorine in Edible Flour by Ion Chromatography

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Abstract
Up to 1 g of edible flour was digested by MIC using 50 mmol L⁻¹ NH₄OH as absorbing solution for further Cl determination by IC.

Introduction
Blanching and carotenoids oxidation of flour is slow, natural and a required process after grinding and storage of the product. The time for this process can be reduced using chlorinated compounds. This process is used industrially to improve the quality of flour. Therefore, some of these compounds are toxic to the health. Sample preparation is a critical step for further halogens determination (e.g., Cl) by chromatographic or spectrometric techniques, requiring bringing the analytes into the solution. In this way, sample digestion methods have been described such as extraction, pyrohydrolysis, microwave-induced combustion (MIC) and others.¹ In this work, a systematic study was performed to demonstrate the feasibility of MIC for digestion of high mass of flour and further Cl determination by ion chromatography (IC).

Results e Discussion
Several flour samples from different raw materials such as potato, corn, wheat and whole wheat flour as well as from different countries such as Austria, Belgium, Brazil and Poland were used. For MIC method, sample pellets (up to 1000 mg) were placed on the quartz holder together with a disc of filter paper and 50 µL of 6 mol L⁻¹ NH₄NO₃ solution. Then, quartz holder was introduced into the quartz vessel containing 6 mL of absorbing solution. After vessels were closed, pressurized with 20 bar of oxygen and placed inside the microwave cavity (Multiwave 3000®, Anton Paar, Austria). The microwave heating program was 1400 W for 5 min and 0 W for 20 min (cooling). Chlorine determination was performed by IC (Metrohm, Herisau, Switzerland) with an anion-exchange column (Metrosep A Supp 5, polyvinylalcohol with quaternary ammonium groups, 150 x 4 mm i. d.) and a conductivity detector. The mobile phase was 3.2 mmol L⁻¹ Na₂CO₃ and 1.0 mmol L⁻¹ NaHCO₃ with flow rate set at 0.7 mL min⁻¹.

Conclusions
The proposed MIC method was suitable for Cl determination by IC in edible flours with different raw materials at low levels. This method allows a safe digestion of high sample mass (up to 1000 mg).

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