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} NH_4OH 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} NH_4NO_3 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} Na_2CO_3 and 1.0 mmol L^{-1} $NaHCO_3$ with flow rate set at 0.7 mL min^{-1}. The reference values for Cl were obtained using a total Cl analyzer (Multi EA® 5000, Analytik Jena AG, Germany). Some parameters of MIC method, such as absorbing solution (H_2O, 10, 25 or 50 mmol L^{-1} NH_4OH and 10, 25 or 50 mmol L^{-1} $(NH_4)_2CO_3$) and sample mass (100 to 1000 mg) were investigated. The results obtained for Cl using different absorbing solution were statistically similar (ANOVA, 95% confidence level), with relative standard deviation lower than 6%. Therefore, 50 mmol L^{-1} NH_4OH was chosen as absorbing solution for evaluation of sample mass. Using 100 to 1000 mg of flour, the maximum pressure reached during the combustion was lower than 40 bar, which corresponds to 50% of the maximum pressure allowed by the manufacturer. Therefore, MIC was considered safe even for higher sample mass (up to 1000 mg). However, when sample was 800 mg or higher, incomplete combustion (residues remaining in the quartz holder) was observed. Nevertheless, the accuracy of the proposed method was not affected by the incomplete combustion and the residual carbon content was lower than 1.5%. Accuracy was evaluated using a certified reference material of rice flour (NIST 1568a) and agreement was about 97%. The proposed method was applied for Cl determination in other flour samples and concentration ranged from 4.9 to 658 µg g^{-1}. The limit of detection for Cl using MIC and IC was 0.25 µg g^{-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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