Application of high pressure microwave-assisted digestion flow system for juice and milk sample preparation

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Sample preparation is usually required for metals determination in food samples by inductively coupled plasma optical emission spectrometry (ICP OES), since matrix effect of carbon in the plasma is a main reason to obtain unreliable data. Microwave-assisted acid digestion is a well-known and widely applied technique for food sample preparation, but, it is usually performed in batch mode which may led to analytical errors due to sample handling. Flow analysis is a good approach to overcome these drawbacks, but care must be taken, since the short residence time of the sample in the irradiated zone led to lower efficiency of digestion compared batch procedures. This can be improved increasing the reactor volume as shown by Marques et al. (2016)[1]. In the literature, there are just few applications of high pressure flow systems for liquid sample digestion, such as fruit juices and milk. These samples are not easily digested in an online flow system, because juices are rich in sugars that promote violent reactions inside the reactor and milk is rich in fats that are not easily degraded. In the work here described the performance of a high pressure microwave-assisted flow digestion system with large volume reactor to digest these samples was evaluated. The digestions were carried out in a coiled perfluoroalkoxy (PFA) tube reactor (13.5 mL) put inside a labmade autoclave pressurized at 40 bar. The system was operated at 500 W microwave applied power and 5.0 mL min^{-1} flow rate. Juice samples of apple and mango were successfully digested in this flow system using 3.7 mol L^{-1} HNO_3 and 0.3 mol L^{-1} HCl. The residual carbon contents (RCC) were 16 and 29% for apple and mango juices, respectively, when juice samples were diluted to a final concentration of 47% v/v. Concentrated HNO_3 (10.5 mol L^{-1}) was effectively applied for digesting milk samples and the RCCs were 23 and 25% for partially skimmed and whole milk, respectively, when milk samples were diluted to a final concentration of 25% v/v. Calcium, Fe, K, Mg, Na, P, S, Sr and Zn were determined in both types of samples and Al, Ba, B, Cu and Mn were determined in juice samples by ICP OES. According to F test the precision of the microwave-assisted digestion flow system was similar to microwave-assisted digestions performed in closed-vessels (batch mode) for most elements. Elemental contents determined in juice and milk samples digested in flow mode were in good agreement with those determined in samples digested in batch mode according to a t-test adopting a confidence interval of 95%.

[1] T.L. Marques, H. Wiltsche, H. Motter, J.A. Nóbrega, G. Knapp, J. Anal. At. Spectrom. 30 (2015) 1898–1905.