MICROWAVE-ASSISTED ACID DECOMPOSITION OF MILK AND DETERMINATION OF MAJOR AND TRACE ELEMENTS USING AN ICP-OES WITH AXIAL VIEW

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Milk is a source of inorganic nutrients for newborns and its chemical analysis can be difficulty owing to its complexity. In this study two strategies were investigated based on microwave-assisted procedures to fast decomposition of milk samples before introducing them in an ICP-OES with axial view (Vista, Varian). The first strategy was implemented in a focused microwave furnace with open borosilicate glass vessels (Star 6, CEM). In this decomposition it was initially employed a mixture of concentrated HNO_3 (10 ml) and H_2SO_4 (3 ml) and during the 2nd step was added five successive aliquots of H_2O_2 (1 ml). The following heating program was used: 1st step: 3 min ramp - 110°C; 2nd step: 5 min ramp and 5 min plateau - 180°C; 3rd step: 10 min - 180°C. The digests were diluted to 50.0 ml with water. The other procedure was based on the use of a microwave furnace with closed-vessels (Ethos 1600, Milestone). The samples were digested with 2 ml HNO_3 conc. plus 1 ml H_2O_2 conc. and applying the following heating cycle: 250 W - 1 min; 0 W - 1 min; 250 W - 5 min; 400 W - 5 min; 650 W - 5 min. The digests were diluted to 25.0 ml with water. Both procedures were implemented using the same sample volume (2.5 ml). The performance of each procedure was evaluated taking into account the final concentration of acid in the digests and the determination of Al, Ca, Fe, Pb, Zn, and residual carbon. Each microwave-assisted procedure led to digests with different acid concentrations and this parameter needs to be considered due to its effect on nebulization and excitation processes. The final acid concentrations were obtained by titration with standard NaOH solution. Therefore, when the acid concentrations was in the 1.2-1.9 mol L^{-1} range, a solution 1.4 mol L^{-1} HNO_3 was used for preparing the reference solutions. The residual carbon content of digested milk samples was determined by ICP-OES with axial view using C 193.025 nm emission line with Y 371.022 nm as an internal standard. The higher the acid volume added in the decomposition, the lesser the residual carbon content. The highest residual carbon content and the smallest acidity were obtained for the whole milk samples due to the higher content of lipids in these samples. The digests obtained with the radiation focused microwave furnace showed higher acidity and lower residual carbon than the digested samples employing the microwave furnace with closed vessels. Use of a greater volume of acid improved the decomposition but the high acidity can degrade the performance of the ICP-OES. The ICP-OES with axial view configuration led to better detection limits, but the direct determination of trace elements, such as Pb, cannot be performed with suitable precision and accuracy. All the other elements were accurately determined in a standard reference material applying the two different microwave furnaces (SRM 8435 - whole milk powder, NIST).

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