Effect of Nitric Acid Concentration on Closed-Vessel Microwave-assisted Digestion of Plant Materials

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An ideal sample digestion procedure should be fast and generate digestates with physical and chemical characteristics compatible with the analytical technique used for analytes measurement. Frequently aggressive conditions are adopted to reach quantitative decomposition of organic compounds in plant materials. At high temperature and pressure in a closed vessel microwave-assisted procedure severe reaction conditions can be attained without using an elevated amount of oxidant acids. Most works described in the literature did not investigate systematically this possibility and thus there is room to improve microwave-assisted procedures considering the amounts of acids employed and the consequent acid concentration of the digestates. The study developed dealt with the closed vessel microwave-assisted digestion of four species of *Paspalum* genera, a native Brazilian forage used for bovine feeding, using a combination of nitric acid and hydrogen peroxide. All experiments were performed employing an Ethos 1600 microwave system (Milestone, Germany) equipped with 10 PFA fluorpolymer closed vessels, and with pressure and optical fiber temperature sensors. The effect of nitric acid concentration on digestion efficiency was tested for solutions (2.0 mL) containing 2.0, 3.0, 5.0, 7.0, and 14 mol L^{-1} plus 1 mL of hydrogen peroxide (30% v v^{-1}). All experiments were carried out using a 5-step heating program with the maximum temperature set at 240°C. Subsequently, five heating programs with different temperatures were evaluated by keeping the acid concentration at 2.0 mol L^{-1}. The digestion efficiency was assessed by determining residual carbon content (RCC), Al, Ba, Ca, Cu, Fe, K, Mg, Mn, P, S, and Zn by inductively coupled plasma optical emission spectrometry with an axial-view configuration (Vista, Varian, Australia). The obtained results did not indicate any pronounced effect of acid concentration on RCC, which varied from 5 to 15 mg L^{-1}. The analytes were accurately determined without any appreciable effect caused by RCC on emission intensities. It can be concluded that *Paspalum* can be efficiently digested using diluted nitric acid solution in spite of its original carbon content around 45% m m^{-1}. In all experimental conditions adopted the temperature- and pressure-digestion time curves showed the same profiles indicating that different acid concentrations were effective to generate proper conditions when used in closed vessels heated by microwave radiation. The remaining acid concentration in the digestates before diluting varied from 1.6 to 5.6 mol L^{-1} depending on the acid concentration of the digestion solution.

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