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MULTIVARIATE OPTIMIZATION OF MICROWAVE-ASSISTED DIGESTION PARAMETERS FOR ELEMENTAL ANALYSIS OF HONEY SAMPLES

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Honey is a complex matrix for elemental analysis due to its wide variability in composition and high organic matter content, composed mainly of sugars such as fructose and glucose[1]. Therefore, proper sample preparation is a crucial step to ensure standardization and reliable results in elemental determinations. This study aimed to optimize the acid decomposition of honey using microwave-assisted digestion, targeting elemental analysis by Inductively Coupled Plasma Optical Emission Spectrometry (ICP-OES) and Inductively Coupled Plasma Mass Spectrometry (ICP-MS). The influence of nitric acid concentration and pre-digestion on the efficiency of the process was evaluated, with the goal of minimize both residual carbon and acidity levels. A full factorial experimental design (n=3) was applied, varying nitric acid (HNO_3) concentrations (2.00, 3.59 and 7.00 mol L^{-1}) and pre-digestion (without and 2 h), using three honey samples from *Apis mellifera* with different colors[2]. Sample masses of 250 mg of honey were weighed into polytetrafluoroethylene (PTFE) vessels and subjected to pre-digestion using concentrated nitric acid in the digestion tubes. After pre-digestion, variable volumes of ultrapure water and 2.0 mL of 30% hydrogen peroxide (H_2O_2) were added. For 2.00, 3.59 and 7.00 mol L^{-1} HNO_3, the respective volumes of concentrated nitric acid were 1.0, 1.8 and 3.6 mL, along with 5.0, 4.2 and 2.5 mL of ultrapure water. Digestion was carried out using a microwave system following a program with temperature of 195º C and power of 1800 W. Residual acidity was determined by acid-base titration, using 10 mL of sample, 1 mol L^{-1} NaOH as titrant and phenolphthalein as indicator. Residual carbon was quantified by inductively coupled plasma optical emission spectrometry in radial view using a calibration curve in the 0 - 5000 mg/L range. The results were evaluated using the global desirability function, complemented by analysis of variance (ANOVA) to define the best condition. Statistical analysis (ANOVA) showed that only acid concentration has significant influence ($p = 0.0029$), while pre-digestion time was negligible ($p = 0.7096$). The condition yielding the highest global desirability ($D_{global} = 0.776$) involved the use of 2 mol L^{-1} HNO_3 and 2 h of pre-digestion, with low residual acidity (1.67%) and good carbon removal efficiency (1791 mg/L). However, the same acid concentration without pre-digestion also showed satisfactory performance ($D_{global} = 0.689$), with residual carbon of 2062 mg/L and residual acidity of 1.76%, making it a viable and more practical option. Higher acid concentrations, such as 3.59 and 7 mol/L, resulted in lower desirability indices ($D_{global} = 0.601$ and 0.250, respectively), mainly due to increased residual acidity, which may increase blank values and require subsequent sample dilution before analysis.

1 Camargo, R. C. R.; Pereira, F. M.; Lopes, M. T.; Wolff, L. F. *Mel: características e propriedades*. Teresina, PI: Embrapa Meio-Norte, dezembro 2006. Documentos 150. 28 p.
2 Barros Neto, B; Scarminio, IS; Bruns, RE. Como fazer experimentos. 4 ed. Porto Alegre: Bookman, 2010. 413p.