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An innovative microwave-assisted digestion method with diluted hydrogen peroxide for rapid extraction of trace elements in coal samples followed by inductively coupled plasma-mass spectrometry

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Abstract

The present work describes the development of a novel and efficient microwave-assisted digestion method based on the use of diluted hydrogen peroxide (H_2O_2) for rapid extraction of trace elements in coal samples prior to inductively coupled plasma-mass spectrometry (ICP-MS) determination. Diluted H_2O_2 was used to reduce acid wastes generated in analytical laboratories. After H_2O_2 digestion, the addition of diluted nitric acid was required to enhance the extraction of trace elements. Key parameters that affected the extraction efficiencies of the target analytes were examined using coal certified reference material (SARM 20). Improved digestion efficiency and quantitative trace element recoveries were obtained when 7 mol L^{-1} , $200 \text{ }^\circ\text{C}$, 0.1 g and 10 minutes were used for $[\text{HNO}_3]$, temperature, coal amount and time, respectively. Agreement of 92-114 % between the obtained results and the certified values was observed under the optimum conditions for most of the investigated trace elements in all three certified reference materials (SARMs 18, 19 & 20). Moderate extraction efficiencies (70-89 %) were achieved for Sc, V, Cr, Y, and La, while Ti and Hf showed poor accuracy (10-50 %). The use of H_2O_2 as the only digestion reagent resulted in colourless digest solutions with 0.084 mol L^{-1} of nitric acid. The diluted acid might have caused low method detection limits (MDLs) of $0.003\text{-}3.5 \text{ } \mu\text{g g}^{-1}$ and method quantification limits (MQL) of $0.01\text{-}12.5 \text{ } \mu\text{g g}^{-1}$ for the current study due to less spectrometric interferences. These interferences are commonly caused by concentrated acids frequently used in classic acid digestion methods. The proposed microwave-assisted digestion method was comparable with literature reports and was successfully applied for trace element analysis in three South Africa coal samples.

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