

Área: QA

DETERMINATION OF POLYCYCLIC AROMATIC HYDROCARBONS IN PYROLIGNEOUS EXTRACT USING UALLE AND GC-FID

Antônio M. G. Lima (PG),¹ Márcia H. S Kurz (PQ),¹ Carlos R. M. Peixoto (PQ),¹ Lenise G. de Oliveira (PQ),¹ Fábio Ferreira Gonçalves (PQ)¹.

antoniosapgomes@gmail

¹Universidade Federal do Rio Grande - FURG. Rua: Barão Cahy, 125 – Cidade Alta, Santo Antônio Da Patrulha – RS, 95500 – 000.

Keywords: Pyrolygneous Extract; Biomass; Sample Preparation; GC-FID

Highlights

A GC-FID method using UALLE–SPE was developed and validated for analysis of 16 PAHs in pyrolygneous extract, meeting INMETRO and SANTE standards, and revealed PAH levels above MAPA limits.

Resumo/Abstract

In response to the escalating global demand for food, the use of biofertilizers, such as pyrolygneous extract (PE), has gained prominence in modern agriculture. However, the presence of potentially carcinogenic polycyclic aromatic hydrocarbons (PAHs) in these products necessitates rigorous quality control. The objective of this study was to develop and validate a robust analytical method for the extraction and quantification of 16 priority PAHs, defined by the USEPA, from PE. The method employed a three-step procedure: Ultrasound-Assisted Liquid-Liquid Extraction (UALLE) of PAHs from the matrix using toluene, followed by a cleanup step with Solid Phase Extraction (SPE) C18 cartridges, and a final pre-concentration under an air flow. The quantification was performed using Gas Chromatography with a Flame Ionization Detector (GC-FID). The method was validated according to the criteria established by INMETRO and SANTE. Recoveries for the PAHs ranged from 38% to 109.5% with a relative standard deviation (RSD) below 13%. All compounds exhibited linearity with coefficients of determination (R^2) greater than 0.99. The Limit of Quantification (LOQ) and Limit of Detection (LOD) values were in the range of 1.6 to 3 $\mu\text{g. L}^{-1}$ and 0.53 to 1 $\mu\text{g. L}^{-1}$, respectively. A significant matrix effect, characterized by signal amplification greater than 20%, was effectively compensated by the use of standard addition curves for calibration. The method was applied to real PE samples produced by small producers from the Paranhama Valley in the State of Rio Grande do Sul, Brazil, revealing total PAH concentrations between <LOQ and 15 $\mu\text{g. L}^{-1}$, values significantly above the regulatory limit of 0.7 $\mu\text{g L}^{-1}$ established by MAPA in Brazil. These findings underscore the need for validated analytical methods for routine quality control of PE to ensure compliance with safety standards and support sustainable agricultural practices.

Agradecimentos/Acknowledgments

FURG, PPGQTA, FAPERGS and CAPES