# Evaluation of microwave-induced combustion for further trace elements determination in pitch by ICP OES

**Leticia S. F. Pereira (PG), Gabrielle D. Iop (IC), Angélica C. Frohlich (IC), Clarissa M. M. Santos (PG), Érico M. M. Flores (PQ), Fábio A. Duarte (PQ)***

*Departamento de Química, Universidade Federal de Santa Maria, 97105-900, Santa Maria, RS.*

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## Introduction

Pitch is obtained after crude oil processing and has been an alternative to obtain high commercial value carbon as carbon fibers and graphite electrode materials. In petroleum industry, the knowledge of content of trace elements defines the quality of the raw material. Conventionally, elements determination requires a preliminary step of sample digestion and this step may be considered as the most critical especially when complex matrices are used, such as pitch that contains high levels of stable aromatic compounds. This sample preparation method was recently developed and has several advantages over classical combustion methods, particularly for samples difficult to digest, such as crude oil and related products for subsequent trace elements determination.[1,2] In this work, microwave-induced combustion (MIC) method was proposed for digestion of pitch sample. Some parameters that influence the MIC method were evaluated as well as a comparison was performed using dry ashing method.

## Results and Discussion

For the MIC method, pellets were prepared using a hot place and 400 mg of sample were weighed and placed in a watch glass which was heated at about 90 °C in order to obtain fused pellets. This procedure was required once the pellets formation by mechanical pressing was not suitable. The filter paper was placed within the quartz holder and the fused pellet was positioned on the paper. Ammonium nitrate solution (50 µL of 6 mol $L^{-1}$) was used as igniter and added on the filter paper. A solution of 6 mL $HNO_3$ was added to the quartz vessel as absorbing solution and its concentration was evaluated from 1 to 14.4 mol $L^{-1}$ $HNO_3$. After closing the vessels, they were pressurized with 20 bar of $O_2$ for 30 s. Microwave heating program was started as follows: 1400 W for 5 min (optional reflux step) and 0 W for 20 min for cooling. Pellets of certificate reference material (CRM) were prepared using a hydraulic press with a 3 ton during 1 min. Accuracy of MIC method was evaluated by analysis of a CRM of coal (BCR 40) and employing dry ashing method. Elements determination was carried out by ICP OES. According to results, it was possible to obtain a suitable solution as diluted as 3 mol $L^{-1}$ $HNO_3$ for all analytes absorption using MIC method (Table 1). However, using dry ashing, losses were observed for all elements, except of Mg and Sr. It is important to mention that the blanks for dry ashing were significantly high for some elements. Agreement between proposed MIC method and certified values ranged from 93 to 97%. The limits of detection using MIC and ICP OES ranged from 0.01 to 0.90 µg $g^{-1}$.

**Table 1.** Results for trace elements determination by ICP OES after dry ashing and MIC.

| Element | Concentration, µg $g^{-1}$ | |
|---|---|---|
| | Dry ashing | MIC |
| Al | 42.5 ± 2.0 | 87.7 ± 4.2 |
| Cd | 5.41 ± 0.25 | 7.32 ± 0.35 |
| Cu | 1.33 ± 0.11 | 2.56 ± 0.12 |
| Fe | 179 ± 9 | 211 ± 11 |
| Mg | 11.6 ± 0.5 | 12.2 ± 1.9 |
| Mn | 3.43 ± 0.16 | 4.08 ± 0.20 |
| Pb | 110 ± 6 | 130 ± 5 |
| Sr | 1.05 ± 0.05 | 1.01 ± 0.05 |
| V | < 0.96 | 0.97 ± 0.05 |
| Zn | 436 ± 20 | 498 ± 47 |

## Conclusions

Using MIC method was possible to determine ten elements in pitch samples without analyte losses and/or contamination. The proposed MIC method allows the combustion of up to 400 mg combining good performance for pitch digestion, safe conditions and relatively high sample throughput.

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