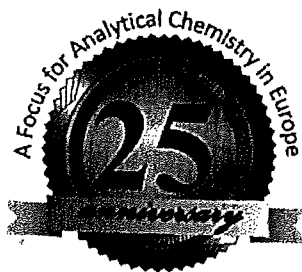
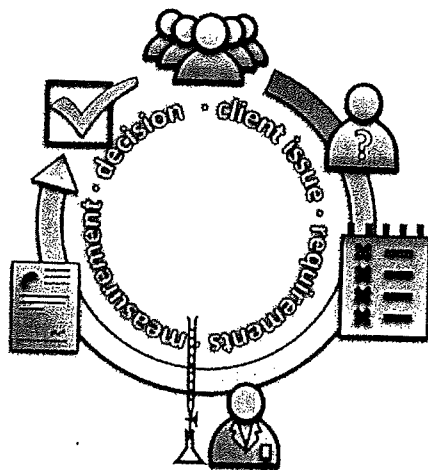




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BOOK OF ABSTRACTS

LISBON, 19-21 MAY 2014



<http://eurachem2014.fc.ul.pt>

Environmentally friendly matrix effect matching evaluation for the major elements in solid biofuel characterization by atomic absorption spectrometry

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The thermo-chemical conversion of biomass has become increasingly important, from the last decade, due to the need to reduce CO₂ emissions and to increase renewable energy sources. Currently, the use of biomass already has a strong economic environmental and social impact.

The biomass source can be very different whereby biomass characterization is a crucial task allowing the prediction and prevention of problems associated with biofuel burning. Major elements (Al, Ca, Mg, Na, K, Fe, Si and Mn) determined by flame atomic absorption spectrometry (FAAS) requires preliminary procedures to convert the solid sample into a solution destroying either organic matter or inert matrix. In this work grinded biofuels (under 0.25 mm) were decomposed following EN 15290:2011 and closed vessels microwave assisted extraction, under controlled conditions of temperature and pressure. A mixture of H₂O₂-HNO₃-HF was used in a first step and then boric acid was added for HF complexation. This procedure leads to an efficiently dissolution, because H₂O₂ - HNO₃ mixture ensures the organic matter destruction and HF dissolves the aluminosilicate matrix; the recovery of each element without loss of volatile species; minimization of contamination; increasing of measurement precision and technician safety. However, resilient matrix effects on the elemental determination by FAAS were noticed due to HF-H₃BO₃ mixture and calibration by standard addition did not compensate constant deviations.

Matching calibration standards with the same reagents and applying the same procedures used for samples could compensate for matrix effects that influence analytical response, but high quantities of toxic reagents must be used.

In this work the need of a fluoboric-boric acid matrix matching was studied using one way ANOVA tests. No significance differences were obtained for Si, Al, Fe and Mn among of seven groups of mixtures of nitric acid and blank reagent in proportions from 0 to 100 %, because the calculated values were lower than the critical value, $F_{6,14}=2.84$ ($P=0.05$). For Ca, Mg, Na and K significance differences were obtained. All samples were analysed using a dilution factor of ten since the mass fractions quite exceed the analytical dynamic ranges. The calibration solutions were prepared using the reagent blank in the same proportion.

The procedure was validated using the standard reference material 1573a, tomato leaves, purchased from National Institute of Standards and Technology. Recoveries within 1 ± 0.1 were obtained.

This research was funded by National Funds through FCT – Fundation for Science and Technology under the project BiomAshTech *PTDC/AAC-AMB/116568/2010*.