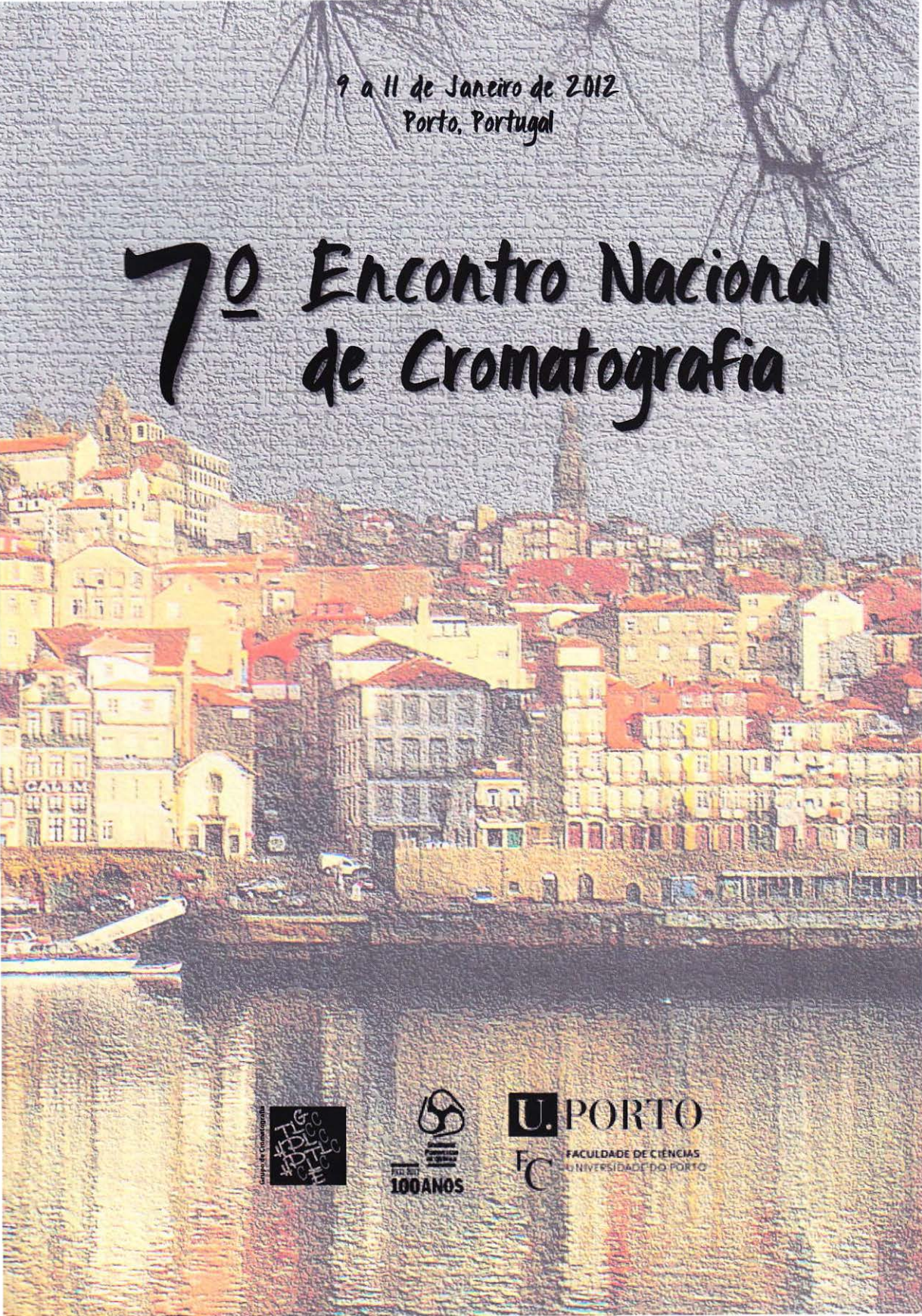
9 a 11 de Janeiro de 2012
Porto, Portugal

7º Encontro Nacional de Cromatografia



Grupo de Cromatografia

100 ANOS

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FACULDADE DE CIÊNCIAS
UNIVERSIDADE DO PORTO

7º Encontro Nacional de Cromatografia

Grupo de Cromatografia
Sociedade Portuguesa de Química

Departamento de Química e Bioquímica
Faculdade de Ciências da Universidade do Porto

9 a 11 de Janeiro de 2012

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7º Encontro Nacional de Cromatografia

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P85. Analysis of acids and sugars in fruit-based drinks by SEC-UV-RI

Cédric Sequeira[a,], Luís G. Dias[a], Jorge Sá Morais[a], Ana C.A. Veloso[b,c], Adélio A.S.C. Machado[d,*], António M. Peres[a,e,*]*

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Beverage industry produces a large and diverse range of soft drinks, beverages containing flavorings and/or fruit juices (sodas and fruit juices), of which the quality and safety must be monitored to protect and satisfy customers. From the raw ingredients to the final product, quality control is needed to ensure product safety, quality, labelling, regulatory compliance and consistency. The development of analytical techniques for simultaneous analysis of different compounds essential to control the product quality, as an alternative to several independent traditional reference methods, is of major importance. Therefore, the present work reports the application of size exclusion chromatography (SEC), which allows carrying out analysis free of organic solvents, using two detectors coupled in series - Ultraviolet (UV) and Refractive Index (RI) - for the simultaneous analysis of acidifiers (citric, tartaric, lactic, acetic, malic and ascorbic acids, by UV), and sweeteners (sucrose, glucose and fructose by RI), in commercial non-alcoholic beverages with different levels of added fruit juice. Ascorbic acid is used as a stabilizer in the soft drinks, improving the beverage shelf-life stability due to its antioxidant properties.

The results showed that the simultaneous calibrations for acid compounds (UV) and for glucose (RI) were straight-forward. On the other hand, for sucrose and fructose simultaneous analysis, the calibrations (RI) were more complex since the predictive models established had to take into account malic and tartaric acids interferences, regardless the good resolution between the peaks of sucrose and fructose.

Finally, the results for sample analysis showed that all the sugars evaluated were present in the juice drinks as well as the citric, tartaric, malic and ascorbic acids. In all samples, lactic and acetic acids were not detected.

Analysis of acids and sugars in fruit-based drinks by SEC-UV-RI

Cédric Sequeira[a,], Luís G. Dias[a], Jorge Sá Morais[a], Ana C.A. Veloso[a], Adélio A.S.C. Machado[c], António M. Peres[a,b,*]*

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Introduction

From the raw ingredients to the final product, beverage industry needs to ensure:

- product safety;
- quality;
- labeling;
- regulatory compliance;
- consistency.

New green analytical methodologies for simultaneous analysis of different compounds are essential to control the beverages quality, as an alternative to several independent traditional reference methods.

Objectives

To apply size exclusion chromatography (SEC) with two detectors coupled in series - Ultraviolet (UV) and Refractive Index (RI) - for the simultaneous analysis of:

Acidifiers compounds:
- citric acid
- tartaric acid
- ascorbic acid
- malic acid
- lactic acid
- acetic acid

Sweeteners compounds:
- sucrose
- fructose
- glucose

Samples

Commercial non-alcoholic beverages with different levels of added fruit juice:

- fruit drinks with addition of fruit juice above 30%;
- fruit drinks with added fruit juice between 14% and 30%;
- gasified juices with a percentage of fruit juice between 6% and 10%;
- ice tea drinks with addition of less than 4% of fruit juice.

Acidifiers compounds in samples were analysed directly.

Sugars compounds in samples were analysed after dilution (1:10).

HPLC equipment

Varian Prostar 220 Pump
Varian 9050 UV (Ultraviolet detector)
Varian RI-4 (refractive index detector)
Rheodyne 7725i manual injector with Loop of 20 μL
Software star chromatography workstation, version 6.4
Jones 7981 chromatography column oven

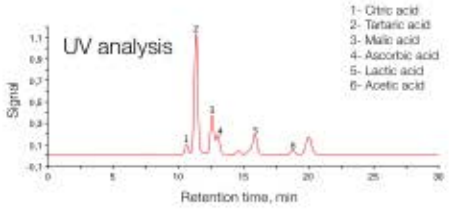
COLUMN
Supelcogel C-610H de 30cm x 7,8mm DI

HPLC conditions

Column temperature: 30ºC
Eluent composition: 1% of phosphoric acid aqueous solution
Eluent elution: isocratic
Flux: 0.5 mL/min
Eluent temperature: 40ºC

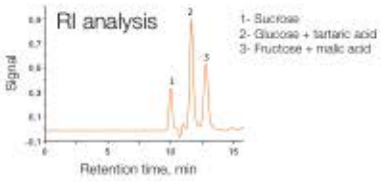
Results

SEC-UV allowed to analyse directly all the acidifiers in samples



SEC-RI allowed to analyse directly only the sucrose in samples.

Overlapping peaks (SEC-RI):
Glucose + tartaric acid
Fructose + malic acid

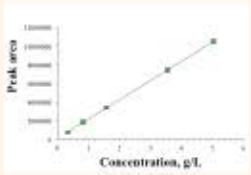


All calibrations: R≥0,9997

Glucose analysis

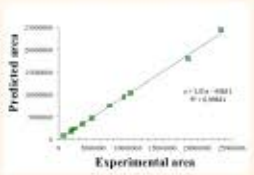
- Simple calibration: glucose without tartaric acid

$$\text{Peak Area} = 2.064\times10^{6} \times [\text{Glucose}] + 1.04\times10^{5}$$



- Multiple calibration: glucose with tartaric acid

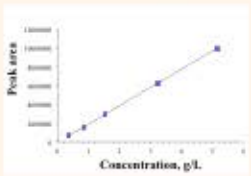
$$\text{Peak Area} = 2.05\times10^{6} \times [\text{Glucose}] + 2.00\times10^{6} \times [\text{Tartaric acid}] + 1.51\times10^{5}$$



Fructose analysis

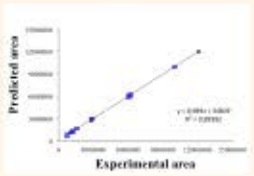
- Simple calibration: fructose without malic acid

$$\text{Peak Area} = 1.948\times10^{6} \times [\text{Fructose}] - 2.70\times10^{4}$$



- Multiple calibration: fructose with malic acid

$$\text{Peak Area} = 1.94\times10^{6} \times [\text{Fructose}] + 1.49\times10^{6} \times [\text{Malic acid}] - 6.62\times10^{3}$$



Conclusions

Quantification of glucose or fructose was a complex task (overlapped peaks with acids):

→ additive signals from tartaric or malic acid presence were obtained;

→ predictive models established must take into account tartaric or malic acid interference.

→ The three sugars evaluated were present in all the juice drinks;

→ Citric, tartaric, malic and ascorbic acids were present in beverages.

→ Lactic and acetic acids were not detected.

→ No statistical differences were observed for slope values obtained for glucose or fructose between simple and multiple calibration models (p=>0.32).