

XVII EDICIÓN PREMIOS JOSÉ ANTONIO GARCÍA DOMÍNGUEZ

En el marco de la XXI Reunión Científica de la SECyTA (50ª Reunión Científica del GCTA), celebrada en Almería del 25 al 27 de octubre de 2022, se otorgaron los premios José Antonio García Domínguez a las mejores comunicaciones orales y tipo cartel presentadas en dicha reunión, patrocinados, como en ediciones anteriores, por la empresa Bruker. Tras debatir los méritos científicos de las presentaciones, los jurados encargados de fallar los premios, tomaron por unanimidad los siguientes acuerdos:

1^{er} Premio a la mejor Comunicación Oral (800 €)

Comunicación: Y-HYP-04

Título: SIMULTANEOUS ANALYSIS OF HIGHLY POLAR AND MULTIRESIDUE-TYPE PESTICIDES BY HEART-CUTTING 2D-LCMS

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2º Premio a la mejor Comunicación Oral (600 €)

Comunicación: Y-OMI-01

Título: FINGERPRINTING BY GAS CHROMATOGRAPHY AND HIGH-RESOLUTION MASS SPECTROMETRY (GC-ORBITRAP-HRMS): A PROMISING TOOL FOR ORIGIN AND PROCESSING AUTHENTICATION OF THYME

Autores: *Araceli Rivera-Pérez**, *Roberto Romero-González*, *Antonia Garrido Frenich*

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1^{er} Premio al mejor Póster (400 €)

Comunicación: P-FUN-04

Título: ENANTIOMERIC SEPARATION OF DRUGS BY NANO-LIQUID CHROMATOGRAPHY USING A CHIRAL COLUMN OF AMYLOSE TRIS(3-CHLORO-5-METHYLPHENYL CARBAMATE). APPLICATION OF A LIQUID-LIQUID MICROEXTRACTION SYSTEM TO WATER SAMPLES

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2º Premio al mejor Póster (300 €)

Comunicación: P-FA-05

Título: OPTIMIZATION OF A GREEN EXTRACTION PROCEDURE TO OBTAIN POLYPHENOLIC COMPOUNDS FROM THE WINE INDUSTRY BY-PRODUCTS

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La entrega de los premios tuvo lugar el 27 de octubre de 2022, durante la ceremonia de clausura de la XXI Reunión Científica de la SECyTA.

Juan Vicente Sancho
Secretario de la SECyTA

1^{er} Premio a la mejor Comunicación Oral (Y-HYP-04)

SIMULTANEOUS ANALYSIS OF HIGHLY POLAR AND MULTIRESIDUE-TYPE PESTICIDES BY HEART-CUTTING 2D-LCMS

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The increasing use of pesticides in food production has raised the demand for multi-residue methodologies capable to cover compounds with a wide range of polarity due to the difficulty associated to highly polar pesticides not amenable to common multi-residue extraction methods [1]. Previous strategies have proposed the analysis of polar and non-polar pesticides in a single run using a parallel column configuration, although the need to inject a sample aliquot with the appropriate solvent composition for each column could be an aspect to optimize [2]. For this purpose, the present work proposes the development of a multi-residue pesticide method by online two-dimensional liquid chromatography based on heart-cutting methodology, combining hydrophilic interaction liquid chromatography (HILIC) in the first dimension (1D) and reversed-phase chromatography (RP) in the second dimension (2D). The aim of this study was to transfer in a single cut the void volume from the HILIC separation (non-polar pesticides) to the 2D for analysis under RPLC conditions, allowing a simultaneous visualization of the 1D and 2D contents in a single analysis. The coupling between both dimensions was achieved by a multiple heart-cutting (MHC) interface equipped with an ASM (Active Solvent Modulation) valve. However, the main disadvantage of this interface is that the algorithm that controls the MHC system starts the 2D analysis as soon as possible, meaning that only the fractions transferred to the 2D will be analysed while the rest of the information from the 1D effluent will be lost [3]. Consequently, to avoid the loss of information and to be able to analyse both dimensions in a single run, the coupling of a column selection valve to the MHC system is proposed. Hence, simultaneous analysis of polar and multiresidue-type pesticides can be achieved with the advantage of acquiring all data in a single file without resorting to additional software and with no sophisticated instrumentation, just by using a column selection valve.

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2º Premio a la mejor Comunicación Oral (Y-OMI-01)

FINGERPRINTING BY GAS CHROMATOGRAPHY AND HIGH-RESOLUTION MASS SPECTROMETRY (GC-ORBITRAP-HRMS): A PROMISING TOOL FOR ORIGIN AND PROCESSING AUTHENTICATION OF THYME

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Thyme is one of the most widely consumed aromatic herbs in Europe due to its organoleptic and medicinal properties [1]. Intentional mislabelling and food fraud activities are increasingly occurring in thyme as a result of its high economic value and the complexity of the supply chains of the condiment industry. Bearing in mind the lack of current methods focused on thyme quality control, assessing its authenticity and traceability in terms of geographical origin and post-harvest processing has become a current challenging task. In this context, this study presents a novel metabolomics approach based on fingerprinting by gas chromatography (GC) coupled with advanced high-resolution mass spectrometry (HRMS) detection for reliable discrimination of thyme samples according to their geographical origin (Spain, Morocco, and Poland) and processing practices (sterilized vs. non-sterilized thyme) [2]. In this study, multivariate data analysis was carried out using supervised statistical models. They provided reliable sample clustering according to the tested classes. High-quality model parameters (R2 and Q2 values > 0.97) and high predictive ability for further samples were achieved (correct classification rate of 100%). Furthermore, this untargeted approach led to the identification of 24 key volatile metabolites (13 metabolites were confirmed) with high discriminant potential, which may be used as marker compounds for origin and processing traceability of the samples, including monoterpenoids, diterpenoids, sesquiterpenoids, alkenylbenzenes, among others. The findings highlighted the impact of the region of production and the post-harvest processing on the metabolomic composition of thyme. Consequently, these metabolomic data may be exploited for the authentication of the product. This study encourages the implementation of this metabolomics workflow as a powerful tool for the authenticity assessment of other high-value condiments.

Acknowledgments

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1^{er} Premio al mejor Póster (P-FUN-04)

ENANTIOMERIC SEPARATION OF DRUGS BY NANO-LIQUID CHROMATOGRAPHY USING A CHIRAL COLUMN OF AMYLOSE TRIS(3-CHLORO-5-METHYLPHENYL CARBAMATE). APPLICATION OF A LIQUID-LIQUID MICROEXTRACTION SYSTEM TO WATER SAMPLES

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In recent years, the development of innovative analytical strategies for the enantiomeric separation of chiral compounds has been attracting the scientific attention due to the different properties that each enantiomer could exhibit. In fact, nano-Liquid Chromatography (nano-LC), combining the advantages of working with a LC system and using the capillary column, is a great separation technique for both fast studies on enantiomeric analysis and test beds of novel stationary phases. In addition, the sample pre-treatment is another point due to the present need of establishing simple, cheap, and environmentally sustainable procedures. In this work, the enantioseparation of different drugs was carried out by nano-LC with a chiral polysaccharide-based column. Specifically, it was evaluated the effect of different salts added to the mobile phase (consisting of ACN/water or MeOH/water, 9:1 v/v, at pH 7.0 and 10.0) on the chiral separation of 10 drugs from different families (alprenolol, oxprenolol, propranolol, metoprolol, mianserin, tolperisone, venlafaxine, lorazepam, oxazepam and temazepam). In general, using a MeOH-containing mobile phase at pH 10.0, a greater number of enantiomers were separated. Subsequently, the effect of the concentration of ammonium carbonate in the MeOH/water (9:1 v/v, pH 10.0) mobile phase from 10 to 75 mM was studied with the aim of simultaneously separate as many enantiomers as possible. Also, a liquid-liquid microextraction system (using isoamyl acetate as the green solvent) was performed to isolate alprenolol, mianserin, tolperisone, and temazepam from real samples of environmental interest (tap water and Tiber River in Rome). Good recoveries (>70%) were achieved for most of the drugs analyzed, except for tolperisone for which low recoveries (~40%) were obtained.

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2º Premio al mejor Póster (P-FA-05)

OPTIMIZATION OF A GREEN EXTRACTION PROCEDURE TO OBTAIN POLYPHENOLIC COMPOUNDS FROM THE WINE INDUSTRY BY-PRODUCTS

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EU agri-food export trade has been led in the last decade by the wine sector, being one of the most important agro-industrial activities in the world[1]. However, more than 5 million tonnes of marc, the main by-product of this industry, are generated each year. Several studies have addressed the reuse of this derivative based on its bioactive power represented by its high polyphenolic content, generating small doses of extract with potential incorporation in the food industry, cosmetics, and its reincorporation in the wine sector[2]. The main objective of this work is focused on the evaluation and optimization of a scalable process with minimum energy requirements MSAT (Medium Scale Ambient Temperature) for obtaining polyphenolic extracts from the white grape marc employing generally recognized as safe (GRAS) solvents including propylene glycol (Pg), ethanol (EtOH) and ethyl lactate (EtLc), as well as their hydro-organic mixtures. In a first approach, through a response surface matrix, the operational parameters, extractive volume, marc mass and its ratio with a dispersant were optimized, looking for an efficient process able to generate higher volumes of extract and bioactivity. In this way, the highest total polyphenolic content (5918 mgGAE·L⁻¹) and antioxidant activity (44 mMTE) values were achieved at a maximum operational volume of 100mL. On the other hand, to obtain an extract suitable for nutraceutical purposes, the affinity profile towards the main polyphenolic compounds and carbohydrates was explored by HPLC-MS/MS and UHPLC-QTOF, respectively. The overall response of the bioactive activity as well as the individual phenolic profile was obtained by using EtLc>EtOH>Pg, whereas the isovolumetric mixture EtLc/water showed the highest concentrations for the polyphenols: quercetin (5.4 mg·L⁻¹), quercetin-3-glucuronide (22.4 mg·L⁻¹), kaempferol (1.0 mg·L⁻¹) and quercetin-3-glucoside (26.0 mg·L⁻¹) together with a lower concentration of reducing sugars, favouring their potential use of the extracts in a solid formulation.

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