XX EDICIÓN DE LOS PREMIOS JOSÉ ANTONIO GARCÍA DOMÍNGUEZ

Durante la ceremonia de clausura del congreso internacional Euroanalysis 2025, que enmarcó la XXIV Reunión Científica de la SECyTA, celebrado en Barcelona del 31 de agosto al 4 de septiembre de 2025, se hizo entrega de los Premios José Antonio García Domínguez en su vigésima edición.

Como en ocasiones anteriores, estos premios, patrocinados por Bruker, reconocen las dos mejores comunicaciones orales y los dos mejores pósteres presentados por jóvenes investigadores miembros de la Sociedad.

Tras la deliberación del jurado, a quien expresamos nuestro agradecimiento por su colaboración, se acordó otorgar los premios que se detallan a continuación:

1er Premio a la mejor comunicación oral (800 €)

EFFICIENT MULTI-CLASS DETERMINATION OF MYCOTOXINS AND PESTICIDE RESIDUES IN FISH FEED AND BEEKEEPING PRODUCTS USING UAE-QuEChERS-BASED EXTRACTION AND LC-MS

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Mycotoxins and pesticides in food and animal feed pose health risks. Aquafeeds increasingly use plant-based ingredients, raising contamination concerns. Maximum levels for certain mycotoxins are regulated under Recommendation (EU) 576/2006 [1], and pesticide residues by Regulation (EC) 396/2005 [2] in fish feed. Bee pollen and honey offer nutritional benefits but are prone to contamination. Regulation (EC) 396/2005 [2] sets màximum residue limits of pesticides in honey, but no mycotoxin limits exist. Moreover, no legislation regulates those contaminants in bee pollen. Implementing rigorous quality control and promoting sustainable beekeeping practices are essential to ensure product safety and quality.

This work proposes a multi-analyte method for the determination of 28 compounds, including 10 pesticides and 20 mycotoxins, in fish feed, bee pollen, and honey. Sample treatment was optimized for each matrix using ultrasoundassisted extraction (UAE) combined with a QuEChERS approach using acetonitrile as the extractant. For fish feed, a dispersive solid-phase extraction (dSPE) cleanup using PSA and C18 sorbents was necessary. For beekeeping products, extraction included n-hexane and an additional re-extraction step for bee pollen; dSPE cleanup was not required in these matrices.

Liquid chromatography (LC) separation was performed using a Hypersil GOLD aQ column (100 × 2.1 mm, 1.9 µm). Detection was performed using Q-Exactive Orbitrap-MS for fish feed and triple quadrupole (QqQ)-MS for beekeeping products. Methods validations were carried out according to the SANTE/11312/2021 guideline [3], with determination coefficients above 0.99 for matrix-matched calibration curves of all compounds in fish feed.

Procedural calibration curves were evaluated for honey and pollen using weighted least squares (WLS) regression to correct heteroscedasticity, achieving good linearity. Limits of quantification (LOQs) ranged as follows: for fish feed, from 0.06 µg/kg (Pirimiphos-methyl) to 100 µg/kg (Ochratoxin α); for honey, from 0.11 µg/kg (ENNA1) to 31.3 µg/kg (3-AcDON); and for bee pollen, from 1.30 µg/kg (ENNA) to 289 µg/kg (DON). Trueness was between 70 and 120% for

most analytes, and satisfactory precision (RSDs < 20%) was achieved in all cases.

The validated method was applied to 36 fish feed samples. Among pesticide residues, pirimiphos-methyl was the most frequently detected (75%). Among mycotoxins, enniatin B and B1 were the most prevalent (81%). Non-targeted analysis also revealed the presence of piperonyl butoxide in 81% of samples. Ochratoxin α was detected at the highest concentration (202 µg/kg). A total of 28 honey and six pollen samples were analyzed. In honey, acetamiprid and azoxystrobin were detected in 29% and 32% of samples, with maximum concentrations of 16.2 µg/kg and 1.57 µg/kg, respectively. No pesticide residues or mycotoxins were found in bee pollen. Importantly, none of the detected contaminant concentrations exceeded the maximum limits established by EU regulations.

Acknowledgements

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2º Premio a la mejor comunicación oral (600 €)

MICROWAVE-ASSISTED EXTRACTION OF PROTEINS FROM CITRUS PEELS. A COMPARATIVE STUDY OF DIFFERENT PROTEASES TO OBTAIN BIOACTIVE HYDROLYSATES

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The agri-food industry is responsible for the generation of a substantial amount of waste during production processes. That waste may contain high-value bioactive compounds that, if properly utilized, can significantly contribute to circular economy and to the sustainability of the food chain. The use of food residues as sustainable sources of value-added products is a growing area of interest, in response to the global demand for environmentally friendly practices in the food industry. Residues from fruits and vegetables, such as citrus peels, are often discarded despite their potential to provide bioactive compounds (proteins, antioxidants, and other functional

compounds). The valorization of these residues through green techniques, such as microwave-assisted extraction (MAE), enables to obtain compounds with applications in various fields, including food, pharmaceutical, and cosmetic industries. In the case of protein extraction, MAE offers advantages in terms of efficiency and reduced environmental impact when compared with the conventional techniques usually employed for this purpose [1].

The aim of this work was to develop an analytical methodology based on MAE for the sustainable extraction of proteins from citrus peels (lemon and mandarin) and to evaluate different protease enzymes to obtain bioactive peptides from protein extracts. A Box-Behnken experimental design was employed to determine the most favorable conditions (percentage of ethanol, temperature, and time) for the extraction of proteins. The optimal conditions were established by maximising the protein content and minimising the formation of Maillard compounds. The protein extracts obtained were then characterized and hydrolysed to obtain bioactive peptides. The experimental conditions used for different protease enzymes were optimized and their potential to obtain bioactive peptides was compared. The peptide content and degree of hydrolysis were determined in the hydrolysates, and diferent bioactivities (antioxidant, antihypertensive, and antimicrobial) were evaluated. Finally, most active hydrolysates were analysed by UHPLC-MS/MS for peptide identification.

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1er Premio al mejor póster (400 €)

ADVANCING IN ANTIBIOTIC ANALYSIS: A NOVEL µSPEed MICROEXTRACTION COMBINED WITH UHPLC-PDA FOR MULTI-RESIDUE ANALYSIS IN ENVIRONMENTAL SAMPLE

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The overuse of antibiotics in animal farming, driven by the increasing demand for animal-based products, has raised concerns regarding the presence of antibiotic residues in food and the environment [1]. Residues of commonly used antibiotics in the environment, such as sulfonamides, tetracyclines, and penicillins, can contribute to antibiòtic resistance, disturb gut bacteria, and cause allergic reactions or toxicity [2,3]. To address these issues, it is crucial to apply stricter regulations and use analytical methodologies that allow the detection of low

concentrations that may exist in the environment. Although traditional extraction techniques such as Solid-Liquid Extraction (SLE), Solid-Phase Extraction (SPE), and Liquid-Liquid Extraction (LLE) have been used to extract and pre-concentrate veterinary antibiotic residues prior to their analysis, there is a growing need for more sustainable and efficient extraction methods based on the miniaturization of these techniques, following green chemical principles [4].

In this work, an analytical methodology was developed based on a semi-automated micro-solid phase extraction (µSPEed) combined with UHPLC-PDA analysis for the determination of six antibiotics — tetracycline, chlortetracycline, oxytetracycline, doxycycline, sulfamethoxazole, and trimethoprim — in wastewater samples. The extraction process was optimized by assessing different sorbent materials (butyl silica (C4), octyl silica (C8), octadecyl silica (C18-RPS), octadecyl silica hydrophilic ODS (C18-ODS), unmodified silica (Sil), aminopropyl silane sorbent (WAX), polyfluoroalkyl substances (PFAS), porous PS/DVB normal phase (PS/DVB), porous PS/DVB reversed phase (PS/DVB-RP), PS/DVB anionic exchange (PS/DVB-SAX), PS/DVB cationic exchange (PS/DVB-SCX), and porous graphitic carbon (µCARB)), sample volumes (500 and 800 µL), sample loading cycles (3-5), pH conditions (2.5, 4.0, 7.0, and 8.5), and elution solvents (methanol, acetonitrile, and acidic methanol (with 5% and 1% formic acid)) to achieve maximum efficiency. The best performance was obtained using a PS/DVB-RP sorbent with three 250 µL loading cycles and two consecutive elutions with 50 µL of acidified methanol (0.1% formic acid). The developed methodology enabled a rapid analysis with a total chromatographic run time of just six min. The validation results confirmed a good linearity, precision, and satisfactory recovery. Detection limits ranged from 0.30 to 1.23 µg L^{-1} and quantification limits from 0.92 to 3.73 µg L^{-1}. The developed µSPEed/UHPLC-DAD methodology proved to be effective for the analysis of wastewater samples, offering a reliable alternative for controlling antibiotics in environmental samples.

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2º Premio al mejor póster (300 €)

VOLATILOMICS FOR THE AUTHENTICATION OF POMEGRANATE JUICE USING GAS CHROMATOGRAPHY COUPLED TO ION MOBILITY SPECTROMETRY

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Pomegranate (*Punica granatum* L.) is considered a luxury fruit, whose juice has well-known healthy benefits, particularly a high antioxidant activity. Its constantly increasing demand always

surpasses the production, resulting in an elevated price. This context makes this juice vulnerable to adulteration. This practice can consist of diluting with cheaper juices like apple and grape juice what allows to increase the profits. Other reported strategy is to add dark juices like black grape or blueberry juices to mask poor-quality juices and to imitate the colour of pomegranate juice. [1-3] In order to detect the adulteration and ensure the authenticity of this product, food industry measures many physicochemical parameters with several instruments, which is expensive and time-consuming. Therefore, an analytical method to control the quality with a single measurement is highly desired. Besides, almost all the reported methodologies are based on the determination of polyphenols, anthocyanins or sugars present in the pomegranate juice and, in this context, the volatile organic compounds (VOCs) profile of this food remains underused. With the aim of studying the applicability of these analytes for the quality control of pomegranate juice, a methodology based on the analysis of VOCs profile obtained by headspace gas chromatography coupled to ion mobility spectrometry (HS-GC-IMS) was developed and validated.

Spectra of 176 genuine pomegranate juices and 120 different mixtures of these pure juices with adulterants were analyzed in a non-targeted way. The mixtures were prepared with seven apple juices and three black grape juices (levels from 1 to 40%) and two blueberry juices (levels from 0.1 and 10%). All collected data were used for the construction of chemometric models resulting in the next strategy. Firstly, a model based on orthogonal partial least squares discriminant analysis (OPLS-DA) was built to classify the sample into pure or adulterated pomegranate juice with 100% validation success rate. If the sample is considered adulterated, knowing the illegally added juice could be of interest. For this reason, another OPLS-DA model was developed to distinguish between the three adulterants studied with 87.5% validation success rate. The incorrectly classified juices correspond to low levels of adulterants (0.1 and 0.5%). Finally, three partial least squares (PLS) regressions were constructed to quantify the percentage of adulterant in the juice. This strategy was successfully applied to seven commercial juices. The classification of these samples corresponds with the declared ingredients in the labels in six out of the seven cases.

In this work, the potential of VOCs for the authentication of pomegranate juice was demonstrated with a chemometric model able to detect until 1% of adulteration with apple and black grape juice and until 0.1% with blueberry juice. In addition, the proposed methodology can distinguish between these three adulterants and quantify their levels in the samples. All this information on a sample can be obtained with a single HS-GC-IMS measurement. This technique has many advantages, such as low operation cost, high sensitivity and it is more eco-friendly than GC-Mass Spectrometry because the IMS spectrometer works at atmospheric pressure. [4] The excellent results obtained together with the minimal sample treatment, the environmental sustainability provided by IMS and the short analysis time (16 min) make the proposed methodology a powerful tool for use in the quality control of pomegranate juice.

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