Plan Overview

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Title: Solventes e Técnicas de Extração Sustentáveis em Química Analítica Verde para Determinação de Contaminantes Emergentes e Inorgânicos em Amostras Ambientais

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Project abstract:

There is an increase in the occurrence of several substances used worldwide that represent an imminent or potential risk to human and environmental life, as a result of this risk, they have been indicated as emerging contaminants (EC). Consequently, there are intensified efforts to develop techniques for the analysis of ECs, based on the principles of green analytical chemistry (GAC), including the miniaturization of methods, reduced consumption of reagents and energy, and avoidance of the use of toxic compounds. The development of green solvents provides an example of the production of new sustainable materials based on the principles of GAC. The present research project proposes the synthesis of natural deep eutectic solvents (NADES) and amino acid-based deep eutectic solvents (AADES) with the components β-alanine, tryptophan, carboxylic acids (malic acid and citric acid), and carbohydrate (xylitol), as well as the synthesis of hydrophobic deep eutectic solvents (HDES) based on DL-menthol, acetic acid, and lactic acid, modified with magnetic Fe3O4 nanoparticles (NPs). Three synthesis methods will be evaluated: controlled heating and stirring, ultrasound-assisted synthesis (UAS), and microwave-assisted synthesis (MAS). Characterization techniques employed to investigate the formation of these new solvents will include infrared spectroscopy (IR), thermogravimetry (TG), and differential scanning calorimetry (DSC), together with polarity, density, and viscosity measurements. These new solvents will be evaluated as extraction solvents in green sample preparation methods for use in environmental monitoring of ECs belonging to the classes of pharmaceuticals and brominated flame retardants, present in samples of surface water, industrial effluent, and sewage sludge. These analytes will be determined by gas chromatography coupled to mass spectrometry (GC-MS). In addition, the elements As, Cd, Cr, Hg, Se, and V will be analyzed by inductively coupled plasma mass spectrometry (ICP-MS). These analyses will be accompanied by

assessment of the possible interferences inherent to the techniques. Finally, metric tools will be used to evaluate the solvent synthesis processes and the analyte extraction methods, considering their green characteristics and compliance with environmental criteria

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Solventes e Técnicas de Extração Sustentáveis em Química Analítica Verde para Determinação de Contaminantes Emergentes e Inorgânicos em Amostras Ambientais

Serão coletados e criados dados qualitativos e quantitativos sobre a presença de contaminantes emergentes e inorgânicos em matrizes ambientais, além de gráficos, cromatogramas, espectros, imagens em formato .jpg e outros documentos associados ao estudo de desenvolvimento e aplicação de métodos analíticos sustentáveis.

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Todos os dados gerados desde a coleta da amostra, obtenção dos resultados analíticos nos laboratórios (massas, volumes, diluições – registradas em caderno Ata, e logbooks para rastreabilidade), dados brutos instrumentais (balanças, equipamentos de grade porte, e outros) serão registrados após a realização dos experimentos. Os diários/cadernos de laboratório e logbooks serão armazenados no laboratório, devendo ser retirados apenas para contribuir nos tratamentos de dados e redação de materiais científicos, sendo armazenados e mantidos por um período de 5 anos após a publicação dos resultados.

Serão seguidos as questões éticas e de todos direitos autorais de acordo coma as normas da UNESP e da FAPESP.

Os artigos e trabalhos apresentados em congressos terão como autores somente os envolvidos em sua produção estabelecendo a organização dos mesmos de acordo com a conduta ética científica, e todos direitos autorais respeitarão as normas da UNESP e da FAPESP.

Serão armazenados dados brutos coletados, tratamentos de dados em geral (planilhas Excel), imagens e figuras obtidas e geradas. Os dados serão armazenados em pastas nomeadas de acordo com as técnicas envolvidas e fases da pesquisam salvos em nuvem e em HD externos.

Todos os dados gerados desde a coleta da amostra, obtenção dos resultados analíticos nos laboratórios (massas, volumes, diluições – registradas em caderno Ata, e logbooks para rastreabilidade), dados brutos instrumentais (balanças, equipamentos de grade porte, e outros) serão registrados após a realização dos experimentos. Os diários/cadernos de laboratório e logbooks serão armazenados no laboratório, devendo ser retirados apenas para contribuir nos tratamentos de dados e redação de materiais científicos, sendo armazenados e mantidos por um período de 5 anos após a publicação dos resultados.

Dados brutos, tratamento de dados em geral (planilhas Excel), imagens e figuras obtidas e geradas.

Os dados de pesquisa gerados serão preservados na nuvem em contas Google Drive e Dropbox, sob supervisão do proponente, onde serão armazenados dados brutos coletados, tratamento de dados em geral (planilhas Excel), imagens e figuras obtidas e geradas. Os dados serão também armazenados em pasta protegida por senha no computador pessoal do pesquisador, e o backup será feito a cada atualização da análise dos dados. Em todos os casos, a FAPESP será mencionada como órgão financiador, além de constar como coproprietária da patente, de acordo com normas vigentes. Os dados obtidos serão mantidos sob sigilo do proponente responsável pelo projeto de pesquisa, até garantia de publicação dos metadados

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Os dados estarão disponíveis para reutilização por terceiros após a publicação dos resultados da pesquisa. Não serão aplicadas restrições à reutilização dos dados desde que respeitem as finalidades científicas e a ética na pesquisa e citações corretas

O proponente Prof. Dr. Mario Henrique Gonzalez

O cumprimento de todos os recursos envolvidos durante a execução do projeto.

Planned Research Outputs

Data paper - "Greenness of procedures using NADES in the preparation of vegetal samples: Comparison of five green metrics"

The principles of green analytical chemistry have led to the development of analytical procedures that are increasingly sustainable. Different metrics have been created for the evaluation of greenness, although determination
of the green nature of new analytical methods remains challenging, including for extraction methods
that involve the use of natural deep eutectic solvents (NADES). In this study, the following five chemical metrics for the evaluation of greenness were considered: National Environmental Methods Index (NEMI), Green Analytical Procedure Index (GAPI), Analytical Eco-Scale, Analytical GREEnness (AGREE), and White Analytical Chemistry (WAC). These methods were applied in evaluation of the environmental and sustainability characteristics
of three different methods for the preparation of plant material samples: microwave-assisted extraction (MAE), ultrasound-assisted extraction (UAE), and microwave-assisted acid digestion (MW-AD). These methods employed different NADES as extraction solvents, as well as dilute nitric acid as an oxidizing agent, for the determination of As, Cd, Pb, and V by inductively coupled plasma mass spectrometry (ICP-MS). The NEMI metric found no differences between the MAE-NADES and UAE-NADES methods. The GAPI metric found differences between the MAE-NADES and UAE-NADES methods and identify the disadvantageous aspects of each step of the methods. The Analytical Eco-Scale and AGREE identified the MAE-NADES method as the greenest, while WAC-12
RGB indicated the UAE-NADES method as the greenest procedure. A detailed discussion is provided of the application of each metric, together with their differences and advantages

Data paper - "Green approaches with amino acids-based deep eutectic solvents (AADES) for determining As in medicinal herbs by ICP-MS"

In this work, an innovative ultrasound-assisted solid-phase matrix dispersion (UA-MSPD) and microwave-assisted extraction (MAE) were applied with amino acids-based deep eutectic solvents (AADES) for the extraction of arsenic (As) from medicinal herbs. Multivariate optimization by Doehlert design (DD) was performed to determine the optimal experimental conditions. The effects of temperature (TP), time (TM), and sample-solvent ratio (SSR) were evaluated, and the optimized conditions were 50 °C, 60 min, and 10:1 mg mL-1 for UA-MSPD and 100 °C, 40 min, and 40:1 mg mL-1 for MAE AADES 2 (β-alanine, citric acid and water), with the hydroxyl and carboxyl groups of the citric acid structure favoring formation of a chelate complex with the analyte. AADES 3 (β-alanine, xylitol and water) was effective for MAE, while AADES 1 (β-alanine, malic acid and water) proved to be inefficient for As extraction The parameters of the analytical methods were evaluated using certified reference materials. The accuracy, based on percentage recovery, was in the range 77 – 101%, while the limits of detection and quantification were in the ranges 0.010 – 0.039 mg kg-1 and 0.011 – 0.130 mg kg-1, respectively. The analytical curves presented $R^2 > 0.99$. The proposed methods were shown to be environmentally friendly, based on the Analytical Eco-Scale and RGB 12 procedures. Both optimized methods were applied for the determination of As in commercial medicinal herbs (0.059 – 0.101 mg kg-1), with the values obtained being within the maximum daily intake limit established by the World Health Organization (WHO). It should be noted that there are no previous reports in the literature concerning the application of a sample preparation method using AADES employing their solid precursors, with no requirement for prior solvent synthesis, as proposed here in the case of the UA-MSPD method.

Planned research output details

Title	Type	Anticipated release date	Initial access level	Intended repository(ies)	Anticipated file size	License	Metadata standard(s)	May contain sensitive data?	May contain PII?
Greenness of procedures using NADES in the prepara ...	Data paper	Unspecified	Open	None specified		None specified	None specified	No	No
Green approaches with amino acids-based deep eutec ...	Data paper	Unspecified	Open	None specified		None specified	None specified	No	No