CONFERENCE PROCEEDINGS

"5th international congress on Green Extraction of Natural Products (GENP2024)"

GENP 2024

The 5th International Congress on

Green Extraction of Natural Products

October 28-30, 2024 • Compiègne, France

Sorbonne Université | utc

BOOK OF ABSTRACTS

October 28-30, 2024 • Compiègne, France.

GENP2024

The 5th International Congress on Green Extraction of Natural Products

October 28-30, 2024 • Compiègne, France

INDUSTRIAL SPONSORS

Platinum Sponsor

Chaire Unesco
CHIMIE VERTE & DURABLE DU VÉGÉTAL

Gold Sponsors

elea

Maguin

Silver Sponsors

LESAFFRE

Celsius

MILESTONE
HELPING CHEMISTS

indena
SCIENCE IS OUR NATURE. SINCE 1921
100+ YEARS

basis
ÉLECTRONIQUE DE PUISSANCE

De Dietrich
PROCESS SYSTEMS
Innovation · Passion · Solutions

pulsemaster

Supported / co-organized by :

ALLIANCE SORBONNE UNIVERSITÉ

utc Recherche
TIMR

Région Hauts-de-France

AVA chimie
escom

CHAIR

Pr. Nabil GRIMI

University of Technology of Compiegne (UTC)
Laboratoire Transformations Intégrées de la Matière Renouvelable (TIMR)
Équipe « Technologies Agro-Industrielles »
Compiègne, France

INTERNATIONAL SCIENTIFIC COMMITTEE

- Giancarlo Cravotto (Turin, Italy)
- Mladen Brnčić (Zagreb, Croatia)
- Francisco Barba (Valencia, Spain)
- Antonio J. Meléndez-Martínez (Sevilla, Spain)
- Anne Sylvie Tixier (Avignon, France)
- Elisabeth Badens (Marseille, France)
- Filomena Corbo (Bari, Italy)
- Marija Badanjak Sabolovic (Zagreb, Croatia)
- Mounir El Achaby (Bengérir, Maroc)
- Amine Moubarik (Beni Mellal, Maroc)
- Nadia Boussetta (Compiègne, France)
- Nabil Grimi (Compiègne, France)
- Anissa Khelfa (Compiègne, France)
- Paula Mapelli Brahm (Sevilla, Spain)
- Mohamed Koubaa (Compiègne, France)
- Nikolai Lebovka (Kiev, Ukraine)
- Houcine Mhemdi (Compiègne, France)
- Maryline Vian (Avignon, France)
- Eugène Vorobiev (Compiègne, France)

LOCAL ORGANISING COMMITTEE – MEMBERS

- Nadia Boussetta, TIMR, UTC-ESCOM, Compiègne, France
- Nabil Grimi, TIMR, UTC-ESCOM, Compiègne, France
- Anissa Khelfa, TIMR, UTC-ESCOM, Compiègne, France
- Mohamed Koubaa, TIMR, UTC-ESCOM, Compiègne, France
- Houcine Mhemdi, TIMR, UTC-ESCOM, Compiègne, France
- Eugène Vorobiev, TIMR, UTC-ESCOM, Compiègne, France

Editors :

Nabil GRIMI

Mohamed KOUBAA

ISBN :

978-2-913923-39-3

EAN :

9782913923393

TABLE OF CONTENT

WELCOME MESSAGE

Dear colleagues,

On behalf of the Organizing and Scientific Committees, it is our great honor and privilege to welcome you to Compiègne, France, for the 5th edition of the International Conference on Green Extraction of Natural Products (GENP2024), taking place from October 28-30, 2024.

We are delighted to present an engaging and comprehensive scientific program, meticulously prepared with the support of the Scientific Committee of GENP2024. The program features lectures from renowned international experts and young scientists in green extraction. This event is designed to provide a solid foundation of knowledge, hands-on skills, and a deeper understanding of the principles and processes involved in green extraction technologies, particularly within the food industry and biorefineries.

The primary goal of this conference is to foster meaningful dialogue and collaboration among experts from academia, research institutions, and industry. We aim to explore advancements in cultivation, extraction, processing, and recycling, with applications across multiple sectors, including agrifood, nutraceuticals, perfumes, cosmetics, chemicals, fuels, and energy.

The congress is organized into four topics:

- **Alternative solvents for green extraction**
- **Sustainable and clean extraction technologies**
- **Valorization of by-products and biorefineries**
- **Innovative technologies for separation and fractionation processes**

We extend our warm thanks to all participants for their presence and valuable contributions to the discussions and sessions. Your involvement is crucial to the success of GENP2024.

Additionally, we express our deep gratitude to our sponsors and exhibitors for their generous support and for recognizing the importance of this event.

We would also like to acknowledge the hard work and commitment of the Scientific and Organizing Committees, whose efforts have been instrumental in making this conference a reality.

Once again, thank you for joining us, and we hope you have an enjoyable and productive stay in Compiègne.

Chair of the conference
Nabil GRIMI

SCIENTIFIC PROGRAM

October 28-30, 2024 • Compiègne, France.

DAY 1 : Monday, October 28, 2024

10:00	14:00	**REGISTRATION** Location : « Technology Transfer Center »
14:00	14:20	**Opening session** Location : « Technology Transfer Center »
Topic 1 : ALTERNATIVE SOLVENTS FOR GREEN EXTRACTION Conference location: amphitheater Colcombet « Technology transfert center »		
Chairs: Giancarlo CRAVOTTO and Antonela NINCEVIC GRASSINO		
14:20	15:05	**Plenary Lecture :** **(OR-01)** "Supercritical Fluid Technology: A sustainable and efficient way to extract and stabilize compounds of interest" **Speaker : Pr. BADENS Elisabeth**
15:05	15:20	**(OR-02)** "Simultaneous recovery of proteins and polyphenols from hemp seeds under subcritical water conditions" **Speaker : CHEMAT Aziadé**
15:20	15:35	**(OR-03)** "Natural dyes eco-extraction using supercritical CO2" **Speaker : SAVOIRE Raphaëlle**
15:35	15:50	**(OR-04)** "Optimization of extraction of polyphenols from Rhamnus alaternus by deep eutectic solvent-based ultrasound assisted extraction" **Speaker : NEKKAA Amine**
15:50	16:20	**Coffee break / Posters** Location: Technology transfert center
Chairs: Elisabeth BADENS and Anne-Sylvie TIXIER		
16:20	16:35	**(OR-05)** "Innovative fractionation process for brewer's spent grains upgrading" **Speaker : KHELFA Anissa**

16:35	16:50	**(OR-06)** "Sustainable valorisation of industrial hemp: novel technologies and a green solvent" **Speaker : CRAVOTTO Christian**
16:50	17:05	**(OR-07)** "Extraction of Bioactive Molecules from Apple Pomace: Evaluation of Antioxidant and Antibacterial activities" **Speaker : BRUNA Lauriane**
17:05	17:20	**(OR-08)** "Evaluation of green and bio-based solvents for the extraction of ß carotene and lipids from the oleaginous yeast R. toruloides" **Speaker : BUCHWEITZ Vanessa**
17:20	17:35	**(OR-09)** "DES-milling combined extraction process targeting pectin from citrus peels and apple pomace" **Speaker : PERRET Lucie**
17:35	17:50	**(OR-10)** "Cascade extraction of polyphenols and carotenoids, using Natural Deep Eutectic Solvents" **Speaker : SAUVETES Paul**
17:50	18:05	**(OR-11)** "Green solutions to extract bioactive compounds from vitro-derived hop plantlets treated with elicitors" **Speaker : LETO Leandra**
18:05	18:20	**(OR-12)** "Efficacy of the bio-solvent 2-methyltetrahydrofuran for microwave-assisted extraction of carotenoids from Chlorella vulgaris. Comparison with other traditional organic solvents." **Speaker : MORON-ORTIZ Angeles**

DAY 2 : Tuesday, October 29, 2024

8:00	8:45	**REGISTRATION** **Location : « Technology Transfer Center »**
Topic 2 : SUSTAINABLE AND CLEAN EXTRACTION TECHNOLOGIES Conference location: amphitheater Colcombet « Technology transfert center »		
Chairs: Nabil GRIMI and Antonio J. MELÉNDEZ-MARTÍNEZ		
8:45	9:30	**Plenary Lecture :** **(OR-13)** " Pulsed electric energy treatments to enhance extraction of natural products" **Speaker : Pr. VOROBIEV Eugène**
9:30	9:45	**(OR-14)** "Ultrasound- and PEF-assisted Industrial Production of EVOO with higher content of Bioactive Compounds" **Speaker : BOFFA Luisa**
9:45	10:00	**(OR-15)** "Green Extraction Technologies Driving Circular Economy in the Sicilian Prickly Pear Industry" **Speaker : CAPALDI Giorgio**
10:00	10:15	**(OR-16)** "Microwave assisted hydrodistillation of hop terpenes: An application in the brewing industry" **Speaker : CARNAROGLIO Diego**
10:15	10:45	**Coffee break / Posters** Location: Technology transfert center
Chairs: Nikolai LEBOVKA and Oleksii PARNIAKOV		
10:45	11:00	**(OR-17)** "Application of DIC technology for the extraction of high added value compounds from Cannabis Sativa L. buds according to the bio-refinery concept" **Speaker : BOUCHEKIOUA Dounia**
11:00	11:15	**(OR-18)** "Alternative recovery of natural acetoin by hybrid pervaporation / distillation process" **Speaker : PERNAK Anne-Sophie**

11:15	11:30	**(OR-19)** "Synergistic Extraction of Bioactive Compounds using Enzyme-Assisted Extraction (EAE) in Combination with High-Pressure Homogenization (HPH)" **Speaker : MOJARRADI Fatemeh**
11:30	11:45	**(OR-20)** "Optimization of enzyme-assisted extraction of bioactive compounds from the pseudo-fruit of Rosa Canina L. and evaluation of the biological potential of the optimum extract" **Speaker : LEMONI Zafeiria**
11:45	12:00	**(OR-21)** "Multi-criteria optimization including environmental impacts of ultrasound-assisted extraction of phenolic antioxidants from blackcurrant pomace by-product" **Speaker : KHALAF Doha**
12:00	13:15	**Lunch** Location : Innovation Center
Topic 3 : VALORISATION OF BY PRODUCTS AND BIOREFINERY Conference location: amphitheater Colcombet « Technology transfert center »		
Chairs: Mohamed KOUBAA and Marija BADANJAK SABOLOVIC		
13:15	14:00	**Plenary Lecture :** **(OR-22)** "Advanced drying methods and the emerging intensification of drying in the valorization of pumpkin by-products " **Speaker : NINCEVIC GRASSINO Antonela**
14:00	14:15	**(OR-23)** "Impact of Time, Temperature, and pH on Hydrothermal Treatment for Hemicellulose Extraction from Fir Sawdust and Characterization by Capillary Electrophoresis" **Speaker : NAJJOUM Nicole**
14:15	14:30	**(OR-24)** "Optimisation of proteins extraction from tenebrio meliotor larvea" **Speaker : JABLAOUI Cherif**
14:30	14:45	**(OR-25)** "Economic and Environmental Impact Analysis of Innovative Cascaded Extraction of Lycopene and Cutin from Tomato Processing Residue" **Speaker : PATARO Gianpiero**
14:45	15:30	**Coffee break / Posters** Location: Technology transfert center

Session I : Innovative Approaches for Biomass Valorization: From Extraction to Sustainable Applications Conference location: amphitheater Colcombet « Technology transfert center »		
Chairs: Nadia BOUSSETTA and Gianpiero PATARO		
15:30	15:45	**(OR-26)** "Standardization of plant biomass and valorization of post-extraction residues by way of an innovative mobile pelletizing technology" **Speaker : COLLANGE Elea**
15:45	16:00	**(OR-27)** "Ultrasound-assisted green extraction of active compounds from citrus fruits: chemical composition and biological activity against skin-aging" **Speaker : EL KAHIA Houda**
16:00	16:15	**(OR-28)** "Enrichment of Refined Sunflower Oil with Phenolic Extracts from Argan (Argania spinosa L. (Skeels)) Co-products Using Ultrasound-Assisted Extraction" **Speaker : HALLOUCH Otmane**
16:15	16:30	**(OR-29)** "Unlocking the Potential of Enological By-products: A Cascade Approach for Comprehensive Biomass Valorization through Sustainable Technologies" **Speaker : CALCIO GAUDINO Emanuela**
16:30	16:45	**(OR-30)** "Kiwi Peel Valorization: Optimized Phenolic Content Extraction and Bioactivity Assessment" **Speaker : LOUKA Nicolas**
Session II : Advances in Lignin and Biomass Valorization: From Edible Films to High-Performance Materials Conference location: amphitheater « Innovation Center »		
Chairs: Erwann GUENIN and Anissa KHELFA		
15:30	15:45	**(OR-31)** "Evaluation of the influence of lignin nanoform on properties of edible films." **Speaker : GUÉNIN Erwann**
15:45	16:00	**(OR-32)** "Optimizing extracted lignin from deproteinated brewer's spent grains for chemical valorization" **Speaker : SALAMEH Sarah-Joe**

16:00	16:15	**(OR-33)** "Enzymes and green solvents for the sustainable extraction of lignin from horse manure: toward a diversification of the biomethane value-chain" **Speaker : DORSCHNER-PELCOQ Lindsay**
16:15	16:30	**(OR-34)** "How Far Can Catalysis Leverage The Transformation of Amorphous Biomass Into Crystalline Carbon-Based Materials?" **Speaker : MENNANI Mehdi**
16:30	16:45	**(OR-35)** "Innovative Conversion of Alfa Plant Cellulose Nanocrystals into Hard Carbon Anodes for Sodium-Ion Batteries" **Speaker : KASBAJI Meriem**
16:45	17:00	**(OR-36)** "Effect of phosphorylation treatment of lignocellulose obtained from Argan Shells on the mechanical, physical properties and formaldehyde emission of particleboard panels" **Speaker : MAARIR Hafida**
17:00	18:30	**Green Extraction Process (demonstration experiments) & Posters** Location : Innovation center and Technology transfert center
20:00	23:00	**Gala Dinner** Location : Château de Pierrefonds, rue Viollet le Duc, 60350 Pierrefonds

DAY 3 : Wednesday, October 30, 2024

Topic 4 : INNOVATIVE TECHNOLOGIES FOR SEPARATION AND FRACTIONATION PROCESSES Conference location: amphitheater Colcombet « Technology transfert center »		
Chairs: Eugène VOROBIEV and Marwen MOUSSA		
8:30	9:15	**Plenary Lecture :** **(OR-37)** " The pivotal role of natural preparations in the flavor industry" **Speaker : ALBERTINO Andrea**
9:15	9:30	**(OR-38)** "Green extraction coupling microwaves and centrifugation : trends and insights" **Speaker : GINISTY Pascal**
9:30	9:45	**(OR-39)** "Extractive fermentation of biobased compounds in the frame of bioeconomy" **Speaker : MOUSSA Marwen**
9:45	10:00	**(OR-40)** "Numerical simulation of membrane electroseparation of mixtures composed of particles of different sizes" **Speaker : LEBOVKA Nikolai**
10:00	10:15	**(OR-41)** "Optimizing stilbene recovery from cell culture media: A comprehensive study of the adsorption process" **Speaker : CHADNI Morad**
10:15	10:45	**Coffee break / Posters** Location: Technology transfert center
Chairs: Houcine MHEMDI and Morad CHADNI		
10:45	11:00	**(OR-42)** "Chestnut Wood Waste Valorization: Innovative Green Methods to Enhance Bioactive Fractionations" **Speaker : AIMONE Clelia**
11:00	11:15	**(OR-43)** "Overcoming the Shortfall in Green Metrics for Extraction Processes: The SIX Score Approach" **Speaker : GRILLO Giorgio**

11:15	11:30	**(OR-44)** "Assessment of the impact of treated wastewater on essential oil yield and agrophysiological responses of Rosmarinus officinalis." **Speaker : KAMMOUN Aya**
11:30	11:45	**(OR-45)** "Eradication of the invasive species Fallopia japonica and valorization of its rhizomes by extraction as a part of a complete value chain" **Speaker : COUPE Florentin**
11:45	12:00	**(OR-46)** "Computational Solvent Screening Using COSMO-RS Approach for Extracting Bioactive Compounds from Cocoa Bean Shells" **Speaker : BRAGAGNOLO Felipe Sanchez**
12:00	12:15	**(OR-47)** "From Black and Green Tea (Camellia sinensis) By-products to Potential Natural Health Boosters: Optimization of Polyphenol Extraction and Assessment of Antioxidant and Antibacterial Potentials" **Speaker : EL DARRA Nada**
12:15	13:30	**Lunch** Location : Innovation Center
13:30	14:00	**Awards** **& Closing Ceremony** Location: amphitheater Colcombet « Technology transfer center »

Poster Overview

Nr	Title	Presenting author
PO-01	A Tuneable process for the extraction and purification of chitosan from mealworm larvae	JABLAOUI Cherif
PO-02	Antioxidant activity, antimicrobial activity, and chemical profile of caraway (Carum carvi) essential oil obtained under different conditions	MILOSEVIC Sanja
PO-03	Application of subcritical water extraction for the production of high value components from agricultural byproducts : Comparison with conventional hydroalcoholic extraction	LEOONARD Kevin
PO-04	Aqueous extraction of clean label oleosomes from plant seeds	MOUSSA Marwen
PO-05	Centrifugal Partition Chromatography fractionation of Stilbenes and Anthraquinones from Japanese Knotweed (Reynoutria japonica) toward Nutraceuticals and Pharmaceutical Applications	HILALI Soukaina
PO-06	Comparison of chemical profile and bioactivity of dill essential oil obtained by conventional and innovative distillation techniques	BOZOVIC Danica
PO-07	Eco-Friendly Synthesis of Lignin Beads for Improved Adsorption of Methylene Blue and Methyl Orange	KASBAJI Meriem
PO-08	Eco extraction of valuable compounds from wood by supercritical CO2 process: influence of raw material and operating conditions	HARSCOAT-SCHIAVO Christelle
PO-09	Eco-extraction of biomolecules of interest from microorganisms by coupling of innovative technologies	MAHFOUD Sarah

PO-10	Exploring Artemisia Absinthium: Dual-Use Phytochemicals for Combating Cancer and Bacterial Resistance	LOUKMAN Salma
PO-11	Extraction and Analysis of Minerals and Heavy Metals from Black Truffle By-Products Using Non-Conventional Methods: Pressurised Liquid Extraction and Supercritical Fluid Extraction	MARTI-QUIJAL Francisco J.
PO-12	Recovery of Polyphenols from Black Truffle By-products Using Non-conventional Technologies: Characterization of Phenolic Profile by Triple-TOF-LC-MS-MS	MARTI-QUIJAL Francisco J.
PO-13	Extraction of mustard bran oil using supercritical CO2	DULLIER Cloé
PO-14	From waste to cosmeceuticals: buccal in situ gelling formulation including a polyphenols-enriched secondary raw material from green recovery of grape processing waste	BELFIORE Elena
PO-15	Xanthophyll-loaded nanoemulsions from plant matrices: enhancement of carotenoid bioavailability with a sustainable process	CONTINO-PEPIN Christine
PO-16	Macromolecular composition of Corchorus olitorius L.: toward the eco-extraction of hydrocolloids and other components	BONNOT Emma
PO-17	Optimization of Pulsed electric fields-assisted extraction parameters of lutein from Marigold (Tagetes erecta L.) Using response surface methodology	PATARO Gianpiero
PO-18	Lignin Phosphorylation for bio-based resin applications	EL KHAYAT DRIAA Yassine
PO-19	Optimizing Cellulose Phosphorylation for Enhanced Thermal Properties and Flame Retardance in Wood Adhesives	EL KHAYAT DRIAA Yassine
PO-20	Physically and chemically cross-linked hybrid hydrogels for the controlled extraction and release of medications	LEBOVKA Nikolai

PO-21	Subcritical Water Extraction of Bioactive Compounds from Red Ginseng Marc	SUH Dong Jin
PO-22	Supercritical CO2 extraction of √ü-carotene and lipids from the oleaginous yeast R. Toruloides	BUCHWEITZ Vanessa
PO-23	Synthesis and characterization of lignin nanoparticles	DENG Yanqin
PO-24	Unveiling the Multiscale Structural Dynamics and Retrogradation Behavior of Potato Starch via Integrated Enzymatic Hydrolysis Enhanced by Microwave	GRIMI Nabil
PO-25	Purification and concentration of C-Phycocyanin from spirulina extract by the combination of CaCl2 precipitation and membrane diafiltration technologies	GRIMI Nabil
PO-26	Cultivation of two psychrophilic microalgae Sphaerocystis sp. And Pleurastreum sp. For carotenoids extraction	GRIMI Nabil
PO-27	Valorization of Chitosan Extracted from Shrimp Shell Waste for Manufacturing of Particleboard Composites	GRIMI Nabil
PO-28	Valorization of Coffee Silverskin Using Extraction Cycles and Water as a Solvent: Design of Process.	CHEMAT Aziade
PO-29	Valorization of silk by-products for lipopeptide surfactant production using green technologies	WEHBE Lara
PO-30	Valorization of Wine By-Products for High Added-Value Extracts	KARASTERGIOU Anna
PO-31	The Protective Role of Exogenous Proline in Alleviating Oxidative Stress Induced by Heavy Metals in Sour Orange Plants (Citrus aurantium L.)	KARASTERGIOU Anna

PO-32	Valorization of forced chicory roots: extraction and thermochemical conversion	RHAYEM Lynn
PO-33	Influence of ohmic heating in the bioactive compounds of extracts from Iranian brown macroalgae (Nizimuddinia zanardini)	KOUBAA Mohamed
PO-34	Evaluation of physical properties of Ajwain (Trachyspermum Copticum (L.)) extract microcapsules prepared by Freeze Drying	KOUBAA Mohamed
PO-35	Stibenoids eco-extraction from vineyard coproducts : vitisin B extraction by ASE compare to Sc-CO2	SAVOIRE Raphaëlle
PO-36	Investigating a Straightforward Process Coupling Extraction and Membrane Separation for Hydroxytyrosol Recovery from "Picholine Marocaine" Cv. Olive Pomace	DHAIDHI Hamza
PO-37	Optimization of Bioactive Compound Extraction from Rosemary Solid Residues Using Box-Behnken Design	BARAR Anissa
PO-38	Optimization of Bioactive Compounds Extraction Assisted by Non-ionic Surfactant from Tetraclinis articulata Solid Residues Using Response Surface Methodology	BARAR Anissa
PO-39	High Voltage Electrical Discharges as a Pre-Treatment for Extraction of Bioactive Compounds from Solid Residues of Thuya	BARAR Anissa
PO-40	Valorization of sesame seed coat waste: phenolic composition, antibacterial efficacy, and nanoemulsion encapsulation for food preservation	EL DARRA Nada
PO-41	Removal of Contaminants from Water using Tomato Leaves as Adsorbent	EL DARRA Nada

DETAILED ABSTRACTS

Topic 1. Alternative solvents for green extraction

OR-01. Supercritical Fluid Technology: A sustainable and efficient way to extract and stabilize compounds of interest

Elisabeth BADENS [1,*], Adil MOUAHID [1], Yasmine MASMOUDI [1], Cristelle CRAMPON [1]

[1] Aix Marseille Univ, CNRS, Centrale Med, M2P2, Marseille, France

***Corresponding author:** Elisabeth BADENS, elisabeth.badens@univ-amu.fr

Keywords: Supercritical fluids, carbon dioxide, extraction, fractionation, process optimization

Summary

The use of supercritical fluids (SCFs) is an alternative to that of liquid organic solvents and allows the implementation of « clean » and compact processes. SCFs have intermediate properties between the ones of liquids and gases. SCFs have liquid-like densities, gas-like viscosities and diffusivities about ten times higher than the ones of liquids. This implies that SCFs can be used as solvents and that transfer phenomena will be favored in such media. Moreover, the interfacial tensions at the interface solid/SCF or fluid/SCF tend to zero. SCFs are thus interesting solvents for extraction from a solid matter. Hence, among the different application fields of SCFs, extraction using supercritical carbon dioxide ($scCO_2$) has been the most widely studied those last forty years and a large number of industrial units have been implemented all over the world in different industry sectors (food, cosmetics, perfumes, nutraceuticals, pharmaceuticals).

In comparison with the processes using organic solvents, $scCO_2$ offers many advantages. Its critical coordinates (Tc=31.06°C; Pc=73.8 bar) are easily reachable and its low Tc makes it possible to treat thermosensitive compounds. Moreover, the solvent properties of $scCO_2$ can be tuned significantly simply by varying the conditions of pressure and temperature. Lastly, the processes using $scCO_2$ can be described as «compact» since the separation of CO_2 from the end-product is spontaneous, CO_2 being gaseous at ambient conditions. When a solid product is treated or if a solid end-product is obtained, this spontaneous separation implies that there is no need to carry out several separation stages that are usually required for conventional processes using liquid organic solvents. Consequently, $scCO_2$ extraction process can be definitely economically competitive.

Supercritical CO_2 can also be used for fractionating compounds from a liquid phase. In that case, the process has the great advantage to become a continuous one, enabling a significant reduction of the production costs as well as a reduction of the size of the industrial facilities.

During this plenary lecture, different case studies dealing with supercritical extraction from solid matrices and fractionation from liquid feeds will be discussed with a specific focus on the chemical engineering tools enabling a reliable scale-up. Some current trends will also be addressed. These include: the use of higher pressures for avoiding the recourse to co-solvents for the extraction of polar compounds, the optimization of operating conditions via the study of energy requirements and exergy losses, the use of new solutions for CO_2 separation in order to minimize CO_2 recycling costs or lastly, process intensification.

OR-02. Simultaneous recovery of proteins and polyphenols from hemp seeds under subcritical water conditions

Aziadé CHEMAT [1,2], Anne-Sylvie FABIANO-TIXIER [2], Salah CHAJI [1], Christian CRAVOTTO [1,2], Giorgio CAPALDI [1], Giancarlo CRAVOTTO [1]

[1] Department of Drug Science and Technology, University of Turin, Via P. Giuria 9, 10125, Turin, Italy.
[2] Avignon Université, INRAE, UMR SQPOV, F-84000 Avignon, France.

***Corresponding author:** Aziadé CHEMAT, aziade.chemat@gmail.com

Keywords: hemp seeds, subcritical water extraction, polyphenols, proteins, green extraction.

Objectives

The use of hemp seeds to develop novel high-value products has garnered considerable attention over the past decade due to their richness in various nutritional and bioactive compounds. Hence, the objective of the present work is to investigate an environmentally sustainable approach for the recovery of protein- and polyphenols-rich extracts from hemp seeds using subcritical water extraction (SWE).

Methods

Prior to the optimization process, various samples of hemp seeds (whole, crushed, and defatted) were subjected to preliminary investigation under similar SWE conditions. Afterwards, a response surface methodology was employed, using defatted seeds, to optimize the extraction process, taking into account three parameters (time, temperature, and sample to solvent ratio (w/v)) and four factors (extraction yield, total phenolic content (TPC), protein selectivity, and protein recovery). The optimization was conducted in two distinct directions. The first was based on the objective of achieving an extract rich in both polyphenols and proteins. The second was chosen based on an energetic consumption point of view. Afterwards, real industrial pellets of defatted hemp seeds were used to recover protein-rich extracts using pH-shift method under 1h ultrasonication at cold temperature (10-12 °C).

Results

Our findings indicated that the defatted seeds gave better results in terms of both phenolic and protein contents, when compared to non-defatted whole and crushed seeds. The following optimized parameters were determined using the response surface methodology: a temperature of 180 °C for an extraction time of 60 minutes, using a solid-to-liquid ratio of 1/25 (w:v). The obtained extract from defatted seeds exhibited an average TPC of 10.04 g gallic acid equivalents (GAE)/100g and a protein content of 47.25 g/100g. In comparison, the crushed seeds exhibited a TPC of 8.60 g GAE /100g and a protein content of 38.86 g/100g. A second set of optimized parameters was also determined, with the aim to minimize the energy consumption of the extraction process. The results unveiled that a ratio of 1/16 with an extraction temperature of 165 °C and an extraction time of 35 minutes seems to be the most energetically efficient extraction conditions. When comparing the results achieved with SWE with those obtained from industrial pellets of hemp seeds using ultrasonication followed by pH-shift method, the outcomes were promising reaching significantly higher values than those extracted with SWE. Therefore, this study contributes to the ongoing exploration and utilization of hemp seed, which plays a pivotal role in promoting sustainability and maximizing resource efficiency in the hemp-related sector.

OR-03. Natural dyes eco-extraction using supercritical CO_2

Alix GOMBERT [1], Christelle HARSCOAT-SCHIAVO [1], Christine BRUNET [2], Arnaud FERTET [2], Anne DE LA SAYETTE [2] and Raphaëlle SAVOIRE [1]

[1] Univ. Bordeaux, CNRS, Bordeaux INP, CBMN, UMR 5248, F-33600 Pessac, France.
[2] ARRDHOR - CRITT Horticole, F-17300 Rochefort, France

***Corresponding author:** Raphaëlle Savoire, raphaelle.savoire@ensmac.fr

Keywords: Supercritical CO_2 extraction, maceration, madder, weld, textile dying

The dye compounds are widely present in various sectors (textile, cosmetics, food) and consumers are increasingly seeking for natural products, therefore including natural dyes. The development of synthetic dyes since the early 20th century has led to a loss of skills in dye plants and in extraction of dye compounds. There is, therefore, a need to rediscover plants and enhance the extraction performances to obtain those dye compounds.

Objective

The aim of the present work is to evaluate the feasibility of using supercritical carbon dioxide (sc-CO_2) for the extraction of natural dyes.

Methods

Supercritical fluid extraction was performed on a 2L SFE unit at 500 bar on 50 g of plant powder (Madder roots or Reseda aerial parts) soaked in the extraction cosolvent. Hydroalcoholic cosolvents with different EtOH:water ratios were tested. Yields, colors (of extracts and of silk dyed with extracts) and extract compositions (anthraquinone and flavonoids) were determined. As sc-CO_2 is a non-polar solvent, hydroalcoholic solutions were added as cosolvents to modulate the polarity of the extraction fluid and its selectivity. For each plant, a reference extraction using maceration with a hydroalcoholic solvent was carried out for comparison.

Results

Madder (*Rubia tincorum* L.) and Weld (*Reseda luteola* L.) extracts presented various colors. Madder roots extracts contain anthraquinones (alizarin, purpurin and glucosides) with a pink-red-orange colour while Weld extracts are rich in flavonoids (luteolin, apigenin and glucosides) responsible for the yellow color. Compared to the reference extraction, the sc-CO_2 process does not impact the extraction yield for both plants (about 32 and 15 %, respectively). Nonetheless, significant differences have been observed on the extract composition and on the colour for both the liquid extract and the resulting dyed silk.

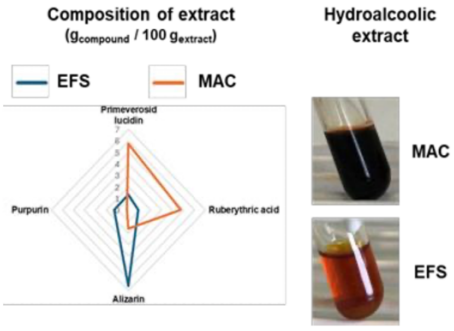


Figure 1. Exemple of two extracts comparison in term of composition and colour obtained by maceration (MAC) and sc-CO_2 (SFE).

OR-04. Optimization of extraction of polyphenols from *Rhamnus alaternus* by deep eutectic solvent-based ultrasound assisted extraction

Amine NEKKAA [1,2,*], Akila BENAISSA [3], Abd El Djalil LALAOUNA [4], Essodjolon PROSPERE KANABYA [5], Christelle MATHE [1], Fabrice MUTELET [1], Laetitia CANABADY-ROCHELLE [1,*]

[1] Université de Lorraine, CNRS, 54000 Nancy, LRGP, France.
[2] Process Engineering Laboratory for Sustainable Development and Health Products, National Polytechnic School of Constantine, Constantine 25000, Algeria.
[3] Drug and Sustainable Development Laboratory (ReMeDD), Faculty of Process Engineering, University Salah Boubnider-Constantine 3, 25000 Constantine, Algeria.
[4] Laboratory of Analytical Chemistry, Faculty of Medicine, University Salah Boubnider, Constantine 3, 25000 Constantine, Algeria.
[5] Department of Chemistry, Faculty of Science, University of Lomé, Togo.

***Corresponding authors:** Amine NEKKAA, amine.nekkaa@univ-lorraine.fr and Laetitia CANABADY-ROCHELLE, laetitia.canabady-rochelle@univ-lorraine.fr

Keywords: *Rhamnus alaternus*, Deep eutectic solvent, Ultrasound-assisted extraction, Polyphenols, Box-Behnken, Antioxidant activity.

Objectives

Rhamnus alaternus, a medicinal plant from the Mediterranean region, is rich in bioactive compounds. However, despite a large number of extraction processes and pharmacological studies concerning *R. alaternus*, there are still insufficient works carried out on the effects and beneficial therapeutic properties of this plant. In this context, this study aimed to explore and optimize novel extraction techniques using a new class of green solvents for the extraction of bioactive compounds from the plant *R. alaternus* and to evaluate its chemical potential and its phytopharmaceutical properties.

Methods

To increase the extraction yield of polyphenols and flavonoids, the extraction process of bioactive compounds combined both deep eutectic solvent DES (ChCl-Gly) and ultrasound assisted extraction. In addition to single factor experiments, a Box-Behnken design was used to optimize the extraction process. The temperature (20 – 60°C), the extraction time (10 – 40 min) and the DES-water content (20 – 50 %) were investigated. The extracts were characterized for their total polyphenols and total flavonoids content, and their antioxidant properties.

Results

The optimal conditions of extraction were: 20 °C of temperature, 31.30 min of extraction time and 31.87 % of water content in DES. In such conditions, the corresponding extract contained 121.18 ± 0.84 mg gallic acid equivalent of polyphenols /g d.w. powder and 38.34 ± 0.63 mg quercitin equivalent of flavonoids /g d.w. powder. These yields of bioactive compounds reached the highest antioxidant capacity (i.e., radical scavenging capacity: IC50 = 19.34 ± 0.14 µg/mL; reducing activity: EC_{50} = 118.97 ± 0.89 µg/mL; iron chelation activity: EC50 = 202.04 ± 1.98 µg/mL). Hence, the antioxidants extraction from *R. alaternus* leaves was successfully optimized using DES-UAE as a green and ecofriendly extraction process [1].

Reference

[1] Nekkaa, A., Benaissa, A., Lalaouna, A, E, D. et al. Optimization of ultrasound-assisted deep eutectic solvent extraction of polyphenols from *Rhamnus alaternus* for their antioxidant activity. Biomass Conversion and biorefinery. (2023). http://doi.org/10.1007/s13399-023-05182-w.

OR-05. Innovative fractionation process for brewer's spent grains upgrading

Karina ANTOUN [1], Malak TABIB [1], Erwann GUÉNIN [1], Gérald ENDERLIN [1], Mohamed KOUBAA [1], Isabelle ZIEGLER-DEVIN [2], Nicolas BROSSE [2], Anissa KHELFA [1]

[1] Université de technologie de Compiègne, ESCOM, TIMR (Integrated Transformations of Renewable Matter), Centre de recherche Royallieu - CS 60 319 - 60 203 Compiègne Cedex, France.
[2] Laboratoire d'Etude et de Recherche sur le Matériau Bois (LERMAB), Faculté des Sciences et Technologies, Université de Lorraine, Vandœuvre-lès-Nancy, France.

***Corresponding author:** Anissa Khelfa, email: a.khelfa@escom.fr

Keywords: brewer's spent grains, lignin, saccharide fraction, pre-treatments, microwaves, ultrasound, steam explosion

Objectives

Brewer's spent grains (BSG), primarily derived mainly from barley, are solid residues from beer production characterized by proteins and fibers (cellulose, hemicellulose, and lignin), with low ash content. Our project ambitiously aims to convert BSG into platform molecules and innovative materials using microwave-assisted fractionation of pretreated and non-pretreated BSG. This biorefinery approach can produce a range of products, including biopolymers such as lignin, known for its antioxidant activity, and platform molecules for the chemical industry like HMF and furfural from polysaccharides. Additionally, microwave-assisted fractionation using natural deep eutectic solvents (NaDES) has demonstrated potential in reducing the time and severity of biomass treatment when compared to conventional methods. NaDES exhibit high solubility and efficient disruption of plant structures, facilitating the extraction of valuable compounds such as lignin, thereby enhancing energy efficiency and environmental sustainability of the process.

Methods

Upon receipt, the BSG (initial moisture content of 75.2 ± 2%) were pressed, dried at 75°C to a moisture content of 6.6 ± 0.7%, and then ground using a ball mill to a particle size of 10-100µm. The grains were characterized using Soxhlet extraction, elemental analysis, Klason lignin and HPAEC-PAD chromatography. For BSG fractionation, a multimode microwave system (2.45GHz) was used to heat the ground BSG mixed with NaDES at 600W, isolating lignin and saccharide fraction. Three NaDES solvents were tested. The fractionation were tested on pretreated BSG (ultrasound and steam explosion pretreatments) et on non-pretreated BSG. A screening method was employed to evaluate and enhance the extraction process by examining two factors: extraction duration and temperature, while keeping the liquid-to-solid ratio (L/S) and solvent type constant. The recovered lignin, after purification steps, was characterized using various techniques: Klason lignin method for purity level, ion chromatography for residual sugar content, FTIR and HSQC-NMR spectroscopyfor detailed lignin structure, and antioxidant activity assessment through DPPH assay. A carbohydrate fraction (filtration residue after lignin purification) was converted to HMF and furfural using microwave mono-mode heating. For this, 250mg of polysaccharide fraction was mixed with 10 ml of 1,2-dichloroethane and 5 ml concentrated HCl. The reaction parameters, including temperature and duration, were varied to optimize the yields of the target molecules. The reaction medium was then analyzed using GC-FID chromatography for HMF and furfural quantification.

Results

The determined specific composition of BSG includes extractables (6.8±0.7%), Klason lignin (14.0±0.6%), cellulose (11.6±0.2%), hemicelluloses (7.7±0.1%) and proteins (17.5±0.5%). For BSG fractionation, the NaDES choline chloride-lactic acid (ChCl-LA) 1:2 was selected due to its thermal stability up to 150°C (from TGA analysis) and its better performance in lignin extraction, achieving an average yield of 18.83%. Microwave-assisted fractionation at 600W using ChCl-LA optimized for time (5 to 30 minutes) and temperature (90°C to 150°C) showed lignin yields of 10-22%, with high purity (72-88%) and low residual sugar. FTIR and NMR-HSQC analyses confirmed the structural integrity and purity of the lignin, while antioxidant activity tests demonstrated the efficacy of the extracted lignins, often surpassing commercial lignin (pure Kraft lignin). Additionally, ultrasound pretreatment at varying amplitudes and durations enhanced the efficiency of subsequent microwave fractionation, achieving consistent and repeatable lignin yields.

OR-06. Sustainable valorisation of industrial hemp: novel technologies and a green solvent

Christian CRAVOTTO [1], Silvia TABASSO [2], Mickaël BARTIER [3], Laurence JACQUES [3], and Anne-Sylvie FABIANO-TIXIER [1]

[1] GREEN Extraction Team, INRAE, UMR408, University of Avignon, Avignon F-84000, France.
[2] Department of Drug Science and Technology, University of Turin, Turin10125, Italy.
[3] EcoXtract, 1bis cour Thévenet, F-59140 Dunkerque, France.

***Corresponding author:** Christian Cravotto, christian.cravotto@alumni.univ-avignon.fr

Keywords: Sustainable food processing; enabling technologies; 2-methyloxolane; cannabinoids; terpenes; hemp seed oil.

Objectives

The aim of this study is the sustainable valorisation of industrial hemp (*Cannabis sativa* L.) by using novel technologies to extract its valuable compounds. Our focus was on the extraction of the two main components of the aerial fraction of hemp, the seeds and the inflorescences.

The study proposes an innovative, sustainable extraction process using a green solvent also in combination with microwave irradiation. Our main objective is to develop a green and economically viable process that maximizes cannabinoids and oil extraction from hemp while promoting sustainable industry practices.

Methods

In this study, we used 2-methyloxolane (2-MeOx) as a green solvent for the extraction of cannabinoids and other phytochemicals (polyphenols and terpenes) from hemp inflorescences, and the oil from hemp seeds. The extraction efficiency of 2-MeOx was compared with that of conventional solvents such as ethanol and hexane under conventional extraction conditions. We also investigated the use of microwave-assisted extraction (MAE) to improve the cannabidiol (CBD) extraction efficiency. Main extraction parameters during MAE were optimised using Response Surface Methodology (RSM), including extraction time, microwave power and water content in the solvent. A single-mode microwave reactor was also tested for the extraction of cannabinoids with 2-MeOx in continuous flow.

Results

Our findings showed that 2-MeOx is an effective green solvent for extracting cannabinoids, polyphenols, terpenes and oil from hemp, providing comparable or superior yields to traditional solvents like ethanol and hexane. Dynamic maceration with 2-MeOx yielded comparable CBD (75.45 mg CBD/g DM) to ethanol (77.71 mg) and hexane (75.09 mg). The use of water-saturated 2-MeOx (4.5% water) increased the CBD recovery to 81.30 mg/g DM and the polyphenols content. Microwave-assisted extraction (MAE) improved the CBD yield and shortened the time (by 3 to 30 times). Optimised MAE resulted in higher CBD yields (84.18 86.76 CBD/DM) compared to a dynamic maceration. Preliminary results using a single-mode microwave reactor showed promising efficiency for continuous flow extraction, indicating potential for industrial scalability. These novel technologies not only improve extraction yields but also reduce environmental impact, contributing to the sustainable valorisation of industrial hemp.

Reference

Cravotto, C.; Fabiano-Tixier, A.-S.; Bartier, M.; Claux, O.; Tabasso, S. Green Extraction of Hemp Seeds Cake (Cannabis Sativa L.) with 2-Methyloxolane: A Response Surface Optimisation Study. Sustainable Chemistry and Pharmacy 2024, 39, 101509, doi:10.1016/j.scp.2024.101509.

OR-07. Extraction of bioactive molecules from apple pomace: evaluation of antioxidant and antibacterial activities

Lauriane BRUNA [1,2], Micheline DRAYE [1], Emilie LYUATEY [3], Giancarlo CRAVOTTO [2,*], Gregory CHATEL [1,*]

[1] EDYTEM, Univ. Savoie Mont-Blanc, CNRS, 5 boulevard de la Mer Caspienne, 73370 Le Bourget-du-Lac, France.
[2] Department of Drug Science and Technology, University of Turin, Via P. Giuria 9, 10125 Turin, Italy.
[3] CARRTEL, Univ. Savoie Mont-Blanc, INRAE, 75 bis avenue de Corzent, 74200 Thonon-les-Bains, France.

***Corresponding authors**: Giancarlo CRAVOTTO, giancarlo.cravotto@unito.it and Gregory CHATEL, gregory.chatel@univ-smb.fr

Keywords: Apple pomace; Antibacterial activity; Antioxidant activity; Subcritical water; Extraction

Objectives

Apple (*Malus domestica* Borkh.) is a common food. Apples are one of the most produced and are the fourth most consumed fruit in the world (Musacchi & Serra, 2018). According to the Food and Agriculture Organization of the United Nations, more than 95.8 million tonnes were produced worldwide in 2022, with 11.8 million tonnes produced in the European Union (FAOSTAT). According to Kammerer et al. (2014), apple juice is the most value-added product, accounting for between 25% to 30% of annual production. Apple pomace (AP) is the residue left after pressing apples. It consists of skin, flesh, seeds, and stems. The quantity of AP waste ranges from 4.7 to 5.6 million tonnes globally. AP can be used, for instance, as animal feed, fuel, ethanol production (via fermentation), compost and biogas from waste treatment (Kennedy et al., 1999). Methanation is used not only for AP but also for crop waste, animal manure, municipal solid waste and municipal wastewater. In the European agricultural community, the methanation process is widely used. In most countries, AP is discarded, and in India, it can be buried in soil (Lyu et al., 2020). By sending waste to methanation centers or burying it, phytochemicals are not valorized. This work focuses on the extraction of bioactive molecules contained in AP using subcritical water for their valorization. Ultrasound (US) pretreatment was used in order to improve the biological properties of extracts.

Methods

The Design of Experiment (DoE) aim to achieve an optimal yield in order to perform efficient extractions. The parameters were extraction time, ratio AP:Water and temperature. When the optimal extraction conditions were determined, AP were extracted with US pretreatment and without pretreatment. To observe any improvement, the extracts were subsequently tested for their antioxidant activity (Total polyphenols content (TPC) and DPPH Radical Scavening Capacity (DRSC)). Additionally, their antibacterial effects are tested against *Escherichia coli*, *Listeria innocua*, and *Bacillus cereus* using a microplate assay to determine their Minimum Inhibition Concentration (MIC) and their EC50.

Results

The DoE for subcritical water extraction (SWE) identified the optimum conditions as 157 °C, 23.4 minutes, and a ratio AP:Water of 1:8 (w/v). The extraction time needs to be sufficient to achieve optimal yield, but should not exceed 30 minutes. While increasing the temperature can enhance extraction, temperatures above 140 °C result in burning of the AP. It could also be beneficial to test a lower AP quantity in water, such as 1:30 (AP, w/v) to observe the different types of molecules extracted under these conditions.

Under the optimal conditions, several compounds were extracted, including carbohydrates, undesirable compounds (5-HMF, FFR) and polyphenols. The optimal conditions were primarily determined based on mass yield. Different optimal conditions may be identified when considering TPC, biological activity, and analytical evaluations. A comparison between US pretreatment and no pretreatment was conducted. The aim of the US pretreatment was to observe any improvement in the biological activities of SWE samples.

TPC and DRSC of both extracts were measured, showing an improvement in TPC extraction but lower DRSC results (Table 1). Against all the strains, the MIC were at a concentration of 50 mg/mL. Against *Listeria innocua* and *Escherichia coli*, US pretreatment was more effective than no pretreatment (Table 2).

Table 1. Characterization of antioxidant activities of extracts AP from subcritical water extraction.

Pretreatment	TPC ($mg_{Extracts}/g$)	EC_{50} ($\mu g_{Extracts}/mL$)
Without pretreatment	110.33 ± 11.55	6.6 ± 0.7
US pretreatment	148.08 ± 7.25	7.6 ± 1.8

Table 2. EC_{50} of samples on different strains bacteria.

Pretreatment	EC_{50} (mg/mL)		
	Listeria innocua	*Escherichia coli*	*Bacillus cereus*
Without pretreatment	15.9 ± 3.1	22.0 ± 1.2	11.3 ± 4.7
US pretreatment	11.8 ± 2.3	19.3 ± 4.3	14.6 ± 2.6

OR-08. Evaluation of green and bio-based solvents for the extraction of ß-carotene and lipids from the oleaginous yeast *R. toruloides*

Vanessa BUCHWEITZ [1], Daili PENG [1], Ahmad ALHADID [1], Mirjana MINCEVA [1]

[1] Biothermodynamics, Technical University of Munich, Germany

***Corresponding author:** Mirjana MINCEVA, mirjana.minveca@tum.de

Keywords: green and bio-based solvents, ß-carotene, oleaginous microorganism, solubility, liquid-liquid equilibrium

Objectives

Organic solvents find widespread use in laboratories and industry. However, their inherent health and environmental risks necessitate exploring solutions to minimize their usage. Seeking greener alternatives is crucial to address these concerns.[1, 2]

The most commonly used extraction methods for the extraction of biotechnologically produced hydrophobic components by oleaginous microorganisms are the Folch [3] method and the Bligh and Dyer [4] method that both utilize the biphasic solvent system containing chloroform, methanol, and water.

In this work, we explored the replacement of harmful conventional solvents with biphasic liquid systems composed of green and bio-based solvents, such as 2-methyltetrahydrofuran (2-MeTHF) and cyclopentyl-methyl-ether (CPME) for the extraction of biotechnologically produced ß-carotene and lipids by the oleaginous microorganism *Rhodosporidium toruloides*.

Methods

The extraction process consisted of two steps. First, ß-carotene and accompanying cellular lipids were extracted from the aqueous lysed biomass by adding a mixture of two solvents of the biphasic liquid system. This step (extraction step) occurred in the single-phase region. Afterward, solvents were added to reach a system composition within the biphasic region (separation step), and two phases were formed: a polar lower phase and a nonpolar upper phase containing the hydrophobic target components.

For the selection of suitable solvent systems and design of the extraction process, knowledge of the liquid-liquid equilibria (LLE) and the solubility of the target component, ß-carotene, in the biphasic system was essential.[5,6]

To circumvent highly time-consuming solubility measurements, the apparent solubility of ß-carotene was measured in a few solvent system compositions and used as a reference to predict the solubility at any other composition within the single-phase region with the thermodynamic model Conductor like Screening Mode for Real Solvents (COSMO-RS).[5]

Results

The LLE of four green and bio-based solvent systems: 2-MeTHF + (ethanol or 1-butanol) + water and CPME + (ethanol or 1-butanol) + water, were measured at 298.15 K and 101.3 kPa.[6]

A good qualitative prediction of the solubility of ß-carotene with the thermodynamic model COSMO-RS was achieved, and regions with different solubility of ß-carotene in solvent system compositions within the single-phase region from lowest to highest for all four solvent systems were determined.

The influence of the ß-carotene solubility in the solvent system composition selected for the extraction step on the extraction yield was evaluated. To ensure that differences in the yield were only influenced by the different solvent system compositions used for the extraction step, the same

composition for the separation step was used for each solvent system. The extraction yield of ß-carotene could be significantly increased by selecting a solvent system composition for the extraction step in which ß-carotene has a higher solubility compared to a composition in which ß-carotene has a lower solubility. Therefore, it was proven that the selection of the solvent system composition for the extraction step influences the extraction yield of ß-carotene when the composition for the separation step was kept the same.

Furthermore, each solvent system and the compositions selected for both the extraction and separation step for all solvent systems were evaluated regarding their solvent consumption, productivity, energy requirement, and carbon footprint to determine the most suitable solvent system.

Acknowledgement:
This work was financially supported by Federal Ministry of Education and Research Germany (BMBF) in the framework of the project OleoBuild (grant number: 031B0853A).

References:

(1) Calvo-Flores, F. G.; et al., Top. Curr. Chem. 2018, 376, 1-40.
(2) Hashemi, B.; et al., TrAC TrendsAnal. Chem. 2022, 116732.
(3) Folch, J.; et al. J. Biol. Chem. 1957, 226 (1), 497-509.
(4) Bligh, E. G.; et al., J. Biochem. Physiol. 1959, 37 (8), 911-917.
(5) Jafari, F.; et al., J. Chem. Eng. Data. 2021, 66(9), 3608-3617.
(6) del Pilar Sánchez-Camargo,A.; et al. TrAC Trends Anal. Chem. 2019, 118, 227-237.
(7) Roth, D. M., et al., Ind. Eng. Chem. Res. 2022, 61(39), 14546-14560.
(8) Buchweitz, V., et al. J. Chem. Eng. Data, 2024. 69,3, 1124-1130.

OR-09. DES-milling combined extraction process targeting pectin from citrus peels and apple pomace

Lucie PERRET [1,*], Carole ANTOINE-ASSOR [1], Suênia DE PAIVA LACERDA [2], Marie-Joo LE GUEN [3], Claire MAYER-LAIGLE [1]

[1] IATE, University of Montpellier, INRAE, Institut Agro, Montpellier, France.
[2] Centre RAPSODEE, University of Toulouse, Albi, France.
[3] Scion, 49 Sala Street, Rotorua 3010, New Zealand.

***Corresponding author:** Lucie PERRET, lucie.perret@umontpellier.fr

Keywords: Deep Eutectic Solvents, pectin extraction, ball milling

Objectives

Pectin is a biopolymer widely used as gelling, thickening, or emulsifying agent in food industry. In other sectors, such as pharmaceuticals or healthcare, its ability to form physical hydrogels is also exploited to manufacture matrices for encapsulating active components (Munarin et al., 2012). It is commonly extracted from food co-products such as lemon peels or apple pomace through acidic extraction. The aim of this study is to develop an alternative method of pectins extraction, based on the use of eutectic solvents coupled with grinding, that maintains its structural integrity and gelation capacity.

Methods

Two different dried biomasses (citrus peels and apple pomace) and three different choline chloride-based DES (CC: lactic acid as DES 1; CC: malonic acid as DES 2; CC: malic acid as DES 3) were used. A reference trial with water as solvent was also performed. The raw materials were milled in a vibratory ball mill in the presence of the DES, with a solid/liquid ratio of 1:6 w/v, using either a ball size of 20 mm or the same mass of balls with 38 beads of 6 mm, besides, two temperatures were investigated: 20°C, 40°C. After milling, centrifugation was carried out to recover the supernatant, in which the solubilized material was then precipitated with ethanol and filtered. The extracts were characterized by FTIR to attest the presence pectin and to quantify the degree of methoxylation by the pic ratio at 1730 and 1630 cm^{-1} using a calibration curve and relate these findings to the process conditions.

Results

The raw materials contain around 15 to 18% of pectin for citrus peels and apple pomace, respectively. The mass yield of the product extracts is from 1% to 4%, which is low compared to acidic extraction reaching around 20% mass yield (Kumar & Chauhan, 2010). FTIR measurements show that pectin is present in all extracts. However, the extraction yield and the degree of methoxylation of the pectin identified in the extract are highly dependent on the process conditions.

The 20mm ball, generating mainly impact, allow for a higher yield of soluble material than 6mm balls, generating mainly friction and impact with smaller energy intensity than the 20mm ball (Rajaonarivony et al., 2019). In the range investigated, DES 3 slightly favours higher yield compared to the others. This could be related to the fact that, this solvent was employed with 30 wt% of water to reduce its viscosity until 97 mPa.s, which is 1.5 and 3.3 times lower than DES 1 and 2, respectively. As a result, the movement of the balls is less hindered and permits a better transfer of the energy to the raw material.

DES 2 favours the extraction of High Methoxy-Pectin (DM>50%) while DES 1 leads to Low Methoxylated-Pectin (DM<50%). Furthermore, 6mm ball favours higher DM recovery compared to

20mm ball. This suggests that less drastic conditions, in terms of energy transmission, could preclude de-esterification of pectin and preserve a higher degree of methoxylation.

Our results suggest that the DES/grinding combination extract soluble compounds, including pectin, even if the yields are low. The composition of the extracted fractions will need to be characterised in order to define the yield and purity of the extracted pectins. Similarly, it will be necessary to determine the impact of solvents on the methylation of pectins in order to verify their potential selectivity as well as the impact of the energy transmission to the materials during the milling.

References

Kumar, A., & Chauhan, G. S. (2010). Extraction and characterization of pectin from apple pomace and its evaluation as lipase (steapsin) inhibitor. Carbohydrate Polymers, 82(2), 454–459. https://doi.org/10.1016/j.carbpol.2010.05.001

Munarin, F., Tanzi, M. C., & Petrini, P. (2012). Advances in biomedical applications of pectin gels. International Journal of Biological Macromolecules, 51(4), 681–689. https://doi.org/10.1016/j.ijbiomac.2012.07.002

OR-10. Cascade extraction of polyphenols and carotenoids, using Natural Deep Eutectic Solvents

Paul SAUVETES [1,*], Nathalie GONTARD [1], Chahinez AOUF [1]

[1] IATE, University of Montpellier, INRAE, Institut Agro, Montpellier, France.

Corresponding author: Paul SAUVETES, paul.sauvetes@inrae.fr

Keywords: Natural Deep Eutectic Solvents, Cascade extraction, polyphenols, carotenoids, agri-residues

Objectives

During the processing of tomato and grape, a large volume of pomace is obtained. These pomaces are mainly composed of peels, seeds and stems and represent around 10% by weight of fresh tomato [1] and 20% by weight of grapes [2]. Currently the pomaces are usually either landfilled or used for livestock feed because of their important nutritional value. Indeed, they contain powerful antioxidants such as carotenoids and polyphenols, high value-added peptides and proteins, and also natural polyesters, namely cutin and suberin (in the case of tomato). Extensive research has been done to increase pomaces value by extracting molecules of interest, and some cascade valorization methods were developed to fully exploit the potential of these residues [1]. However, fossil-based solvents such as hexane, acetone or methanol are used for the extraction, greatly increasing the environmental impact, processing hazard and infrastructure complexity. Meanwhile, the last 30 years witnessed the emergence of alternative extraction solvents such as supercritical fluids, ionic liquids and natural deep eutectic solvents (NADES). A NADES is a eutectic mixture of two or three natural components, interacting through hydrogen bonding, that have a lower melting point than that of each component. Thanks to their intricated hydrogen bonds matrix, NADES acquire interesting extraction properties that can be tailored by changing the composition or adjusting the mix proportions. In addition, these solvents can act as stabilizers for the extracted molecules. [3] As a part of the European project Agriloop (2023-2027 www.agriloopproject.eu), the purpose of this research is to develop a NADES-based extraction technology of carotenoids and polyphenols from tomato and grapes pomaces, and integrate it in a larger sustainable cascade extraction including proteins and polyesters extraction.

Methods

- Pretreatment

Agri-residues are dried to prevent microbial contamination and allow matrices grinding.

- Polyphenols/carotenoids extraction and characterization

Freshly prepared NADES were added to the residue, and extraction was performed under heating and constant stirring in the dark. After extraction the matrix and extract are separated by centrifugation. In parallel, extractions with conventional solvents were carried out and used as a reference. After precipitation of undesired coextracts, UPLC-DAD-MS was used to identify and quantify the extracted molecules.

- Developing the cascade extraction

To define the optimal order of biomolecules extraction, extracts and residual matrices were exchanged between the project partners.

Results

- Pretreatment

Dry fractionation of the residues (grinding) was performed and optimized, and its impact on extraction yield evaluated.

- Polyphenols/carotenoids extraction and characterization

NADES constitution, and extraction conditions (temperature, reaction time, matrix to solvent ratio, successive extraction) of target molecules (carotenoids and polyphenols) were determined for tomato peels, tomato seeds and grape pomace.

Polyphenols present in tomato peels and seeds were identified as flavonoids and phenolic acids derivatives. As the polyphenols contained in grape pomace are proanthocyanidins (condensed tannins), a depolymerization method enabled an accurate identification and quantification of these polyphenols.

- Developing the cascade extraction

The identification of the impact of each extraction on the yield and structural integrity of other extracts is ongoing.

References

[1] A. Casa Marcello, B. Michele Miccio, C. Giovanni De Feo, D. Andrea Paulillo, E. Roberto Chirone, F. Dalia Paulillo, G. Paola Lettieri, H. Riccardo Chirone. Detritus, no 15, 31 (2021).
[2] A. Amico, V., B. R. Chillemi, C. S. Mangiafico, D. C. Spatafora, E. C. Tringali, Bi e ce Tech g 99 13, 5960 66 (2008).
[3] A. Karoui, Iness Jabri, B. Emna Chaabani, C. Imen Dali, D. Abdelkarim Aydi, E. Ma di Ha a i, F. Ma ef Abde abba, J a f F d Scie ce 88, 9, 3714 24 (2023).

OR-11. Green solutions to extract bioactive compounds from vitro-derived hop plantlets treated with elicitors

Leandra LETO [1], Anna AGOSTI [1], Samreen NAZEER [1], Lorenzo DEL VECCHIO [1], Andrea DI FAZIO [1], Valeria GUARRASI [2], Jorge JUAN-VICEDO [3], Martina CIRLINI [1], Benedetta CHIANCONE [1,2]

[1] Department of Food and Drug, University of Parma, Viale Parco Area delle Scienze 27/A, 43124 Parma, Italy
[2] Institute of Biophysics, National Research Council (CNR), Via Ugo La Malfa 153, 90146 Palermo, Italy
[3] Universidad Católica de Valencia San Vicente Mártir, Instituto de Investigación en Medio Ambiente y Ciencia Marina (IMEDMAR), Calle Guillem de Castro, 94, 46001, Valencia, Spain.

***Corresponding author:** Leto LEANDRA, leandra.leto@unipr.it

Keywords: Antioxidant activity, methyl jasmonate, salicylic acid, secondary metabolism, total polyphenol content

Objectives

Hop plants (*Humulus lupulus*) are rich in bioactive compounds such as terpenoids, phenolic compounds (e.g xanthohumol), alkaloids and bitter acids (humulone and lupulone) which are used as additives in the agri-food, pharmaceutical and cosmetics industries, especially today, due to their antioxidant, antimicrobial and antiviral properties. Recent studies have shown that the same compounds present in plants in the open field are also synthesised in smaller amounts in vitro-derived plantlets.

In this study, two elicitors, methyl jasmonate (MeJa) and salicylic acid (SA) were added in the culture medium to stimulate the secondary metabolism of in vitro cultured hop plants, in order to increase the synthesis of bioactive compounds. Moreover, to validate the efficacy of green extraction methods for extracting bioactive compounds from vitro-derived hop plants, only water was used for the extraction protocol in addition to ethanol/water.

Methods

In this study, hop cv. Columbus, plantlets were in vitro cultured in 50 ml glass test tubes, containing a culture medium (solid phase-SP) with the following composition:

Murashige and Skoog (MS) basal salt mixture (1×), MS vitamin mixture (1×) (Murashige and Skoog, 1962), 30 g/l sucrose and 8.0 g/l agar, pH 5.8. Test tubes and culture medium were sterilized at 121°C for 20 min. An uninodal microcutting was cultured per each test tube and then, stored in a growth chamber at 25±1°C, 20 mol $m^{-2}s^{-1}$, and a 16-hour photoperiod. After one week of culture, 2 ml of a liquid culture medium (FP) was added over the SP. The composition of the FP was as follows: ½ KNOP salts (Serrano-Martínez et al., 2011), 60 g/l sucrose, pH 5.0. MeJA and SA were added to the FP at two concentrations (2 µM and 4 µM) to stimulate secondary metabolism. The experimental design comprised 24 tubes for each thesis. The following theses were considered: 1) Control (C): SP+FP; MeJa-2: C + 2 µM of MeJa; MeJa-4: C + 4 µM of MeJa; AS-2: C + 2 µM of SA; AS-4: C + 4 µM of SA. After 5 weeks, vitro-derived plantlets were lyophilized using a Freeze dryer Lio-5P (5Pascal, Milan, Italy) pulverized, and extracted with either an Ethanol/Water (EW) solution (80/20 v/v) or Water-Only (WO). A dilution factor of 1:40 was used for the extraction. The extraction was performed using a shaker (HS 501 digital shaker, IKA-Werke GmbH & Co, Staufen, Germany) set at 200 strokes/minute for 2 h at room temperature. The extracts were then centrifuged at 5,000 rpm for 10 min at room temperature (Centrifugette 4206 centrifuge, Alc International, Pévy, France). Supernatants were furtherly diluted 1:5 with distilled water, and each extraction procedure was repeated twice to evaluate the total polyphenol content (TPC) and antioxidant activity (AO). Statistical analysis was carried out using a three-way ANOVA, considering the influence of the factors Elicitor Type-ET, Elicitor Concentration-EC and E traction Solvent-ES .

Results

For the TPC parameter, none of the factors tested showed a significant influence on the hop plantlet response. This result is particularly interesting; in fact, statistically comparable TPC values (8.08 mg GAE/g ± 0.93 for EW vs. 7.85 mg GAE/g ±1.22 for WO) were recorded for the two extraction methods tested, confirming that water-only extractions (WO) are a valid green alternative.

For the DPPH and FRAP assays, extracts from plantlets grown in MeJa-enriched culture medium exhibited an AO significantly higher than those from plantlets grown in SA medium. Furthermore, the DPPH and FRAP tests showed that extracts obtained with EW solvent exhibited higher antioxidant activity than those extracted with WO. Additionally, the ABTS assay highlighted a significant interaction between the factors “ET” and “EC”; within the extracts from plantlets grown with MeJa, those from MeJa-4 showed the statistically highest antioxidant activity (AO).

Results obtained in this study, although preliminary, lay the groundwork for further investigations on the use of elicitors to increase the synthesis of bioactive compounds in vitro cultured hop plantlets. Further studies on the use of different elicitors at various concentrations are warranted. Moreover, even with a smaller TPC and lower AO, water- only extractions were confirmed to be a valid green solution to exploit vitro-derived hop plantlets as source of bioactive compounds. Future studies will be focused on exploring different green solvents and improving their efficacy, to make the extraction process more cost-effective and environmentally friendly.

OR-12. Efficacy of the bio-solvent 2-methyltetrahydrofuran for microwave-assisted extraction of carotenoids from *Chlorella vulgaris*. Comparison with other traditional organic solvents

Ángeles MORÓN-ORTIZ [1], Paula MAPELLI-BRAHM [1,*], Antonio LEÓN-VAZ, [2], Rosa LEÓN [2], Antonio Jesús MELÉNDEZ-MARTÍNEZ [1]

[1] Food Colour and Quality Laboratory, Facultad de Farmacia, Universidad de Sevilla, 41012 Sevilla, Spain.
[2] Laboratory of Biochemistry, Faculty of Experimental Sciences, Marine International Campus of Excellence (CEIMAR), Universidad de Huelva, Spain.

***Corresponding author:** Paula MAPELLI-BRAHM, pmapelli@us.es

Keywords: microalgae, bioactive compound, sustainability, green techniques, 2-methyloxolane

Objectives

Carotenoids are isoprenoid compounds with different properties beneficial to human health. In addition, they play an important role in the food industry, ranging from serving as colorants to acting as health-promoting ingredients in various food products, among others. The food industry is placing a great emphasis on sustainable technologies, such as microwave-assisted extraction (MAE). MAE is gaining popularity due to its ability to enhance carotenoid extraction compared to other traditional extraction methods (maceration, Soxhlet, etc.). The use of green solvents that are non-toxic, recyclable, and biodegradable, is also in demand to replace other less desirable solvents commonly used for carotenoid extraction such as n-hexane or dichloromethane. 2-Methyltetrahydrofuran (2-MeTHF) is a promising green solvent that requires further investigation.

This study aimed to compare the individual and total carotenoid content (TCC) extracted through organic and emerging green solvents and to evaluate the extraction efficiency of sustainable technologies.

Methods

The matrices evaluated were *Chlorella vulgaris*, represented in both freeze-dried and fresh forms. MAE was performed at 300 W for 5 minutes, employing a solid-to-solvent ratio of 1 g/20 mL, and a temperature of 90 °C. Individual carotenoid contents and TCC obtained by MAE were compared to that obtained by maceration extraction (control), conducted for 5 minutes, at a 1 g/20 mL ratio, at 90 °C, in a water bath shaker (at 15 U/min). Three different solvents were investigated: n-hexane, dichloromethane (DCM), and 2-MeTHF.

Results

The carotenoids identified in the matrices, listed in descending order quantitatively, were lutein, α-carotene, β-carotene, (9Z)-antheraxanthin, zeaxanthin, and (9Z)-β-carotene. The application of the MAE resulted in a significant increase in carotenoid extraction compared to the conventional method for all solvents and matrices investigated. Thus, the TCC in the extracts obtained by MAE were 1.7 – 4.4 times higher than those obtained by maceration, depending on the solvent and the matrix employed. These findings may be attributed to the cell wall disruption induced by MAE in *C. vulgaris*.

Among the evaluated solvents, 2-MeTHF exhibited the highest efficacy in recovering TCC in the fresh matrix, both in the control and in the microwave-treated samples (Table 1.A).

In the MAE-treated freeze-dried sample, the most efficient solvent was 2-MeTHF, followed by DCM, with no significant differences noted between them (Table 1.B). When taking into account the humidity percentage of the sample (78.5%), the expected TCC of the freeze-dried sample (expressed

on a fresh weight basis), among all solvents and treatments used, would be 832.12 µg/g. Therefore, considering the average values obtained with all the solvents evaluated, the extraction of total carotenoids in the fresh microalgae was 2.8 times higher than that of the freeze-dried sample.

Table 1. TCC obtained with different solvents (n-hexane, DCM, 2-MeTHF) and treatments (MAE and maceration extraction (control)) from fresh (A) and freeze-dried (B) *C. vulgaris*. Different capital letters refer to differences between solvents in each treatment. P-value represents significant differences between control and MAE: *, p<0.05; **, p<0.01; ***, p<0.001. -

A)

	CONTROL		MAE		*p*-value
***n*-Hexane**	0.71 ± 0.02	C	3.16 ± 0.20	C	***
DCM	70.74 ± 5.77	B	216.68 ± 15.86	B	***
2-MeTHF	160.86 ± 6.73	A	490.72 ± 19.07	A	***

B)

	CONTROL		MAE		*p*-value
***n*-Hexane**	55.78 ± 5.28	C	95.73 ± 5.16	B	***
DCM	511.36 ± 46.52	A	857.28 ± 42.34	A	***
2-MeTHF	273.94 ± 21.01	B	880.25 ± 10.36	A	***

2-MeTHF is a promising novel emerging green solvent as an alternative to other organic solvents (DCM and hexane) for the extraction of carotenoids from fresh and freeze-dried *C. vulgaris*. The application of MAE, regardless of the solvent used, significantly increased the extraction of each individual carotenoid in all matrices.

Acknowledgments:

Grants PID2019-110438RB-C21 (NEWCARFOODS) and PID2019-110438RB-C22 (BESTALGAE4CAROT), funded by MCIN/AEI/10.13039/501100011033. AMO holds a FPI fellowship associated with NEWCARFOODS (PRE2020-096617). PMB was supported by a postdoc fellowship from Consejería de Transformación Económica, Industria, Conocimiento y Universidades de la Junta de Andalucía (PAIDI 2020).

Topic 2. Sustainable and clean extraction technologies

OR-13. Pulsed electric energy treatments to enhance extraction of natural products

Eugene VOROBIEV [1,*]

[1] Université de Technologie de Compiègne, Laboratoire TIMR, 60200 Compiègne, France

***Corresponding author**: Eugene VOROBIEV, eugene.vorobiev@utc.fr

Keywords: Pulsed electric field, extraction, pressing, bioactive compounds

Abstract

For the effective extraction of natural products (carbohydrates, colorants, antioxidants, proteins, aromas, flavours, etc.) from food plants and biomass (roots, pomaces, peels, seeds, microalgae, yeasts, different wastes, etc.), preliminary damage of cellular structure of raw materials is needed. Conventional thermal and mechanical methods of cell destruction are highly energy consuming and non-selective. Consequently, undesirable cell compounds (impurities) are extracted from biomaterials complicating following purification of extracts.

This lecture presents the mechanisms of cell damage by pulsed electric energy (PEE), its impact on the physical properties of biological media, and gives examples of mass transfer enhancement in electroporated cell networks. Different methods to detect and quantify electroporation phenomena in biological tissue are presented. Impacts of electroporation on the mechanical, diffusional and electrophysical properties of biological media are illustrated by numerous examples. Physical models of liquid expression and compounds diffusion in electroporated biological tissue are presented. Several innovative green technologies based on the pulsed electric energy are presented, including selective extraction, pressing, and drying of food plant materials and biomass.

OR-14. Ultrasound- and PEF-assisted Industrial Production of EVOO with higher content of Bioactive Compounds

Luisa BOFFA [1], Emanuela CALCIO GAUDINO [1], Giorgio GRILLO [1], Arianna BINELLO [1], Giancarlo CRAVOTTO [1]

[1] University of Turin, Department of Drug Science and Technology, Via P. Giuria 9, 10125, Turin.

***Corresponding author:** Luisa BOFFA, luisa.boffa@unito.it

Keywords: Ultrasound; Pulsed Electric Field; Extra Virgin Olive Oil Production; EVOO; Polyphenols; Tocopherols; Industrial Scale; Shelf-life.

Objectives

The aim of the present study was to implement new technologies in olive oil mills and to develop an efficient industrial process for the continuous extraction of a healthier extra virgin olive oil (EVOO), enriched with bioactive compounds such as polyphenols and tocols. EVOO is a cornerstone of the Mediterranean diet. Many studies have highlighted its crucial preventive role against cardiovascular disease, neurodegenerative disorders, metabolic syndrome and cancer, due to the anti-inflammatory and antioxidant synergistic activities of polyphenols and tocols [1].

Methods

Non-thermal technologies, namely ultrasound (US) and pulsed electric field (PEF) have been tested separately or in combination to eliminate the need for traditional malaxation. Extensive literature supports the efficacy of ultrasound-assisted extraction (UAE) and PEF treatments in EVOO production [2,3]. Starting from pilot scale trials, different US and PEF equipment were evaluated in the extraction of EVOO from several autochthonous olive cultivars such as Taggiasca, Mattea and Coratina. Extraction yields, physico-chemical and organoleptic characteristics, and polyphenol and tocopherol contents were monitored throughout the trials.

Results

Our study introduced a unique continuous flow setup integrating a US device with an industrial PEF chamber, which resulted in improved EVOO production (ton/die) through potent non-thermal physical effects such as acoustic cavitation and electroporation. In addition, these innovations enriched the resulting EVOO with nutritionally relevant minor components (8-12% polyphenols, 3-5% tocols), thereby elevating its quality and market value, as well as overall shelf-life. This combined UAE and US-PEF process not only increased daily oil production (near 40%), but also eliminated the need for kneading during malaxation, resulting in significant energy savings (approximately 30%).

The introduction of continuous-flow US and PEF technologies heralds a remarkable innovation in the EVOO industry, offering benefits to both producers and consumers. The resulting EVOO from non-thermal continuous-flow production meets the growing demand for healthier, nutrient-enriched products.

References

[1] Foscolou, A., Critselis E., Panagiotakos D. (2018). Olive oil consumption and human health: A narrative review. Maturitas, 118, 60-66. https://doi.org/10.1016/j.maturitas.2018.10.013.

[2] Clodoveo, M. L. (2019). Industrial Ultrasound Applications in The Extra-Virgin Olive Oil Extraction Process: History, Approaches, and Key Questions. Foods (Basel, Switzerland), 8(4), 121. https://doi.org/10.3390/foods8040121.

[3] Veneziani, G., Esposto, S., Taticchi, A., Selvaggini, R., Sordini, B., Lorefice, A., Daidone, L., Pagano, M., Tomasone, R., & Servili, M. (2019). Extra-Virgin Olive Oil Extracted Using Pulsed Electric Field Technology: Cultivar Impact on Oil Yield and Quality. Frontiers in nutrition, 6, 134. https://doi.org/10.3389/fnut.2019.00134.

OR-15. Green extraction technologies driving circular economy in the Sicilian prickly pear industry

Giorgio CAPALDI [1,*], Giorgio GRILLO [1], Christian CRAVOTTO [1], Monica VOSS [1], Alice HOLKEM [2], Giancarlo CRAVOTTO [1]

[1] Drug and Science Department, University of Turin, Turin, Italy (2) Universidade Federal de Santa Maria, Santa Maria, Brazil

***Corresponding author:** Giorgio CAPALDI, giorgio.capaldi@unito.it

Keywords: Green Extraction, Industrial Application, Circular Economy, Prickly Pear, Natural Pigment, Bioactivity, Nutraceutical

Objectives

In the last decade, green extraction has emerged as a sustainable and economically alternative to conventional methods. Nowadays, the challenge is the application of the so-called enabled technology on industrial scale in real economic scenario. This collaborative project, involving the University of Turin, Tropical Food Machinery, and a Sicilian start-up La Deliziosa, focus on processing cosmetically imperfect prickle pears and studying by-products, such as peels (PPP), seeds (PPS), and cladodes (PPC) for new products development. The project encompasses three milestones: lab-scale process optimization, semi-industrial scale-up to assess technology readiness level (TRL), and evaluation of extract composition, bioactivity, and downstream technology.

Methods

Microwave-assisted extraction (MAE), subcritical water extraction (SWE) and ultrasound-assisted extraction (UAE) have been optimized for the recovery of metabolites from PPP and PPC. The optimal protocol for PPP on semi-industrial SWE prototype processed 30 kg of peels (scale-up factor of ×300). The investigated downstream technologies included membrane filtration (bioactive fractionation and concentration), followed by water removal techniques (freeze-drying vs. spray-drying). The extracts were characterized for phenolic compounds, carbohydrates, and betalain content together with antioxidant activity (chemical and electrochemical methods), enzymes inhibition potential and thermal stability.

Results

The optimized extractions showed higher yield and bioactivity for thermal technology (MAE, 80 °C, 20 min, S/L 1:5) compared to non-thermal technology (UAE, RT, 10 min, 20 kHz, 500 W, S/L 1:5). Membrane filtration on PPP extract allowed to recover two products (pectin and betalain) promoting also water recycling. The PPP extracts e hibited antio idant acti ities but no inhibition of -glucosidase and -amylase. On the contrary, -glucosidase inhibition was observed for PFC extract, rich in polysaccharides with antioxidant activity, as well. The process for PPP valorization reached TRL 7 being ready to be tested for commercial activities. Future studies should examine economic viability with long-term data and the impact on consumer acceptance and market dynamics for PPP, testing also on industrial scale the valorization strategies for seeds and cladodes.

OR-16. Microwave assisted hydrodistillation of hop terpenes: An application in the brewing industry

Diego CARNAROGLIO [1,*], Lorenzo ROSSI [1], Giancarlo CRAVOTTO [2]

[1] Milestone Srl, via Fatebenefratelli, 1-5, 24010 Sorisole, Italy.
[2] Dipartimento di Scienza e Tecnologia del Farmaco, University of Turin, Via P. Giuria 9, 10125 Turin, Italy.

***Corresponding author:** Diego CARNAROGLIO, d.carnaroglio@milestonesrl.com

Keywords: green extraction; hops (*Humulus lupulus* L.); microwave-assisted hydrodistillation; terpenes; pilot-scale extraction

Objectives

Interest in essential oils has steadily increased in recent years. Essential oils are used in numerous areas, including the food, cosmetics and pharmaceutical industries. The volatile fraction of hops (*Humulus lupulus* L.) falls into this area, as it can be used in the brewing industry for the aromatization of beer, and is responsible for the floral and fruity tones. The aim of this work is to present an optimised extraction protocol for the essential oil of hops using microwaves.

Methods

Microwave-assisted hydrodistillation (MAHD) is an innovative, environmentally friendly process for extracting volatile components from plant material, with which excellent extraction results can be achieved in terms of quality and quantity, with a focus on sustainability.

Results

A significant reduction in time and energy and water consumption is achieved.[1] The application of MAHD in the hopping process for the production of craft beers is presented, allowing an increase of up to 10% in brewing yield and a reduction in hop consumption of up to 40%.[2]

References

[1] L. Lamberti, G. Grillo, L. Gallina , D. Carnaroglio, F. Chemat, G. Giancarlo Cravotto Foods 2021, 10, 2726.
[2] https://www.milestonesrl.com/products/microwave-extraction-for-processors/ethos-x-line-for-hops-extraction

OR-17. Application of DIC technology for the extraction of high added value compounds from *Cannabis Sativa* L. buds according to the bio-refinery concept.

Dounia BOUCHEKIOUA [1,2,*], Tanguy BAUFFE [1,2], Victor LEFRANCOIS [2], Mohammed BENALI [1], Houcine MHEMDI [1].

[1] Université de Technologie de Compiègne, ESCOM, TIMR (Integrated Transformations of Renewable Matter), Centre de recherche Royallieu - CS 60 319 - 60 203 Compiègne Cedex, France
[2] Abcar DIC Process SAS, 11 Rue du Four Saint-Jacques, 60200 Compiègne, France

***Corresponding author:** Dounia BOUCHEKIOUA, dounia.bouchekioua@abcar-dic.com

Keywords: *Cannabis Sativa* L., Extraction, Essential Oil, CBD, DIC

Objectives

Instant Controlled Pressure Drop (DIC) is an emerging agri-food technology that applies high-temperature, short-time treatments for biomass texturation by expansion while preserving product quality. Compared to conventional thermal processes, DIC application enhances the kinetics of extraction, drying and preserves the product quality while reducing energy costs.

This research aims the application of DIC pretreatment on Cannabis Sativa L. buds for the extraction of essential oils and cannabidiol (CBD). Objectives include decarboxylating the plant, improving the extraction efficiency, preserving extracts quality, increasing storage stability, and exploring product formulation. This work investigates how to enhance process efficiency from harvest to product formulation by integrating DIC technology for hemp pretreatment, addressing scientific challenges, and offering higher-quality hemp products according to the bioreffinery concept.

Methods

DIC processing was performed using pilot equipment from ABCAR DIC Process (France). The DIC process involves increasing the pressure in the treatment chamber, maintaining it, and then depressurizing the chamber. Typical parameters include:

- Pressure
- Duration
- Number of cycles

Treatment parameters were optimized based on the plant material and the target outcomes, aiming to decontaminate and decarboxylate the flowers, and improve the extraction of bioactive compounds. Parameters ranged from 1 to 35 seconds, 1 to 6 bars, and 1 to 8 cycles.
CBD extraction kinetics were studied by dynamic maceration on samples of hemp buds:

- Non-treated
- Treated by DIC
- Non-treated and steam distilled
- Treated and steam distilled

Extracts were analyzed by gas phase chromatography.

Results

DIC treatment significantly enhanced the extraction kinetics of CBD. The conventional extraction method yielded 140.94 mg of CBD after 15 minutes, whereas with DIC pre-treatment, the extraction time was reduced to only 5 min. Moreover, the decarboxylation rate of CBDA to CBD was assessed by comparing concentrations of CBDA and CBD in the extracts. DIC treatment alone achieved a decarboxylation more than 80% vs. 11% for the control sample without DIC.

The aromatic profile of the essential oils, analyzed by GC, revealed differences between oils extracted from DIC-treated buds and those obtained by conventional extraction methods (steam distillation and dynamic maceration). Oils from DIC-treated buds demonstrated a fractionated extraction of volatile and non-volatile compounds. In the other hand, results showed that DIC treatment significantly enhanced drying kinetics, shortening the drying time from more than 32h to only 10h.

The effects of DIC on microbial inactivation was evaluated by comparing microbial colony counts (bacteria and fungi) in untreated and DIC treated (2 bars and 25 s) hemp buds. DIC treatment reduced microbial counts by approximately 4 log (cfu/g of dried buds), highlighting DIC's effectiveness for plant product decontamination and improved storage stability.

Finally, it was shown that essential oils in the discharge waters generated by DIC formed stable emulsions, suitable for bio-pesticides formulation. These emulsions, classified between nanoemulsions and macroemulsions, exhibited potential as fungicidal pesticides, insecticides, and nematicides, indicating additional applications for DIC-treated hemp flowers.

OR-18. Alternative recovery of natural acetoin by hybrid pervaporation / distillation process.

Anne-Sophie PERNAK [1,*], Guillaume MOREL [1], Gaëlle FOUCAMPREZ [1], Thomas AUXENFANS [1], Vincent BELLEVERT [2], Pierre TIBAYRENC [2] and Hélène HENNEBEL [1].

[1] LESAFFRE INSTITUTE OF SCIENCE & TECHNOLOGY, Marcq-en-Barœul, France.
[2] ENNOLYS BY LESAFFRE, Soustons, France.

***Corresponding author:** Anne-Sophie PERNAK, as.pernak@lesaffre.com

Keywords: natural aromatic molecule, acetoin purification process, organic solvent-free extraction, eco-designed process of purification

Objectives

Acetoin (3-hydroxybutanone) constitutes a high-value added platform chemical and is widely used in food, cosmetics, detergents, chemical synthesis or cosmetics. Acetoin is a pale to yellowish liquid with a fatty creamy butter taste. It works largely as chemical precursor, flavour or fragrance. Current supply of commercial acetoin is largely from chemical synthetic methods from fossil feedstocks. Some plants and animals also synthetize trace amount of acetoin. Since the fossil resources are becoming increasingly scarce, extensive efforts have been made to develop natural acetoin production using fermentative methods. However, both productivity and product quality are generally insufficient.

Our main challenges were thus to develop competitive approach in acetoin production from fermentation to satisfy the regulations requirements (purity > 95 %) as well as to avoid any use of organic solvents for product recovery, especially in the context of sustainability and natural labelling. More specifically, the main process specification was to obtain (i) a natural acetoin grade, (ii) a final purity at least 98 %, (iii) a good sensory and aromatic profile as well as (iv) the demonstration of both technical and economic viability at large scale.

Methods

An alternative solvent-free process was developed for the acetoin recovery from fermentation broth. In this aqueous system, direct acetoin distillation was challenged due to the azeotropic composition. Azeotropic distillation was not an option regarding the regulation specifications, requiring the need of an entrainer for azeotrope breaking. Different preconcentration methods were thus unsuccessfully explored to achieve an acetoin amount in water of at least 50 %, such as evaporation, combination of nanofiltration followed by reverse osmosis. Among these alternatives, pervaporation, a technology combining membrane separation and vacuum evaporation, in association with distillation is emerging as a suitable choice, mostly due to the independence presented by the membrane technologies with the phase equilibrium.

A proof of concept was firstly carried out on a laboratory scale, including membrane screening and the definition of operating parameters. Both physicochemical composition and sensory profile of prototypes were also validated. Finally, pilot scale study was conducted to validate the scalability of the process and assess its economic feasibility.

Results

Preliminary membrane screening was performed for evaluating the most adapted pervaporation mode between the acetoin concentration in permeate or in the retentate. Based on these results, we demonstrated that the dehydration of the broth is more relevant than filtration, using a hydrophilic silica-based membrane. By using this filtration media, water and hydrophilic molecules are preferentially

transmitted in the permeate and acetoin is recovered in the retentate. Under optimal temperature and vacuum conditions, pervaporation allows for obtaining a concentrated solution with acetoin purity of at least 70%. Combined with distillation step, a final product with a purity > 98% was obtained, validating the proof of concept.

The robustness of this process was then evaluated at pilot scale from several fermentation batches. This study showed that the chemical stability of the feed is a critical point, especially the residual sugars amount causing irreversible membrane fouling due to Maillard reactions in the pervaporation unit. This disappointment needed to redesign the initial process, from the fermentation to the downstream process. Feed stability was thus achieved by nanofiltration.

This new optimal process demonstrated the technical feasibility to obtain a natural grade of acetoin with a purity > 98%. However, the global extraction yield was less than the expected one. In these conditions, this free-solvent purification process seems to be uncompetitive compared to the chemical synthesis process. Apart from this, other constraints also remained for acetoin recovery from broth fermentation by pervaporation at industrial scale, such as membrane longevity. Further studies are in progress to overcome these challenges.

OR-19. Synergistic extraction of bioactive compounds using enzyme-assisted extraction (EAE) in combination with high-pressure homogenization (HPH)

Fatemeh MOJARRADI [1], Alessandra PROCENTESE [1], Francesco DONSÌ [1,2,*]

[1] Department of Industrial Engineering, University of Salerno, Fisciano (SA) – Italy.
[2] ProdAl scarl, c/o University of Salerno, Fisciano (SA) – Italy.

***Corresponding author:** Francesco DONSÌ, fdonsi@unisa.it

Keywords: Extraction, bioactive compounds, EAE, HPH.

Objectives

The main focus of the research activity is the investigation of the qualitative and quantitative impacts of enzyme-assisted extraction (EAE), and high-pressure homogenization (HPH) techniques separately and in combination with each other on the extraction of the valuable compounds contained in different agrifood residues, as well as to analyzing the operating parameters effects on the extraction yield improvement and identifying optimum parameters (enzymes concentration, pH, and temperature for EAE, pressure and the number of passes for HPH).

Methods

The quality of the obtained extracts have been assessed using several techniques and tests, including the total phenolic compounds (TPC), ferric reducing antioxidant power (FRAP), and 2.2-diphenyl-1-picryl hydrazyl (DPPH) radical scavenging capacity. Additionally, the rate of cellular damage has been evaluated by measuring the amount of ATP released and the change in electrical impedance upon cell permeabilization, as well as through the scanning electron microscopy (SEM) technique. Besides, the effectiveness of the mentioned techniques, separately and in combination, has been demonstrated through HPLC analysis to identify and compare the types of bioactive compounds released based on the utilized extraction techniques.

Results

The extraction yield of bioactive compounds and the antioxidant activity of the obtained extracts by EAE, and HPH have been significantly increased compared to control samples. Particularly, the use of combined techniques, under optimized conditions, enhanced the extraction efficiency in a synergistic way. This research aims to demonstrate the considerable potential of EAE, and HPH techniques for extracting bioactive compounds from agri-food residues.

OR-20. Optimization of enzyme-assisted extraction of bioactive compounds from the pseudo-fruit of *Rosa Canina* L. and evaluation of the biological potential of the optimum extract

Zafeiria LEMONI [1], Styliani KALANTZI [1], Theopisti LYMPEROPOULOU [2], Andromachi TZANI [3], Anastasia DETSI [3] and Diomi MAMMA [1,*]

[1] Biotechnology Laboratory, Zografou Campus, School of Chemical Engineering, National Technical University of Athens, 15780 Athens, Greece.
[2] Processes and Products Quality Control Horizontal Laboratory, Zografou Campus, School of Chemical Engineering, National Technical University of Athens, 15780 Athens, Greece.
[3] Laboratory of Organic Chemistry, Zografou Campus, School of Chemical Engineering, National Technical University of Athens, 15780 Athens, Greece.

***Corresponding author:** Diomi MAMMA, dmamma@chemeng.ntua.gr

Keywords: *Rosa canina* L. pseudo-fruit; Enzyme Assisted Extraction (EAE); Box-Behnken experimental design; Total flavonoid content (TFC); Total phenolic content (TPC); Antioxidant activity; Biological activities

Objectives

The pseudo-fruit of Rosa canina L. is a rich source of bioactive compounds with antioxidant, anti-inflammatory, anti-cancer, gastroprotective, and antimicrobial activities.

Enzyme-assisted extraction (EAE) uses specific enzymes to break down or modify cellular walls to release bioactive compounds. The aim of the present study is the optimization of a green process based on the synergistic action of hydrolytic enzymes (cellulases, hemicellulases, and pectinases) for the extraction of bioactive compounds from the pseudo-fruit of *Rosa canina* L. of Greek origin, as well as the evaluation of the biological activities of the optimum extract.

Methods

A Box–Behnken Design–Response Surface Methodology (BBD–RSM) experimental design was applied to determine the optimal conditions of EAE. The variables were the enzyme load of Cellic® CTec3 (cellulolytic enzyme), the enzyme load of Pectinex® Ultra Color (pectinolytic enzyme), the enzyme load of Viscoferm® (hemicellulolytic enzyme), and extraction time. The responses were the total phenolic content (TPC), total flavonoid content (TFC), and antioxidant activity. The TPC was determined using the Folin–Ciocalteu method, while the TFC with the Dowd method. The antioxidant activity was evaluated with the DPPH radical scavenging method. Simultaneous optimization of the three responses was performed using Derringer's desirability function method. The optimum extract was characterized for its antimicrobial activity (against Escherichia coli, by broth microdilution method), anti-aging activity (tyrosinase inhibition), and anti-diabetic activity (a-glucosidase inhibition).

Results

The optimal conditions were found enzyme load of 0.10% for Cellic® CTec3, 0.12% for Pectinex® Ultra Color, and 0.30% for Viscoferm®, with an extraction time of 44 minutes, achieving a desirability (D) value of 0.73. The optimum values of the responses were: TPC 125 mg GAL/$g_{raw\ material}$, TFC 41 mg CAT/ graw material and IC50 0.88 $\mu L_{extract}/mL_{solution}$. The optimum extract showed 63% inhibition of the *E. coli* growth, while 81% antiaging activity and 55% anti-diabetic activity. The findings offer a solid foundation for the scalable extraction of high-value compounds from pseudofruit of *Rosa canina* L.

OR-21. Multi-criteria optimization including environmental impacts of ultrasound-assisted extraction of phenolic antioxidants from blackcurrant pomace by-product

Doha KHALAF [1,2,*], Delphine PRADAL [1,2], Krasimir DIMITROV [1]

[1] UMR-T 1158 BioEcoAgro INRAe, Institut Charles Violette, Université de Lille, France.
[2] Icam, site de Lille, 6 Rue Auber, France.

***Corresponding author:** Doha KHALAF, doha.khalaf@icam.fr

Keywords: Green extraction, life cycle assessment, energy consumption, polyphenols, anthocyanins, blackcurrant pomace.

Objectives

This study seeks to integrate different aspects of eco-design: (a) Propose a way for a valorization of blackcurrant by-product by extracting antioxidant phenolic compounds; (b) Apply a green extraction process, namely ultrasound-assisted extraction (UAE), for antioxidants recovery; and (c) Optimize the UAE process using an original multi-criteria optimization tool considering in the same time the extraction yields, the antioxidant activity of the extracts, the energy consumption, and the environmental impacts of the process assessed by Life Cycle Assessment (LCA) methodology.

Methods

Extraction of antioxidant compounds is conducted by the use of UAE, then the analytical measurements are performed for sample characterization including Total polyphenols (TP), total anthocyanins (TA), and antioxidant activity (AA) tests. In addition, a multi-criteria optimization tool is used to optimize the UAE process and the Life Cycle Assessment (LCA) methodology using Simapro 9.4.0.2 software is exploited to assess the environmental impacts of the process.

Results

The obtained results showed that the blackcurrant pomace is a by-product rich enough in antioxidant phenolics (21.12 mg GAE.g^{-1} (d.b.)), especially in anthocyanins (5.45 mg CGE.g^{-1} (d.b.)) and that the UAE is an appropriated process for their recovery and for the valorization of this by-product by production of antioxidant-rich natural extracts. The ultrasound US assistance enabled to reach twice higher yields in TP, TA and AA compared to the extraction without the US. The detailed study of the effect of operating parameters on the extraction kinetics enables to find the right moment to stop the extraction process, save energy and time and reduce the environmental impacts (EIs). The used multi-criteria optimization model made it possible to estimate simultaneously the extraction yields TP and TA, the AA of the extracts, the energy consumption EC, and 28 EIs at each experimental condition. This model allowed for the optimization of the process under different constraints and to compare different scenarios. The detailed analysis of the environmental impacts using LCA could suggest options to decrease EIs without a significant decrease in productivity criteria (antioxidant activity and extraction yields).

Topic 3. Valorisation of by products and biorefinery

OR-22. Advanced drying methods and the emerging intensification of drying in the valorization of pumpkin by-products

Antonela NINČEVIĆ GRASSINO [1,*]

[1] University of Zagreb, Faculty of Food Technology and Biotechnology, Pierottijeva 6, 10000 Zagreb, Croatia.

***Corresponding author:** Antonela NINCEVIC GRASSINO, aninc@pbf.hr

Keywords: drying, emerging technologies, pre-treatments, pumpkin, by-products, quality

Objectives

The production of various agricultural products has increased rapidly due to consumer demand for fresh, high-quality food. However, among agricultural products, fruits and vegetables lose the most on the way from the field to the plate due to high water activity. Pumpkin is one such example. Once harvested, pumpkin with high water activity can only be kept for a few months. To minimize the water activity and preserve the chemical composition, drying processes are therefore a good choice. In combination with drying, innovative technologies such as ultrasound, high hydrostatic pressure, etc. can also be used as pre-treatment agents to intensify drying and maintain product quality.

Methods

The different analysis techniques and methods were used to evaluate the effect of hot air, vacuum and conductive drying as well as vacuum and hybrid hot air-microwave drying in combination with high hydrostatic pressure (50 and 500 MPa, 4 and 8 minutes) and ultrasound (amplitude of 60 and 90 %, 15, 30 and 45 minutes) to confirm and determine the suitability of processing parameters that do not alter the physicochemical and nutritional quality not only of the pumpkin pulp but also of the peel, which normally remains unused after consumption of the pulp in raw or processed form.

Results

The technologies used led to an improvement in the quality of the dried pulp and peel, while at the same time increasing the efficiency and capacity of the production processes. However, different settings of the respective technologies have a significant impact on the final quality of the pumpkin, and which of the drying processes and pre-treatments are preferable depends on the specific analytes and further use of the pumpkin.

Acknowledgements: The work was supported by the Croatian Science Foundation (research project "Hybrid drying and valorization of plant food waste and by-products" IP-2019-04-9750) - HYDRYBY.

OR-23. Impact of time, temperature, and pH on hydrothermal treatment for hemicellulose extraction from fir sawdust and characterization by capillary electrophoresis

Nicole NAJJOUM [1,2,*], Mohammed BENALI [1], Patrice CASTIGNOLLES [2], Nabil GRIMI [1]

[1] Université de technologie de Compiègne, ESCOM, TIMR (Integrated Transformations of Renewable Matter), Centre de recherche Royallieu - CS 60 319 - 60 203 Compiègne Cedex, France.
[2] Sorbonne University - Paris Institute of Molecular Chemistry, UMR 8232, Polymer Chemistry, Pierre and Marie Curie Campus, 75252 Paris, France

***Corresponding author:** Nicole NAJJOUM, nicole.najjoum@utc.fr

Keywords: Fir sawdust, Extraction, Hemicelluloses, Characterization, Capillary electrophoresis.

Abstract

Humanity faces a major challenge: managing limited resources while dealing with rapid population growth. Our global economy relies on non-sustainable technologies that depend on petrochemical raw materials, raising economic, environmental, and political concerns. It is crucial to invest in renewable energy sources to reduce our excessive dependence on fossil fuels by 2030 [1]. Furthermore, agriculture and agro-industry generate vast amounts of waste, harming the environment. Agricultural waste, particularly lignocellulosic biomass, could be used more efficiently to produce energy and high-value-added products. Biorefineries, which transform this biomass into useful products, are essential to achieve this goal. Hemicelluloses, abundantly present in wood, are promising materials for various applications, including film production [2]. However, to maximize their use, it is necessary to optimize the hemicellulose extraction process, considering experimental parameters and the complex chemical characteristics of these polymers. After isolation, various analytical techniques for hemicellulose characterization are available and can be categorized into separation, characterization, and spectroscopic methods. Spectroscopic techniques include Infrared (IR) and Nuclear Magnetic Resonance (NMR) spectroscopies. Separation methods, such as High-performance Anion Exchange Chromatography/Pulsed Amperometric Detection (HPAEC/PAD), Mass Spectrometry (MS) or capillary electrophoresis (CE) lead to the average chemical composition of hemicelluloses from complex biomass matrices after depolymerizing them, assuming no degradation. Separation and characterization of intact hemicelluloses are currently not employed to characterize chemical composition, including its heterogeneity (dispersity). Novel methods are essential to further explore the complexity, diversity, and heterogeneity of these polymers.

The aim of this study is to efficiently extract hemicelluloses from fir sawdust using a neutral (H_2O) and basic medium (1M NaOH) without physicochemical activation. Extraction time, temperature and pH were varied. Experiments were conducted using a high-temperature and high-pressure reactor (Parr reactor). Extraction parameters included different durations (10, 20, 30, and 60 minutes) and temperatures (120, 150, 180, and 210°C). The fir sawdust used had a particle size of 0.8 to 1.1 mm. The solid/liquid ratio was 1/15 (w/w). After extraction, separation and purification, hemicelluloses obtained under different conditions were characterized. One of those characterization methods is Capillary electrophoresis.

This work allowed us to determine the extraction time ranges and treatment temperatures that result in the highest yield of hemicellulose polymer in two different media (neutral and basic). The findings of this study showed a strong interaction between these extraction parameters, with the most remarkable yield at 180°C for 10 minutes in a neutral medium and 180°C for 30 minutes in a basic medium. In addition, in this research, we present a novel capillary electrophoresis (CE) approach for

the detection of neutral and non-UV-absorbing extracted polysaccharide, using an alkaline electrolyte solution (pH 12.6) at 266 nm. The results mark the first successful hemicellulose detection and determination of their heterogeneity. This study demonstrates CE's potential for examining structural changes of hemicelluloses under different extraction conditions and proposes a unique detection method without any derivatization step.

References

[1] F. Cherubini, "The biorefinery concept: Using biomass instead of oil for producing energy and chemicals," Energy Convers Manag, vol. 51, no. 7, pp. 1412–1421, Jul. 2010, doi: 10.1016/j.enconman.2010.01.015.
[2] Y. Lu, Q. He, G. Fan, Q. Cheng, and G. Song, "Extraction and modification of hemicellulose from lignocellulosic biomass: A review," Green Processing and Synthesis, vol. 10, no. 1. De Gruyter Open Ltd, pp. 779–804, Jan. 01, 2021. doi: 10.1515/gps-2021- 0065.

OR-24. Optimisation of proteins extraction from *Tenebrio meliotor* larvea

Cherif JABLAOUI [1,*], Nadia BOUSSETTA [1] et Houcine MHEMDI [1]

[1] Université de technologie de Compiègne, ESCOM, TIMR (Integrated Transformations of Renewable Matter), Centre de recherche Royallieu - CS 60 319 - 60 203 Compiègne Cedex, France.

***Corresponding author:** Cherif JEBLAOUI, cherif.jablaoui@utc.fr

Keywords: proteins, extraction, PEF, DIC, , kinetic modelling.

Objectives

With the world's population predicted to reach 9 billion by 2050, it is essential to find new sustainable agricultural practices and protein sources for the feed and food sectors. Edible insects are promising alternative biomass that can meet the growing demand for proteins, presenting many advantages over conventional farming, such as minimal resource needs, low environmental impact, low feed conversion ratio and above all their integration into a logic of circular economy. This study aims to the extraction of protein extraction from insect larvae by varying the operating extraction conditions. Response surface methodology was used to optimise the extraction conditions. The model of Crank was used to model the extraction kinectics.

Methods

To optimize alkaline method for extracting proteins from insect meal, larvea are first blanched and then defatted using hydraulic press to obatin a dry meal rich in proteins (49.5%). Proteins extraction was then studied and optimised by varying the temperature (20 to 60°C) and the pH (7 to 12.5). TheL/S ratio and the sterring velocity was fixed to 1/10 and 500 rpm respectively. The extraction kinectics were followed and the extraction yield was calculated.

Results

The main results showed that both temperature and pH has a significant factor affecting solubility and recovery of protein. A simultaneous increase of pH and temperature resulted in increased protein extraction yield. The high experimental extraction yield (67.08 %) was obatianed at pH=12.5 and T=60°C. Cranck model was used to model the extraction kinetics in order to calculate the effective diffusivity. It was observed that the applied model does not fit the experimental for the beginning of extraction (10 min) which corresponds to the external mass transfer. For the diffusional mass transfer (t>10 min), the model fitted well the experimental data. The effective diffusivity coefficient Deff was increased from 0.96 10^{-12} m²/s at T=26°C and pH = 7.8 to 5.59 10^{-12} m²/s at T=40°C and pH = 12.5.

OR-25. Economic and environmental impact analysis of innovative cascaded extraction of lycopene and cutin from tomato processing residue

Giovanni LANDI [1], Miriam BENEDETTI [2], Elham ESLAMI [3], Gianpiero PATARO [3,*]

[1] ENEA, Portici Research Center, Piazzale Enrico Fermi, Località Granatello, 80055 Portici, Italy.
[2] ENEA, Casaccia Research Center, Via Anguillarese 301, 00123 Rome, Italy.
[3] Department of Industrial Engineering, University of Salerno, Via Giovanni Paolo II, 132, 84084, Fisciano, SA, Italy

***Corresponding author:** Gianpiero PATARO, gpataro@unisa.it

Keywords: Tomato processing residues; Pulsed electric fields (PEF); Cascade extraction; Lycopene; Cutin; Payback Period; Environmental impact

Objectives

Within the framework of the 2030 Agenda for Sustainable Development, the valorization of agri-food waste is crucial in addressing global challenges and promoting sustainable practices. Adopting innovative waste valorization based on a cascade approach allows for the efficient and sustainable utilization of agri-food residues by sequentially recovering valuable compounds from a single waste stream.

Methods

This study focused on the cascade extraction of two valuable compounds, lycopene and cutin, from industrial tomato processing residue. It evaluated the economic and environmental impact of integrating innovative extraction technologies, such as pulsed electric field (PEF)-assisted extraction, into a conventional cascaded extraction process. An economic analysis was conducted using two main indicators: Net Present Value (NPV) and Payback Period. By also considering environmental indicators, such as the CO2 emission factor, the study assessed the economic and environmental feasibility of investing in PEF technology.

Results

The analysis, based on mass and energy balances, literature review, industry websites, and experimental data, enabled the evaluation of operational and installation costs for both solutions. Results showed that investing in PEF technology leads to a higher extraction yield of lycopene, a more valuable compound than cutin, with an annual revenue increase of 30% compared to a traditional extraction line (Control). Despite the higher initial investment, a PEF extraction line has a Payback Period of about 1.13 years compared to 1.6 years for a Control line. The real difference lies in the profit margin, with an NPV that, after 10 years, will be about 35% higher for a PEF investment.

It was also observed that investing in PEF technology allows for a higher extraction yield of lycopene, a more valuable compound than cutin. This translates into an annual revenue increase of 30% compared to what can be obtained from the sale of lycopene extracted with a traditional line (Control). Although PEF technology requires a higher initial investment, it has a Payback Period of about 1.13 years compared to 1.6 years for a Control line. The significant difference is in the profit margin, as evidenced by an NPV that, after 10 years, is 35% higher for the PEF technology investment.

These conclusions were confirmed by the analysis of three alternative scenarios, considering the energy crisis and the consequent increase in electricity and fuel prices. For the specific line under examination, although no substantial difference in terms of operational costs and environmental impact emerges, it is evident that applying PEF technology, compared to traditional methods, can be a significant solution for increasing revenue and efficiency.

Session I: Innovative approaches for biomass valorization: from extraction to sustainable applications

OR-26. Standardization of plant biomass and valorization of post-extraction residues by way of an innovative mobile pelletizing technology

Eléa COLLANGE [1,2], Charline COLLET [1], Thibaud MARCHAIS [2], Grégory CHATEL [1,*]

[1] Univ. Savoie Mont Blanc, EDYTEM, CNRS, 73000 Chambéry, France. (2) PEPS SAS, Bourget-du-Lac, France.

***Corresponding author:** Grégory CHATEL, gregory.chatel@univ-smb.fr

Keywords: grinding, drying, pellets, cascade valorization, scale-up, mobility

Objectives

Collection and pretreatment of biomass are the crucial initial stages of all valorization chains, significantly influencing subsequent extraction efficiency. It is essential for the input material to be stable and standardized to ensure consistent parameters and optimal process performance. However, biomass presents challenges due to its highly variable composition, high moisture content, low density and tendency for spontaneous degradation. These factors make it a difficult resource to manage. This thesis aims to develop a pretreatment method for plant biomass using a mobile pelletizing unit. The proposed technology involves four key steps: grinding, drying, additivation and densifying. The mobile granulation process, in particular, offers several benefits:

- Homogenizes and fractionates the material, increasing the specific surface area of the particles and the extraction interface.
- Removes moisture and prevents microbiological contamination early in the chain.
- Densifies the biomass directly at source, reducing its volume and consequently lowering transportation costs and carbon footprint

Additionally, the thesis seeks to valorize post-extraction residues by transforming them into advanced fertilizers in pellet form, achieved by combining them with mineral residues and beneficial microorganisms.

Methods

The biomasses studied in this research were vine roots and coffee grounds. The latter were collected in representative batches ranging from 5 to 250 kg. These were then processed using a FRITSCH P19 cutting mill and sieved to achieve a particle size distribution between 4 mm and 200 µm. The batches were dried using various technologies, including hot air flow, fluidized bed drying and lyophilization. Different temperatures and drying speeds were employed to achieve a final moisture content of 10-15%. After drying, the biomass was compressed using the SMARTWOOD PLT100 pellet mill with varying compression rates, utilizing a 6 mm flat die. The resulting pellets were subjected to extraction by a reflux method, using a solvent mixture of 60/40 EtOH/H2O at 60°C for 45 min. The extracts obtained from this process were analyzed for Total Phenolic Content (TPC) and DPPH radical scavenging activity.

Results

Throughout the thesis, the influence of pelletizing parameters - namely grinding, drying, and compression - on the antioxidant activities and extraction efficiency of polyphenols was thoroughly investigated. The underlying mechanisms involved in these processes were studied at a molecular scale,

focusing on evaporation phenomena, friction forces, and mechanochemistry. Regarding the development of pellet fertilizers, various assays were conducted to control and optimize key properties such as air porosity, water-holding capacity, nutrient release rate, and the carbon-to-nitrogen (C/N) and nitrogen-phosphorus-potassium (N/P/K) ratios. These tests aimed to enhance the efficacy and sustainability of the bio-based fertilizers produced from the residues.

OR-27. Ultrasound-assisted green extraction of active compounds from citrus fruits: chemical composition and biological activity against skin-aging enzymes

Houda EL KAHIA [1,*], Ezzouhra EL MAAIDEN [1], Majid MOUNIR [(2)], Lamfeddal KOUISNI [(1)], Youssef EL KHARRASSI [(1)]
[(1)] African Sustainable Agriculture Research Institute (ASARI), Mohammed VI Polytechnic University (UM6P), Laâyoune, Morocco
[(2)] Biotechnology and Biotransformation Laboratory, Department of Food Science and Nutrition, Hassan II Institute of Agronomy and Veterinary Medicine, Rabat, Morocco

***Corresponding author:** El Kahia Houda, **Houda.ElKahia@um6p.ma**

Keywords: Citrus extracts, polyphenols, flavonoids, antioxidant, anti-aging

Objectives

This study aimed to examine the effectiveness of citrus aqueous extracts derived from three Moroccan citrus fruit varieties by comparing the inhibitory activities of the peel and pulp extracts against skin aging-related enzymes. Antioxidant activities and chemical composition were also investigated.

Methods

The biomass was provided by farmers in the province of Kenitra, Morocco. The aqueous extracts were generated using ultrasound-assisted extraction method for each variety. The optimized extraction conditions were applied to enhance the recovery yield of biomolecules [1]. Citrus peel and pulp extracts were evaluated for their total phenolic and flavonoid contents by spectrophotometric methods, as well as their antioxidant activities involving DPPH and FRAP assays. The anti-aging activities were determined through *in-vitro* tests on three key enzymes: elastase, tyrosinase, and hyaluronidase.

Results

The comparative study between citrus fruit parts of different Moroccan varieties showed that the highest phenolic content was observed in the peels, in contrast to flavonoids, whose highest content was found in the pulp for all tested varieties. All the samples showed remarkable antioxidant and anti-aging activities. The pulp extracts showed the best activity against the three enzymes implicated in the anti-aging activities. Our results indicate the potential of citrus fruit parts and their possible use in cosmetics formulation and skincare. As perspectives, other additional analyses will be considered to (i) identify the main bioactive compounds using advanced analytical tools, and (ii) evaluate their pharmacological properties.

References

[1] El Maaiden, E., Qarah, N., Ezzariai, A., Mazar, A., Nasser, B., Moustaid, K., ... & El Kharrassi, Y. (2023). Ultrasound-assisted extraction of isoquercetin from *Ephedra alata* (Decne): optimization using response surface methodology and in vitro bioactivities. Antioxidants, 12(3), 725. https://doi.org/10.3390/antiox12030725

OR-28. Enrichment of refined sunflower oil with phenolic extracts from argan (*Argania spinosa* L. (skeels)) co-products using ultrasound-assisted extraction

Otmane HALLOUCH [1,2,*], Bouchra BOUNASSER [1], Moussa NID AHMED [1], Samira OUBANNIN [1], Abderrahim ASBBANE [1], Majourhat KHALID [1,2] and Said GHARBY [1]

[1] Biotechnology, Analytical Sciences and Quality Control Team, Polydisciplinary Faculty of Taroudant, University Ibn Zohr, Agadir, Morocco.
[2] Geo-Bio-Environmental Engineering and Innovation Laboratory, Molecular Engineering, Biotechnology and Innovation Team, Polydisciplinary Faculty of Taroudant, University Ibn Zohr, Agadir, Morocco.

***Corresponding author:** Otmane HALLOUCH, hallouch.o@gmail.com

Keywords: Enrichment, antioxidants, valorization, argan, co-products, sunflower oil, ultrasound

Objectives

The objective of our study is to investigate the antioxidant capacity, chemical profile, and potential of argan co-products extracts to extend the shelf life of refined sunflower oil.

Methods

The ability of ethanolic extracts extracted by ultrasound assisted extraction from argan co-products to improve the oxidation stability of refined sunflower oil under accelerated aging conditions was examined at 60°C for 90 days and by Rancimat test at 283.15 and 293.15 K. For this purpose, the oxidative stability was evaluated for oil supplemented with co-product extracts (at 100 ppm and 200 ppm %) and compared to a negative control (refined oil) and a positive control with vitamin E at the same concentrations (100 ppm and 200 ppm %). The progression of oxidation was monitored by measuring quality indices such as peroxide value, acidity, p-anisidine value, specific extinctions (K232 and K270), iodine value, pigment content (chlorophyll and carotenoids), fatty acid composition, antioxidant activity using DPPH, FRAP, and ABTS assays, and phenolic composition including polyphenols and flavonoids.

Results

Our results reveal that argan co-products are significant sources of proteins, carbohydrates, fibers, minerals, phenolic compounds, and flavonoids, and they exhibit high antioxidant capacity. Regarding oil stability, the results showed that oils enriched with various co-products had higher stability compared to both the negative and positive controls. We can conclude that argan co-products represent a promising natural antioxidant, providing an alternative to synthetic antioxidants for improving the oxidative stability of edible oils.

OR-29. Unlocking the potential of enological by-products: a cascade approach for comprehensive biomass valorization through sustainable technologies

Emanuela CALCIO GAUDINO [1,*], Silvia TABASSO [1], Carlotta VALLE [1], Giorgio GRILLO [1], Paola PONSETTO [2], Roberto MAZZOLI [2], Enrica PESSIONE [2], Emilia GARCIA-MORUNO [3], Antonella COSTANTINI [3], Giancarlo CRAVOTTO [1]

[1] University of Turin, Dep. of Drug Science and Technology, Via P. Giuria 9, 10125, TO, Italy.
[2] University of Turin, Dep. of Life Sciences and Systems Biology, Via A. Albertina 13, 10123 TO, Italy.
[3] Research Centre for Viticulture and Enology CREA-VE, Via P. Micca 35, 14100 (AT), Italy.

***Corresponding author:** Emanuela CALCIO GAUDINO, emanuela.calcio@unito.it

Keywords: Agri-food waste; grape stalks; ultrasound; microwave; polyphenols, cellulose fermentation, lactic acid, levulinic acid, circular economy

Objectives

The wine industry, a key part of Italy's agri-food sector, produces significant by-products like grape stalks (GS), which are costly to dispose of but can serve as valuable, low-cost feedstock for biorefineries. This study proposes a sequential GS valorization protocol featured by a multidisciplinary strategy that combines unconventional techniques, green solvents, and biological approaches. The early stage involved sequential GS fractionation, yielding product streams enriched first in polyphenols, hemicellulose and pectins, and then in lignin, and cellulose. During the cascade process, ultrasound (US) and microwave (MW) technologies were both exploited for polyphenols recovery, harnessing natural deep eutectic solvents (NADES) and subcritical water [1,2]. The post extracted solid fraction so obtained was then delignified under alkaline conditions. The cellulosic fraction thus recovered was tested as substrate for both (i) fermentation into lactic acid (LA) and (ii) catalytic conversion into levulinic acid (LevA). Finally, the recovered phenolic fraction was tested against *Brettanomyces bruxellensis* and *Acetobacter pasteurianus*, key wine spoilage microorganisms. This cascade protocol could represent a promising example of a biorefinery approach for GS, aligning with Green Chemistry and Circular Economy principles.

Methods

GS of different cultivars from Piedmont region (Nebbiolo, Barbera, Dolcetto) were provided by CREA-VE (Asti IT). The US-assisted extraction (UAE) of raw GS has been performed in NADES (Choline Cloride-LevA 1:2) at 40 KHz and 200 W (60 min).

The MW-assisted extraction (MAE) of raw GS has been performed in water under subcritical condition (150°C and 5 bar of N_2 pressure) using a multimode MW reactor (Milestone S.r.l., Italy). Both UAE and MAE extracts were characterized in term of total phenolic content (TPC) and antioxidant activity by means of Folin-Ciocalteu and DPPH assay. The subsequent delignification of the so recovered GS solid fraction was performed under MW (150°C, 120 min, 5 bar N_2) in alkaline conditions to collect the GS cellulose enriched fraction. Engineered *Clostridium thermocellum* strains, (*C. thermocellum* LL1111 and LL1630) [3] were exploited for the cellulose fraction fermentation into LA. While a fast catalytic conversion of the cellulose-rich fraction into LevA was investigated under acidic condition in MW (225°C, 2 minutes, under N_2 pressure of 40 bar). [4]

Results

This work optimized a sustainable biorefinery process for GS valorization, following a zero-waste approach. The cascade fractionation yielded a polyphenol enriched fraction, lignin, and cellulose exploiting sustainable technologies coupled with green solvents. The best polyphenol recovery, exploiting subcritical water MAE, yielded 185.78 mgGAE/gExtr and 75.99 mgGAE/gMatr in only 7.5 minutes. The delignified, cellulose-enriched fraction was then used to produce LA with engineered *C. thermocellum* strains, resulting in a 30% yields of the theoretical maximum. In parallel, the MW-assisted catalytic conversion of the same cellulose fraction enabled an 85% molar yield of LevA. Remarkably, the phenolic fraction effectively acted as a biocide against wine spoilage microorganisms. This study demonstrates the complete valorization of GS using advanced green extraction methods, eco-friendly solvents, and biotechnological processes. The resulting products have potential applications in the cosmetic, nutraceutical, and winemaking sectors, aligning with a Circular Economy approach. Globally, this study represents a proof-of-concept of a 2nd generation biorefining process based on locally available biomass.

References

[1] Grillo, G., Boffa, L., Talarico, S., Cravotto G. (2020) Batch and Flow Ultrasound-Assisted Extraction of Grape Stalks: Process Intensification Design up to a Multi-Kilo Scale. Antioxidants 10;9(8):730. doi: 10.3390/antiox9080730.

[2] Grillo, G., Capaldi G., Radošević K., Jakopović Ž, Markov K., Brncic M., Gallina L., Calcio Gaudino E., Cravotto G. (2023) Unlocking the Bioactive Potential of Pomegranate Peels: A Green Extraction Approach. Antioxidants, 12, 1796. doi: 10.3390/antiox12101796.

OR-30. Kiwi peel valorization: optimized phenolic content extraction and bioactivity assessment

Nicolas LOUKA [1,*], Batoul SHKEIR [2], Bilal AZAKIR [3], Elie Salem SOKHN [4], Richard G. MAROUN [1], Espérance DEBS [5], Nada EL DARRA [6]

[1] Centre d'Analyses et de Recherche, Unité de Recherche Technologies et Valorisation Agro-alimentaire, Faculté des Sciences, Saint -Joseph Université, P.O. Box 17-5208, Riad El Solh, Beirut 1104 2020, Lebanon.
[2] Department of Nutrition and Dietetics, Faculty of Health Sciences, Beirut Arab University, Tarik El Jedidah, Riad El Solh, P.O. Box 115020, Beirut 1107 2809, Lebanon.
[3] Molecular and Translational Medicine Laboratory, Faculty of Medicine, Beirut Arab University, Beirut 1107 2809, Lebanon.
[4] Department of Medical Laboratory Technology, Faculty of Health Sciences, Beirut Arab University, Beirut 1107 2809, Lebanon.
[5] Department of Biology, Faculty of Arts and Sciences, University of Balamand , P.O. Box 100, Tripoli, Lebanon.
[6] Department of Nutrition and Dietetics, Faculty of Health Sciences, Beirut Arab University, Tarik El Jedidah, Riad El Solh, P.O. Box 115020, Beirut 1107 2809, Lebanon.

***Corresponding author:** Nicolas LOUKA, nicolas.louka@usj.edu.lb

Keywords: Kiwi peels, Infrared-assisted extraction, Optimization, Total phenolic content, biomolecules

Objectives

Kiwi peels, a by-product of kiwi production, are often considered waste. However, they contain a significant amount of flavonoids, insoluble fibers, antioxidants, and biomolecules with anti-inflammatory and antiallergenic properties. This study aims to optimize the extraction of phenolic compounds from kiwi peels using water-bath extraction (WBE) and infrared-assisted extraction (IRAE). Additionally, the antimicrobial and anticancer activities of the two extracts were assessed, and their phytochemical profiles were analyzed.

Materials and Methods

Response Surface Methodology (RSM) was used to determine the optimal parameters for polyphenol extraction. Two experimental parameters, time and temperature, were tested. Total phenolic content antiradical activity were measured using Folin-Ciocalteu and DPPH method, respectively. The disc diffusion method and minimum inhibitory concentration (MIC) assays were employed to evaluate the antimicrobial activity of the two extracts against Escherichia coli, Salmonella Typhimurium, Staphylococcus aureus, and Bacillus cereus. The anticancer activity of the lyophilized extract was assessed against A549 lung cancer cell lines. High-Performance Liquid Chromatography (HPLC) was used to analyze the phytochemical profiles of the WBE and IRAE extracts.

Results

The optimal conditions for polyphenol extraction were determined to be 75°C for 35 minutes for WBE, and 90°C for 11.5 minutes for IRAE. The yield of TPC obtained under optimal conditions was significantly higher for IRAE (13 mg GAE/g DM) compared to WBE (4.8 mg GAE/g DM), indicating that IRAE is more efficient for phenolic extraction from kiwi peels and can save energy. The maximum antioxidant activity measured was 11.36 mg trolox equivalents/mL for WBE and 10.26 mg trolox equivalents/mL for IRAE at 90°C for 40 minutes. Both extracts exhibited antibacterial activity against Bacillus cereus, while the IRAE extract also demonstrated activity against Staphylococcus aureus. No antibacterial effect was observed on gram-negative bacteria. The anticancer activity assessment showed

a significant 30% reduction in A549 lung cancer cells. Notably, catechin (5.44 ppm) was identified as a major polyphenol in the IRAE extract but was absent in the WBE extract. In conclusion, IRAE was found to enhance both the quantity and quality of polyphenols extracted from kiwi peels compared to WBE. These findings support the potential use of kiwi peels as a valuable source of antioxidants, antibacterial, and anticancer agents.

Session II: Advances in lignin and biomass valorization: from edible films to high-performance materials

OR-31. Evaluation of the influence of lignin nanoform on properties of edible films

Zhao ZHANG [1], Rita ARGENZIANO [2], Adama KONATE [1], Xiangru SHI 1, Sarai AGUSTIN SALAZAR [3], Pierfrancesco CERRUTI [3], Lucia PANZELLA [2,*], Vincent TERRASSON [1,*], Erwann GUÉNIN [1,*]

[1] Université de Technologie de Compiègne, ESCOM, TIMR (Integrated Transformations of Renewable Matter), Centre de Recherche Royallieu-CS 60 319-60 203 Compiègne Cedex, France.
[2] Department of Chemical Sciences, University of Naples "Federico II", Via Cintia 4, I-80126 Naples, Italy.
[3] Institute for Polymers, Composites and Biomaterials, National Research Council of Italy, Via Campi Flegrei 34, 80078 Pozzuoli (NA), Italy.

*** Corresponding authors:** Erwann GUÉNIN, erwann.guenin@utc.fr, Vincent TERRASSON, v.terrasson@escom.fr, and Lucia PANZELLA, panzella@unina.it

Keywords: Chitosan, Lignin nanoparticles, Composite films, Antioxidant properties, Food packaging

Objectives

As the second most abundant natural polymer material after cellulose, lignin has received extensive attention in recent years and the development of bio-based products from lignin is a growing domain because of their biocompatibility and biodegradability [1]. Lignin nanoparticles (LNP) have potential applications in antioxidants, thermal/light stabilizers, reinforced materials and nanocarriers owing to their advantages of non-toxicity, environmental resistance, excellent thermal stability and biocompatibility [2]. Recently we proposed a new synthesis of LNP with a control over size and a high dispersion and stability in water [3]. Here we proposed their utilization to enhance the property of chitosan bioplastic films. Following previous work on the production of chitosan-cellulose bioplastic films and evaluation of the use of nanoparticles of these biopolymers to enhance various properties [4] we proposed to compare the utilization of LNP vs lignin and evaluate their influence on film properties.

Methods

The lignin nanoparticle (LNP) were prepared as recently described from alkali lignin [2]. Different ratios of nanolignin stable dispersion in water 0.25 %, 0.5 %, 1 % were evaluated. Nanochitosan (NCH) particles were synthesized by the ionotropic gelation method using sodium tripolyphosphate following our previously described protocol [4]. The morphology and size of nanoparticle samples were characterized by transmission electron microscopy (TEM). Hydrodynamic diameter as well as zeta potential were measured by Dynamic Light Scattering measurement (DLS) over several days to assess nanoparticle stability. FTIR and NMR were used to characterized LNP and compare it to the parent lignin. The film composite films were prepared as described previously [4] from a dispersion of NCH and LNP or lignin by the solvent casting methodology. Nanocomposites films were characterized for their morphology by Scanning Electron Microscopy and Thermo Gravimetric Analysis was used to study their thermal stability. FTIR and X-Ray diffraction were utilized to study their structure. Redox properties were measured by various standard assays. UV absorbance properties and opacity measurement were also evaluated by UV spectrometry. And finally,

the food preservation effect of prepared films was studied in model experiments on grape and cheese samples.

Results

LNP were prepared with controlled size and affording highly stable LNP water suspension with long term stability. They were used at different concentrations as filler for chitosan bioplastic films and compared to the use of untreated lignin. Preparation of biocomposite by solvent casting yield uniform films of few micrometer width and a smooth surface. Surface morphology was improved by addition of LNP compared to chitosan alone or addition of lignin. The FTIR show a tight interaction between both nanoforms (NCH and LNP) in the films and presence of LNP in the formulation improves crystallinity of the biocomposite films. Utilization of LNP rather than untreated lignin permit to greatly enhance UV adsorption of the film as well as improve their redox properties. LNP has also less impact on film transparency and color than lignin.

Therefore, we showed that properties were enhanced using the nanoform of lignin. These results are currently extrapolated to different sources of lignin to evaluate the influence of lignin origin.

References

[1] Becker, J., Wittmann, C.: A field of dreams: Lignin valorization into chemicals, materials, fuels, and health-care product, Biotechnol. Adv., 2019, 37, 107360.

[2] Zhang, Z., Terrasson, V., Guénin, E.: Lignin Nanoparticles and Their Nanocomposites, Nanomaterials, 2021, 11(5), 1336

[3] Zhang, Z., Belda Marin, C., Lefebvre, M., Lefebvre, C., Terrasson, V., Guénin, E.: The preparation of stable spherical alkali lignin nanoparticles with great thermal stability and no cytotoxicity, Int J. Biol. Macromolec., 2022, S0141-8130(22)02212-7. https://doi.org/10.1016/j.ijbiomac.2022.09.272

[4] Pinem, M.P., Wardhono, E. Y., Nadaud, F., Clausse, D., Saleh, K., Guénin, E.: Nanofluid to nanocomposite film: chitosan and cellulose-based edible packaging, Nanomaterials, 2020, 10, 660.

OR-32. Optimizing extracted lignin from deproteinated brewer's spent grains for chemical valorization.

Sarah-Joe SALAMEH [1,*], Anissa KHELFA [1], Estelle LEONARD [1]

[1] Université de technologie de Compiègne, ESCOM, TIMR (Integrated Transformations of Renewable Matter), Centre de recherche Royallieu - CS 60319 - 60203 Compiègne Cedex, France.

Corresponding author: Sarah Joe Salameh, sarah-joe.salameh@utc.fr

Keywords: Brewer's spent grains, proteins, lignin, microwaves, ultrasound, design of experiment.

Objectives

Beer has been one of the world's most appreciated and consumed fermented beverages since ancient times [1]. Brewer's spent grains (BSG) represent a prominent by-product of the brewing industry. This by-product is characterized by its high moisture content (about 80 wt%) and a composition rich in proteins, minerals, and bio-polymers, including lignin, cellulose, and hemicelluloses [2]. Following the extraction of proteins, the primary objective is to explore the most effective method for lignin extraction using the Organosolv process and subsequently investigate its chemical valorization through diazo couplings. After optimizing the lignin extraction process to maximize its yield and purity, the diazo coupling will enhance the extracted lignin chemically. Azobenzene, a compound present in up to 70% of all organic dyes globally, is one of the most commonly used photochromes [3]. It has various applications, including its use in antimicrobial compounds. One of the simplest ways to synthesize azobenzene compounds involves coupling natural phenolic molecules with anilines in water. However, pure lignin and macromolecules containing phenols are also suitable materials for synthesizing azobenzene. In this context, the objective is to graft purified lignin with azobenzene to impart antimicrobial properties.

Methods

Proteins were extracted from the dried and ground BSG sample using ultrasonication in 110 mM NaOH. Microwave-assisted fractionation in an organic acid aqueous solution was then employed to isolate lignin from deproteinized BSG. An optimization matrix was developed using response surface methodology with a central composite design. This method was employed to systematically evaluate and enhance the extraction process by examining several critical factors: the extraction duration and the liquid-to-solid ratio (L/S), while keeping the process temperature and solvent composition constant. Subsequently, the extracted lignin was characterized using NMR and FTIR spectroscopy, along with an assessment of lignin purity. The antibacterial properties of the lignin extracts and the azobenzene formed were evaluated using a modified version of the clinical and laboratory standards institute (CLSI) broth microdilution technique. For the functionalization of the extracted lignin, various aniline compounds were employed to synthesize azobenzene derivatives. Elemental analysis was performed to measure the nitrogen content incorporated into the functionalized polymers to determine the degree of functionalization.

Results

For protein extraction, a yield of 72.22 % was obtained using the ultrasound. After BSG fractionation, the maximum extracted lignin purity achieved was approximately 85%. FTIR results allowed for identifying key functional groups within the lignin structure. In addition, NMR analysis revealed significant signals in the aromatic region, including the monomeric units of lignin syringyl (S), guaiacyl (G), and p-hydroxyphenyl (H). In the context of lignin valorization, the degree of lignin functionalization with aniline was around 15%, compared to 20-30% achieved with commercial lignin

samples [4-5]. Future experiments will enhance this grafting rate and evaluate the antimicrobial efficacy of the resulting azobenzene formed with lignin.

References

[1] F. stats 2019, “Production mondiale de la bière par pays.” https://www.atlasbig.com/fr-fr/pays-par-production-de-biere
[2] T. Bonifácio-Lopes et al., “Correction: Bioactive extracts from brewer’s spent grain,” Food Funct., 2021, doi: 10.1039/d1fo90072b.
[3] J. Wang, B. Wu, S. Li, G. Sinawang, X. Wang, and Y. He, “Synthesis and characterization of photoprocessable lignin-based azo polymer,” ACS Sustain. Chem. Eng., 2016, doi: 10.1021/acssuschemeng.6b00975.
[4] J. Wang, S. Li, R. Liang, B. Wu, and Y. He, “Synthesis and characterization of water-soluble PEGylated lignin-based polymers by macromolecular azo coupling reaction,” Chinese Chem. Lett., vol. 29, no. 1, pp. 143–146, 2018, doi: 10.1016/j.cclet.2017.07.008.
[5] Y. Deng, H. Zhao, Y. Qian, L. Lü, B. Wang, and X. Qiu, “Hollow lignin azo colloids encapsulated avermectin with high anti-photolysis and controlled release performance,” Ind. Crops Prod., 2016, doi: 10.1016/j.indcrop.2016.03.056.

OR-33. Enzymes and green solvents for the sustainable extraction of lignin from horse manure: toward a diversification of the biomethane value-chain.

Lindsay DORSCHNER PELCOQ [1], Clément DUMONT [2,3], Tiphaine RICHARD [2,3], Mathieu SAUTHIER [2], Catherine SARAZIN [1,*], Éric HUSSON [1,*]

[1] Unité de Génie Enzymatique Et Cellulaire GEC, UMR CNRS 7025, Université de Picardie Jules Verne, 33 Rue Saint-Leu, 80039 Amiens Cedex, France
[2] Unité de Catalyse Et Chimie du Solide UCCS, UMR CNRS 8181, Université de Lille, Centrale Lille, ENSCL, Université d'Artois, 59000 Lille Cedex, France
[3] Institut Catholique d'Arts Et Métiers (ICAM) Site de Lille, 6 Rue Auber, 59 016, Lille Cedex, France

***Corresponding authors:** Catherine SARAZIN, catherine.sazarin@u-picardie.fr and Éric HUSSON, eric.husson@u-picardie.fr

Keywords: Horse manure, Ionic liquid, Lignin, Organosolv, Telomerization, BMP

Objectives

To address the high lignin content of horse manure in the methanization process, eco-fractionation and chemical modification strategies of lignin have been proposed. This opening up prospects for new lignin-based thermoplastic materials while the sugar-enriched co-fractions were studied for anaerobic digestion and biogas production.

Methods

Two distinct green fractioning strategies were implemented: (i) a biochemical process consisting in a 1-ethyl-3-methylimidazolium acetate [Emim][OAc] pretreatment followed by enzymatic hydrolysis of polysaccharidic fractions catalyzed by the industrial cocktail Cellic®CTec 2 and (ii) the CoffeeCat process, a revisited organosolv process. The isolated lignins were chemically modified using palladium-catalyzed 1,3-butadiene telomerization while the sugar-enriched co-fractions werestudied in terms of biochemical methane potential. TGA, SEM analyses, 2D 1H-13C HSQC and 31P NMR were used for the thorough characterization of isolated lignins before and/or after telomerization reaction.

Results

The biochemical process allows to recover sugars after enzymatic hydrolysis and a lignin-enriched fraction named hydrolysis lignin was isolated (80% w/w) with ionic liquid. The CoffeeCat process allows selective extraction of a lignin-enriched fraction (> 80% w/w almost sugar-free) and a partially delignified sugar solid fraction. For both pathways, recyclability and biodegradability considerations were taken into account. The two lignins differed slightly in phenylpropane units but exhibited a similar preserved aryl-ether linkages content. The morphological, thermal and structural properties suggested promising valorization for lignin-based materials with a successful palladium-catalyzed telomerization on the organosolv lignin, whereas optimization is still required on hydrolysis lignin. Whatever the strategy used, carbohydrates fractions showed competitive biochemical methane potential for methanization.

OR-34. How far can catalysis leverage the transformation of amorphous biomass into crystalline carbon-based materials?

Mehdi MENNANI [1,*], Anass AIT BENHAMOU [2], Meriem KASBAJI [1], Siham ESSAHRAUI [3], Mounir EL ACHABY [1], Amine MOUBARIK [4], Zineb KASSAB [1]

[1] Materials Science, Energy and Nanoengineering (MSN) Department, Mohammed VI Polytechnic University, Lot 660 – Hay Moulay Rachid, 43150, Ben Guerir, Morocco.
[2] Department of Wood and Forest Sciences, Laval University, Quebec, QC G1V 0A6, Canada.
[3] Applied mathematics and information systems (MASI) laboratory, Mathematics and Computer Science (SMI) Department, Multidisciplinary Faculty of Nador, Mohammed Premier University, B.P. 524 Oujda 60000, Morocco.
[4] Chemical Processes and Applied Materials Laboratory, Polydisciplinary Faculty, Sultan Moulay Slimane University, BP 592, Beni Mellal, Morocco.

***Corresponding author:** Mehdi MENNANI, email: mehdi.mennani@um6p.ma

Keywords: Biomass, Lignin, Catalyst, Carbon, Graphite, Crystallinity, Energy.

Objectives

The primary aim of this research is to assess the effectiveness of catalysts in the conversion of amorphous biomass into carbon-based materials with a crystalline structure. The study evaluated the efficiency of various catalytic processes, analyzing the structural and chemical characteristics of the resultant crystalline products. Moreover, the study aims to identify the most optimal reaction conditions, including temperature, time, and catalyst concentration, to improve both yield and crystallinity.

Methods

The catalytic conversions were conducted under controlled settings, with a focus on manipulating key parameters to generate crystalline materials and utilizing Artificial Intelligence (AI) tools for investigation. The resultant carbon-based materials with a crystalline structure were analyzed through techniques such as XRD, FTIR, SEM, TEM, and Raman spectroscopy.

Results

The research revealed significant variations in the conversion rates of amorphous biomass and lignin into crystalline materials based on the reaction conditions. Extending the reaction duration (1-2 hours) and elevating the temperature (600-1000°C) proved to be more effective in achieving the desired conversion. However, an increase in these parameters seemed to impact the yield, especially under harsh reaction conditions. XRD and Raman indicated differing levels of crystallinity among the materials produced, while SEM and TEM offered detailed insights into their morphological characteristics. Optimal reaction conditions that maximized both yield and crystallinity were determined. The performance of these materials is under evaluation for potential applications in areas such as electrical conductivity, thermal stability, and mechanical strength, particularly in energy storage devices like batteries and supercapacitors.

OR-35. Innovative conversion of alfa plant cellulose nanocrystals into hard carbon anodes for sodium-ion batteries

Meriem KASBAJI [1], Sara NOUREDDINE [1], Mehdi MENNANI [1], Mustapha OUBENALI [2], Amine MOUBARIK [3], Zineb KASSAB [1], Mouad DAHBI [1], Mounir EL ACHABY [1]

[1] Materials Science, Energy and Nanoengineering (MSN) Department, Mohammed VI Polytechnic University, Lot 660 - Hay Moulay Rachid, 43150, Ben Guerir, Morocco.
[2] Laboratory of Engineering in Chemistry and Physics of Matter, Faculty of Science and Technologies, Sultan Moulay Slimane University, BP 523, Beni-Mellal, Morocco.
[3] Laboratory of Chemical Processes and Applied Materials, Polydesciplinary Faculty, Sultan Moulay Slimane University, BP 592, Beni-Mellal, Morocco.

***Corresponding author:** Meriem KASBAJI, Meriem.KASBAJI@um6p.ma

Keywords: Alfa plant; Cellulose nanocrystals; Hard carbon; Anode; Sodium-ion batteries

Objectives

Low-cost sodium-ion batteries (SIBs) are being extensively explored for large-scale applications due to the abundant availability of sodium resources. However, advancing SIB technology presents significant challenges, particularly in optimizing anode materials. To address this, we have developed hard carbon (HC) from cellulose nanocrystals (CNC), specifically sulfated CNC (S-CNC) derived from the widely available alfa plant, using a straightforward carbonization method. We conducted detailed studies on the electrochemical properties of these carbonaceous materials, establishing a clear correlation between pyrolysis temperature, microstructure configuration, and electrochemical performance. Our results indicate that the plateau strength of these anode materials is highly dependent on carbonization temperature, and that the nature and properties of the CNCs significantly influence the HC structure. The hard carbon sample pyrolyzed at 1300°C exhibited the highest reversible specific capacity, excellent rate capability, and outstanding cycle stability. These promising electrochemical properties highlight the potential of this type of hard carbon for the development of cost-effective, high-performance sodium-based anode materials.

Methods

Raw Alfa fibers (AF) were initially subjected to a thorough washing process with distilled water at 60°C for 1 hour under mechanical agitation. Following this pre-treatment, the fibers were treated twice with a 4 wt% NaOH solution at 80°C for 2 hours, with continuous stirring to ensure complete interaction. Subsequently, the alkali-treated Alfa fibers were bleached using a carefully prepared solution consisting of equal parts of acetate buffer (comprising 27 g NaOH and 75 mL glacial acetic acid diluted to 1 L of distilled water) and an aqueous sodium chlorite solution (1.7 wt% $NaClO_2$ in water). This bleaching procedure was repeated three times, each for 2 hours at 80°C, resulting in the production of cellulose microfibers (CMF).

The extraction of sulfated cellulose nanocrystals (S-CNC) from the prepared CMF was achieved through sulfuric acid hydrolysis. Specifically, CMF were introduced into a preheated sulfuric acid solution (64 wt%, 9 M) maintained at 50°C and subjected to mechanical stirring for 30 minutes. To terminate the reaction, the mixture was promptly diluted with ice cubes. The resulting mixture underwent successive centrifugations at 12,000 rpm and 15°C for 15 minutes per cycle to remove residual acid and by-products. This was followed by dialysis against distilled water until the mixture achieved a neutral pH. The final step involved homogenizing the CNC aqueous suspension using a probe-type ultrasonic homogenizer for 5 minutes in an ice bath. The product, freeze-dried S-CNC, is depicted in Fig. 1. Following the preparation of S-CNC, hard carbon (HC) materials (HC_S-CNC) were

synthesized by carbonizing the S-CNC at 800°C and 1300°C under an argon-nitrogen atmosphere for 2 hours. The electrochemical performance of the resulting hard carbon was then evaluated through a series of electrochemical tests to determine its suitability and efficiency as an anode material for sodium-ion batteries.

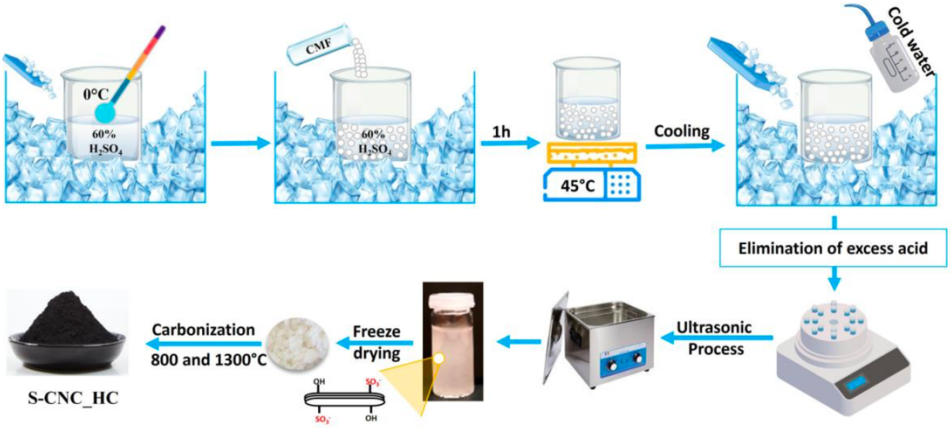


Figure 1. Illustration of the process involves extracting S-CNC and synthesizing HC from Alfa plant fibers.

Results

Cellulose Nanocrystals (CNC) Performance and Structure

Cellulose nanocrystals (CNCs) display exceptional mechanical characteristics and a high degree of crystallinity, rendering them appropriate for a variety of advanced applications, notably as reinforcing agents in nanocomposites. Research indicates that CNCs obtained from alfa plants retain these favorable attributes while also taking advantage of an environmentally sustainable and economically feasible production method. The notable level of sulfation present in CNCs derived from alfa plants amplifies their dispersibility in water and other polar solvents. This enhanced dispersibility proves particularly advantageous in electrochemical applications, where the uniform distribution of materials plays a crucial role in ensuring consistent performance. In the realm of electrochemical applications, the notable surface area, and superior structural characteristics of CNCs significantly contribute to their efficacy. Incorporating CNCs into composite materials leads to substantial enhancements in mechanical robustness, thermal endurance, and barrier properties. Moreover, CNCs can act as a precursor for generating top-notch carbon materials via carbonization procedures. The conversion of CNCs into hard carbon materials conserves their beneficial structural traits, thereby bolstering the exceptional electrochemical performance of the resultant hard carbon.

Hard Carbon (HC) from Cellulose Performance and Structure Carbonized at 800°C

Upon carbonization at 800°C, cellulose-derived materials yield hard carbon (HC) typically characterized by a more disordered structure featuring a notable quantity of micropores. This porous configuration proves advantageous in sodium-ion storage applications as it offers sufficient room for ion adsorption, thereby yielding moderate reversible capacities ranging from 250-300 mAg^{-1}. The substantial surface area and porosity of these hard carbons facilitate effective ion transport and storage, rendering them appropriate for scenarios where a moderate energy storage capacity suffices. Nevertheless, the disordered nature of HC carbonized at 800°C might hinder its electronic conductivity when compared to materials carbonized at elevated temperatures. Despite this drawback, the material showcases commendable cycling stability and rate performance, establishing it as a feasible choice for specific applications within sodium-ion batteries. The lower carbonization temperature also results in a streamlined and potentially more cost-efficient production process.

Carbonization conducted at a temperature of 1300°C

By contrast, the carbonization of materials derived from cellulose at 1300°C yields rigid carbons characterized by a higher level of graphitization. This heightened graphitization notably boosts the electrical conductivity of the substance, a crucial aspect for applications in high-performance batteries.

Carbons of high hardness produced under these conditions frequently demonstrate reversible capacities that exceed 300 mAh g^{-1}. For example, a study showcased a reversible specific capacity of 373.2 mAg^{-1} at 30 mAg^{-1}, alongside exceptional cycling stability and 89.7% capacity retention after 200 cycles. The superior performance of hard carbons carbonized at 1300°C can be attributed to the emergence of nanovoids and a more ordered carbon framework. These structural attributes facilitate enhanced sodium ion insertion and movement within the material, ultimately leading to improved electrochemical characteristics. Further analyses utilizing techniques such as cyclic voltammetry (CV) and galvanostatic intermittent titration technique (GITT) suggest that the mechanism of sodium storage in these rigid carbons is impacted by the carbonization temperature. The increased graphitization level and the existence of nanovoids contribute to a reduced apparent diffusion coefficient of sodium ions in the plateau area, thereby amplifying the rate performance of the electrode material.

Summary of Findings

The performance of hard carbon materials derived from cellulose in sodium-ion batteries is significantly impacted by the temperature of carbonization. While lower temperatures like 800°C yield materials with a more disordered structure and moderate capacities, higher temperatures such as 1300°C produce highly graphitized carbons with exceptional electrochemical properties. The capacity to finely adjust the structure and performance of these materials through controlled carbonization processes presents new opportunities for the development of cost-effective, high-performance sodium-ion batteries.

These findings underscore the potential of utilizing cellulose nanocrystals sourced from alfa plants as a precursor for hard carbon anode materials in sodium-ion batteries. The environmentally sustainable and economically viable production method, coupled with the outstanding electrochemical performance of the resultant hard carbons, emphasizes the feasibility of this approach for large-scale energy storage applications. Through optimization of the carbonization temperature, researchers can create customized materials that cater to the specific requirements of energy storage systems demanding high capacity and stability.

OR-36. Effect of phosphorylation treatment of lignocellulose obtained from Argan Shells on the mechanical, physical properties and formaldehyde emission of particleboard panels.

Hafida MAARIR [1,*], Yassine EL KHAYAT DRIAA [1,2], Abdelghani BOUSSETTA [3], Hassan CHARII [1], Nabil GRIMI [2] and Amine MOUBARIK [1,*]

[1] Laboratory of Chemical Processes and Applied Materials, Polydisciplinary Faculty, Sultan Moulay Slimane University, BP 592, Beni Mellal, Morocco.
[2] Université de technologie de Compiègne, ESCOM, TIMR (Integrated Transformations of Renewable Matter), Centre de recherche Royallieu - CS 60319 - 60203 Compiègne Cedex, France
[3] Materials Science, Energy and Nanoengineering (MSN) Department, Mohammed VI Polytechnic University, Lot 660 – Hay Moulay Rachid, 43150, Ben Guerir, Morocco.

***Corresponding authors:** Hafida MAARIR, hafidamaarir891@gmail.com and Amine MOUBARIK, a.moubarik@usms.ma

Keywords: Particleboard, wood adhesives, formaldehyde emission, Lignocellulose, Argan, Phosphorylation.

Objectives

The aim of this recent work is to exploit the unexplored potential of the abundant Moroccan by-product Argan Shell (AS) in order to develop new environmentally-friendly materials in various applications, particularly in particleboard adhesives, with a view to improving mechanical and physical properties and reducing formaldehyde emissions.

Methods

The processing of the AS modification to produce Phosphorylated Hydrolysed Argan Shell (P-HAS) was carried out in 2 steps, including hydrolysis and phosphorylation.

1st step; the hydrolysis process was carried out using hot water at 70°C during 2 hours in order to remove the hemicellulose and retain the lignocellulosic materials (HAS).

2nd step; 5 samples have been prepared with different ratios of HAS (g): Urea (g): DAP (g), and the phosphorylation reaction was carried out at 150°C for 1 hour.

Results

After obtaining the results of the various types of physical and chemical characterization (FTIR, SEM/EDX, conductivity titration, and DRX). All the formulations prepared (UF: AR, UF: HAS and UF: P-HAS) were also tested for and IB, MOE, MOR, SS mechanical tests, WA and TS physical tests and formaldehyde emission (FE), and it can be seen that the best physico-chemical and mechanical results were obtained. This enabled us to choose one of the 5 samples and formulations prepared.

Topic 4. Innovative technologies for separation and fractionation processes

OR-37. The pivotal role of natural preparations in the flavor industry

Andrea ALBERTINO [1,*]

[1] Esarom Austria and Honorary President of the British Society of Flavourists

During my presentation I first introduce the British Society of Flavourist that I represent here, explaining our role in the flavour industry, our heritage and our main recent activities. Being the Honorary President of this respectable and historical Society has given me the honor of lecturing here at GENP, as well as being a speaker at Flavorcon 2024 (October, Atlantic City, NJ) and at the 2024 BSF Symposium on Chocolate (October, Brussels). Formulating successful (and stable, and cheap, and easy to produce, and...) flavours is a hard job (art? game?) and it involves a lot of experience, passion, chemical knowledge, creativity, in order to juggle with thousands of volatile and non-volatile chemicals as well as flavouring extracts, to be perfectly blended in the targeted end-use products.

The aim of my talk is to explain why flavouring preparations are essential tools for flavourists (and perfumers), going through standard and non-standard extraction methods for industrial production of natural extracts, presenting a few commonly used and well-known (at least to the flavourists) materials and eventually smelling some flavours formulated with a targeted use of naturals.

OR-38. Green extraction coupling microwaves and centrifugation: trends and insights

Pascal GINISTY [1,*], Marc VALAT [2]

[1] IFTS, 3 Rue Marcel Pagnol, 47510 Foulayronnes, France
[2] I2M (UMR 5295), Université de Bordeaux- Bat A11, 351 Cours de la Libération, 33405 Talence, France

***Corresponding author:** Pascal GINISTY, pascal.ginisty@ifts-sls.com

Keywords: Green extraction, microwaves, centrifugation, hybrid process, natural products.

Objectives

Current environmental concerns such as the depletion of fossil resources, the emission of greenhouse gases or global warming require manufacturers to reduce their impact on the environment and to take a more eco-responsible approach. This has been reflected for several years by the development of a greener chemistry and an increasingly strong orientation towards natural products. Processes are currently used for high valuable molecules extraction from natural products. Traditional methods as Soxhlet extraction, maceration, percolation and sonication have been used for many decades but are time consuming and use solvents. New methods have been developed since the last decade as super/sub critical fluid extraction, pressurized solvent extraction, pulsed electrical fields and microwave assisted extraction to increase yields, to reduce operation time and solvent consumption, energy and costs. Among them, the innovative combination of microwave extraction with high-speed centrifugation appears to be promising as it allows a high yield extraction of high valuable products (bioactive non-volatile compounds, essential oils...) at moderate temperature (surface temperature < 60°C) without using any exogenous solvent. The paper aims to review the successful applications of this new technique, its comparison with conventional techniques, its actual limitations and to present future work to better understand the extraction mechanisms and ways of optimization/scale-up.

Methods

Microwave extraction at high rotation speed (4000 rpm max) were performed in an automated prototype, of filtering centrifuge (diameter: 320 mm, height: 200 mm). The natural product (mass close to 1 kg) is disposed, as homogeneously as possible in the basket and centrifugation at high speed is first applied during 5-15 min to check if any extract volume can be recovered by mechanical pressure. Microwave power up to 1200 W is then applied without stopping basket rotation. The waveguide was specially designed in two parts, connected with a contactless wave trap. This is necessary to protect the magnetron from centrifuge vibrations. Extract mass is measured vs time and temperature of product is controlled by pyrometers. Vapors formed in the centrifuge are condensed at the outlet. The microwave application time varies from 20 to 40 min, depending on the quantity of water present in the natural product and is stopped when a significant increase of reflected power is observed, in spite of optimal impedance adjustment with tuners. Final centrifugation at maximal speed is performed at the end during 5-15 min to drain the residual extract, accumulated in the basket and in the interstitial volume of natural product. The 3 fractions are analyzed separately and the residual cake in the machine is recovered, weighed and analyzed. In some tests, exogeneous water was added to help the recovery of volatile compounds or to reslurry the cake before new extraction cycle in order to increase yields of non-volatile compounds.

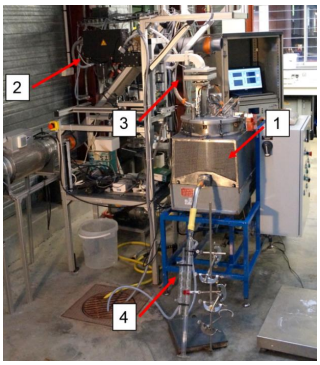


Figure 1. Pilot for extraction assisted by microwave and centrifugation (1: microwave generator, 2: microwave guide, 3: centrifuge device, 4: filtrate outlet equipped with condenser)

Results

Extraction efficiency has been evaluated for different vegetal products as a function of rotational speed and microwave incident power by experimental designs for model products for non-volatile phenolic compounds (lettuces) and volatile compounds (essentials oils from orange peels). Results are encouraging in terms of extract quality and/or yields in comparison with conventional techniques (hydrodistillation) and highlight the influence of superimposition of dielectric heating and centrifugal force on extraction. The mechanisms of vegetal cells disruption and squeezing were pointed out during Alice Angoy PhD work (2019). Research work with tobacco has compared extraction results from fresh, unfrozen, ensiled and dried leaves to conventional method (Soxhlet extraction with multiple cycle of 0.5 h with L/S=20 at 80°C) and highlight that the mechanism of nicotine recovery by solubilization/cell membrane permeabilization seem to be predominant, allowing maximal yields until 99%, with MW combined with other principles and production of nicotine extracts until 7 g/L, depending on the applied conditions. Good performances of phenolic compounds recovery from brown and green algae and sweet potato roots were also observed as well as terpenic alcohols from tea leaves. Recent tests highlight the colorings extraction from artichoke leaves and broom stems.

A new PhD work in under preparation to better understand the extraction mechanisms, depending on the natural product characteristics and molecules properties by a fine analysis of the microstructure of the vegetal cells and development of specific devices to decorrelate effect of physical, chemical, mechanical and thermal effects in the extract recovery process. This understanding will help to develop a more capacitive prototype with higher process optimization potentialities in order to fulfill the local agro-industrial sectors.

OR-39. Extractive fermentation of biobased compounds in the frame of bioeconomy

Ana-Karen SANCHEZ-CASTANEDA [1], Claire SAULOU-BERION [1], Cristian TRELEA [1], Catherine BEAL [1], Violaine ATHES [1], Marwen MOUSSA [1,*]

[1] Université Paris-Saclay, INRAE, AgroParisTech, UMR SayFood, 91123, Palaiseau, France

***Corresponding Author:** Marwen MOUSSA, marwen.moussa@agroparistech.fr

Keywords: fermentation, extraction, membrane processes, process integration, process intensification, green chemistry, biorefinery

Fermentation processes are a cornerstone of the bioeconomy. Interest in using them towards the sustainable production of bio-based volatile organic compounds (VOCs) and polymer building blocks from renewable biomass resources is growing. In order to develop robust processes for full scale industrial implementation, these must be combined with other pre/post-treatment steps and optimized within a multi-criteria approach. Indeed, favorable conditions are needed for high cellular activity, whereas some "new" complex substrates expose cells to inhibition/toxicity effects. Moreover, when metabolites accumulate in the bioreactor, similar deleterious effects can occur, which results in low productivity and dilute product streams. The integration of fermentation with extraction (extractive fermentation) aims to tackle these limitations and intensify the bioproduction. However, there are still several questions to be answered towards the mastery of extractive fermentation, such as using adapted and robust microorganisms, finding optimal bioprocess conditions that comply with the integrated recovery process and choosing biocompatible recovery operations.

Within SayFood, Paris-Saclay Food and Bioproduct Engineering Joint Research Unit, we have been conducting several interdisciplinary research projects on extractive fermentation for the production of bio-based organic acids (lactic acid, 3- hydroxypropionic acid) and VOCs (butanol, ethanol, 2-phenylethanol, ethyl acetate, sulfur compounds). Lactic acid bacteria, acetic acid bacteria and yeast are studied as cell models. Membrane-based techniques of pervaporation and membrane contactors are studied given their expected biocompatibility and compliance with green chemistry principles. Throughout this integrated strategy, we focus on understanding and modelling the mechanisms governing inhibition at the cellular level, fermentation performance and membrane mass transfer, in order to unlock technical issues at the process level.

Such an approach raises scientific questions of utmost importance that need interdisciplinary work strategy in line with the scientific challenges exposed above: bioprocess engineering, downstream processing, integrated process modelling and environmental assessment.

In this presentation, we propose to share some examples illustrating our scientific approach and a selection of results, wishing to contribute to the insightful discussion with the audience.

OR-40. Numerical simulation of membrane electroseparation of mixtures composed of particles of different sizes

Nikolai LEBOVKA [1,*], Nabil GRIMI [2], Eugene VOROBIEV [2], N. VYGORNITSKII [1]

[1] The Ovcharenko Institute of Biocolloidal Chemistry of National Academy of Sciences of Ukraine, Kyiv, Ukraine
[2] Université de Technologie de Compiègne, ESCOM, TIMR (Integrated Transformations of Renewable Matter), Centre de Recherche Royallieu-CS 60 319-60 203 Compiègne Cedex, France

***Corresponding author:** Nikolai LEBOVKA, lebovka@gmail.com

Keywords: separation, electric fields, electro-membrane filtration, cross flow filtration, electrophoretic migration, Monte Carlo model, two-component suspension

Objectives

Membrane filtration and separation techniques have many important applications related with the extraction processes and removal of various pollutants suspended in water, wastewater treatment, and treatment of bioproducts. Therefore, development of model for discription of the two-sided electroseparation of the multi-component suspension of particles placed between two membranes is the important task.

Methods

The universal Monte Carlo model was developed to describe the electroseparation. Membrane is supposed to be permeable for the small particles and impermeable for the large ones. Pure liquid (filtrate) circulates between the membrane and electrode evacuating small particles penetrated through the membrane. The electric field is directed perpendicularly to the membrane and the field direction is periodically alternated. During the simulation, the Brownian motion of particles and their electrophoretic displacement under the action of an electric field were taken into account. The large particles form barrier at the both sides of the filter chamber between two electrodes.

Results

The periodical formation of the layers of large particles on the membranes was observed. These layers act as supplementary filters retaining the small particles. The electroseparation kinetics revealed significant dependence of the total electroseparation time tf required for the elimination of all small particles versus the period of electric field alternating TE. The effects of the particles concentration, distance between the electrodes and electrophoretic velocity on the electroseparation kinetics are discussed. It was revealed that there exists some optimal values of TE to get the minimal value of t_f.

OR-41. Optimizing stilbene recovery from cell culture media: A comprehensive study of the adsorption process

Ruxandra TOMA [1], Julien LEMAIRE [2], Amandine L. FLOURAT [1], Blandine MARANT [3], Mathilde DUVAL [4], Florent ALLAIS [1], Morad CHADNI [1,*]

[1] URD Agro-Biotechnologie Industrielles, CEBB, AgroParisTech, 51110 Pomacle, France
[2] Université Paris-Saclay, CentraleSupélec, Laboratoire de Génie des Procédés et Matériaux, Centre Européen de Biotechnologie et de Bioéconomie (CEBB), 3 rue des Rouges Terres, 51110 Pomacle, France
[3] Université de Reims Champagne Ardenne, INRAE, RIBP USC 1488, 51100 Reims, France
[4] Novéal, 16 Rue Maurice Berteaux, 95500 Le Thillay, France

***Corresponding author:** Morad CHADNI, morad.chadni@agroparistech.fr

Keywords: stilbenes, resveratrol, grapevine cell cultures, resins, adsorption, purification

Objectives
The objective of this study is to develop a purification process using adsorption technology to isolate stilbenes from grapevine cell culture medium. Stilbenes, a class of phenolic compounds, are renowned for their bioactive properties and hold substantial promise for health benefits and applications in pharmaceuticals and nutraceuticals. Traditional purification methods typically involve the use of organic solvents, which raise environmental and safety concerns.

Methods
Firstly, five microporous resins (XAD-7, XAD-16, XAD-4, XAD-1180, and FPX-66) were tested for a preliminary screening step to determine the optimum polymeric adsorbent. The second step consisted of studying the kinetics of adsorption by following the evolution of the adsorbed and desorbed quantity of stilbenes. Two kinetics-based models (pseudo-first and pseudo-second order) and one diffusion-based model (intraparticle diffusion) were used to interpret the process. Thirdly, adsorption isotherms were studied using a multicomponent Langmuir model taking into account the competition between the stilbenes. Finally, experiments were conducted to optimize various process parameters, such as the ratio and ethanol content of the desorption solution, but also influence of the water quantity, pH, and washing time on the final purity of stilbenes.

Results
XAD-7 was chosen as the optimum adsorbent, displaying the highest adsorption quantity (86.94 ± 4.90 mg stilbenes/g dry resin) and desorption yield (74.28 ± 0.38 mg stilbenes/g dry resin) capacities. Adsorption kinetics using XAD-7 followed a pseudo-second-order model, with intraparticle diffusion limiting approximately 10% of total adsorption. Isotherm curves fitted well to a multicomponent Langmuir model, indicating a maximum adsorption capacity of 0.280 to 0.360 mmol stilbenes/g dry resin.

Stilbene affinity for XAD-7 decreased in the order ε-viniferin > (labruscol, E-resveratrol, leachianol) > δ-viniferin.

The optimal desorption yield of 59.74 ± 0.14 mg stilbenes/g dry resin was achieved with a 70% ethanol solution and a 160:1 desorption solution-to-adsorbent ratio. Using XAD-7 resin, coupled with an optimized washing step, increased stilbene purity by 4.6 times (from 5 ± 0.05% to 23.19 ± 0.31% w/w).

OR-42. Chestnut wood waste valorization: innovative green methods to enhance bioactive fractionations

Clelia AIMONE [1,*], Giorgio GRILLO [1], Giorgio CAPALDI [1], Salah CHAJI [1], Giancarlo CRAVOTTO [1]

[1] University of Turin, Department of Drug Science and Technology, Via P. Giuria 9, Turin, Italy.

***Corresponding author:** Clelia AIMONE, clelia.aimone@unito.it

Keywords: Green Extraction, Chestnut Wood, Polyphenols, Tannins, Metabolites, Downstream, Membrane Filtration, Resins

Objectives

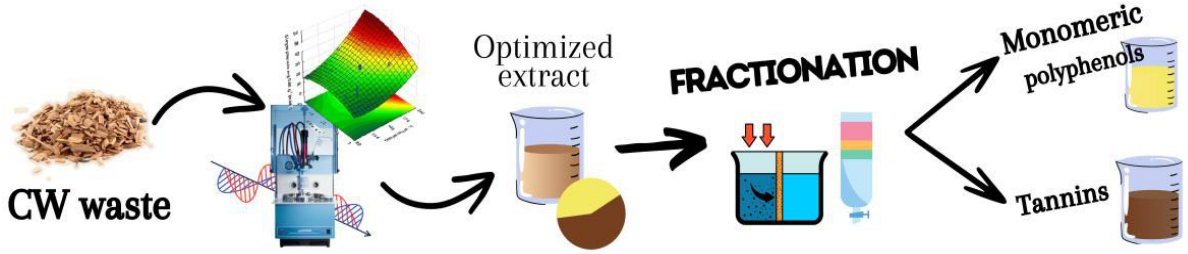
Within the framework of Circular Economy and Green Extraction, this study focuses on the extraction of chestnut wood (CW) waste (Castanea sativa, Mill.), due to its high concentration on active metabolites with antimicrobial, antioxidant, and antiproliferative properties. This study explores the potential of sustainable and environmentally conscious extraction and fractionation protocols, aiming to recover products enriched in specific metabolite classes.

Methods

Microwave-assisted Subcritical Water Extraction (MASWE) and green, scalable downstream techniques have been employed to isolate several enriched extracts from chestnut wood residues. Central Composite Design (CCD) was employed to identify the optimized extraction conditions, to maximise the recovery of overall metabolite content, followed by a crucial downstream process, comprising membrane filtration and resins fractionation. Comprehensive analyses were conducted to establish the composition of the products, both in terms of total polyphenol content (TPC) and tannin characterization, complemented by HPLC investigation and biological activity screening.

Results

After conducting a series of experiments provided by the CCD, the optimal condition to balance monomeric polyphenols and tannic fraction has been identified (174 °C, 14 min, S/L ratio 1:20). The extracted material was further fractionated using membrane filtration (Ultrafiltration and Nanofiltration with different cut-offs) and adsorption on resins (e.g. Sephadex). The resulting fractions, characterized by different population of polyphenols each, have been characterized with colorimetric methods (TPC, tannins content, flavonoids, sugars, antioxidant capacity) and tested as potential enzymes inhibitors.



OR-43. Overcoming the shortfall in green metrics for extraction processes: the SIX score approach

Giorgio GRILLO [1,*], Giorgio CAPALDI [1], Clelia AIMONE [1], Emanuela CALCIO GAUDINO [1], Silvia TABASSO [1], Giancarlo CRAVOTTO [1]

[1] Department of Drug Science and Technology, University of Turin, Turin, Italy

***Corresponding author:** Giorgio GRILLO, giorgio.grillo@unito.it

Keywords: Extraction, green metrics, green extraction principles, software interface.

Objectives
While green metrics have been well-established for synthesis and analytical procedures, there is a notable gap in the development and application of these metrics specifically for extraction protocols: current methodologies do not properly address the unique challenges and parameters associated within the framework. This research seeks to provide a comprehensive framework for quantifying the "greenness" of extraction processes, addressing key parameters such as energy efficiency, solvent use, and waste generation.

Methods
The methodology of this study is structured around several key aspects, essential for evaluating the procedures' greenness. The Six Green Extraction Principles serve as the primary guidelines for defining crucial aspects of sustainability in the filed. A comprehensive review of literature was conducted, utilizing intelligent algorithms (IA) to cluster and analyse references according to their relevance. This study not only considers environmental hazards but also evaluates human health risks, energy consumption, and equipment intakes to provide a holistic assessment of the extraction processes.

Results
By taking in consideration existing methodologies and introducing new metrics, this study adopted the commonly accepted "Penalty Points" (PP) approach, to quantify the impact of various factors. PP are assigned according to the degree of deviation from the ideal green principles. Protocols are assessed defining six distinctive factors: i) Raw Material; ii) Solvents & Additives; iii) Extraction; iv) Process & Equipments; v) Waste; vi) Product. To each of these factors is assigned a score, resulting from the combination of several sub-items. To merge the various aspects into a single comprehensive metric, a general value was calculated that integrates all considered factors. The ***S**ustainability **I**mpact for E**x**tractions* (*SIX*) Score offers a clear and concise evaluation, combining the environmental and health impacts, energy requirements, and operational efficiency, facilitating the identification of the most sustainable methods. The refined procedure has been embedded into an open-source software with an integrated database, enabling a constant system evolution. The ambitious goal of *SIX Score* is to delineate a standardized boundary, in which the multifaceted extractions field could find a dedicated and shared metric. The backbone of the proposed approach relies on readily comprehensible quantitative estimations. This initiative is crucial for developing more sustainable and less impactful extraction processes.

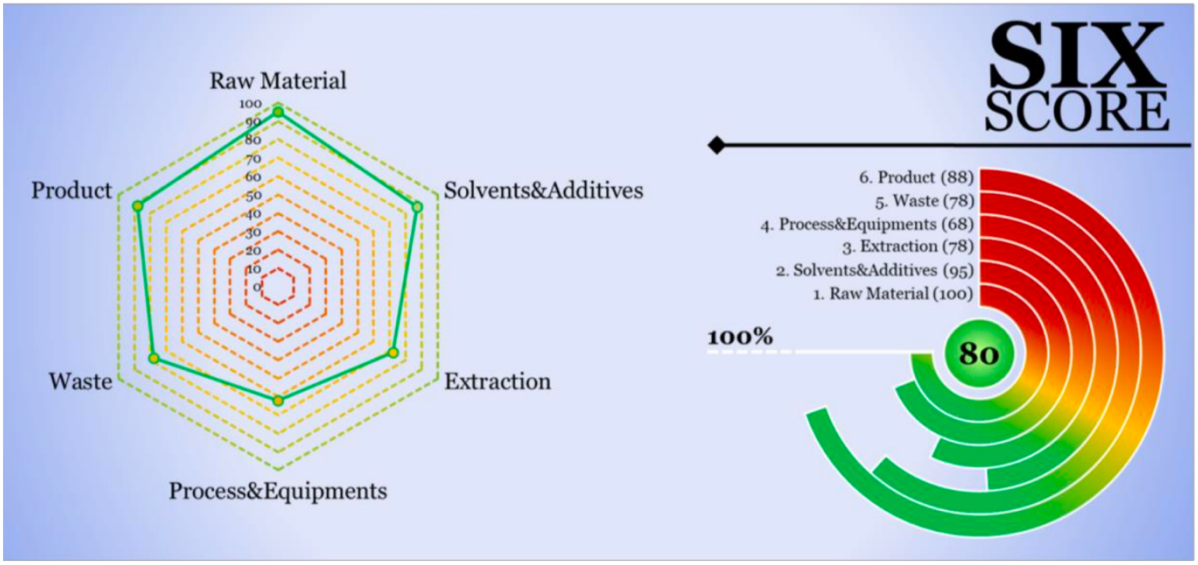


Figure. Example of SIX Score Evaluation.

OR-44. Assessment of the impact of treated wastewater on essential oil yield and agrophysiological responses of *Rosmarinus officinalis*

Aya KAMMOUN [1,2], Naaila OUAZZANI [1,2], Abderrahmane ROMANE [3], Anne-sylvie FABIANO-TIXIER [4], Laila MANDI [1,2]

[1] National Center for Studies and Research on Water and Energy (CNEREE), Cadi Ayyad, University, Marrakech, Morocco.
[2] Laboratory of Water, Biodiversity and Climate change (EauBiodiCc), Faculty of Sciences Semlalia, University Cadi Ayyad, Marrakech, Morocco.
[3] Laboratory of Applied Chemistry and Biomass, Faculty of Sciences Semlalia, University Cadi Ayyad, Marrakech, Morocco.
[4] UMR 408 INRA-UAPV, Sécurité et Qualité des Produits d'Origine Végétale, Université d'Avignon, France.

***Corresponding author:** Aya KAMMOUN, kamouneaya2@gmail.com

Keywords: Treated wastewater; multi-soil-layering system; irrigation; *Rosmarinus officinalis*; essential oil yield; agro-physiology

Objectives

The aims of this study were to investigate the effects of irrigation with treated wastewater, using a hybrid multi-soil-layering (MSL) technology, on the agro-physiological characteristics, essential oil composition and yields of Rosmarinus officinalis (rosemary).

Methods

In this experiment, nine plots were planted with rosemary and irrigated with different water qualities: raw wastewater (RWW), treated wastewater (TWW), and well water (WW) for four months. At maturity, plants were selected from each field on the basis of treatment (RWW, TWW and WW). Parameters studied on each selected plant are: stem height, dry weight, fresh weight, total chlorophyll, sugar, Malondialdehyde (MDA), superoxide dismutase activity (SOD), and proline. Microwave extraction of essential oil from 15 g of Rosmarinus officinalis in 150 ml of solvent (distilled water) (plant/water ratio 10 g/100 ml) was performed with 60 min extraction time at 500 W microwave power. For each sample, the average yield of essential oil was taken as the mean of 3 distillations. The essential oils obtained were stored at -4°C, protected from light, until further used.

Results

The study revealed notable differences in the plants' responses to the three types of irrigation water. Rosemary irrigated with RWW showed higher levels of sugar (55.46 mg/L), MDA (0.096 ng/g plant), SOD (10.30 U/mg proteins), and proline (8.57 µM/g FW) compared to those irrigated with TWW (54.98 mg/L, 0.062 ng/g plant, 6.40 U/mg proteins, and 6.68 µM/g FW) and WW (54.3 mg/L, 0.028 ng/g plant, 5.93 U/mg proteins, and 1.32 µM/g FW), respectively. Additionally, biomass parameters such as stem height and dry and fresh weight were positively impacted by RWW and TWW irrigation compared to WW. The highest chlorophyll content was observed in plants irrigated with RWW (12.7 mg/g), followed by TWW (10.09 mg/g). For the extraction of essential oil, the results indicated a beneficial increase in essential oil yield for RWW (2.53%), followed by MSL TWW (2.42%) and WW (0.89%).

OR-45. Eradication of the invasive species *Fallopia japonica* and valorization of its rhizomes by extraction as a part of a complete value chain

Florentin COUPÉ [1], Grégory CHATEL [2,*], Luc JAGER [1]

[1] Rhizomex SAS, 199 Rue Pré Gaut, 73420 Voglans, France.
[2] Univ. de Savoie Mont Blanc, CNRS, Laboratoire Environnements, Dynamiques et Territoires de Montagne (EDYTEM), 73000, Chambéry, France.

***Corresponding author:** Grégory CHATEL, gregory.chatel@univ-smb.fr

Keywords: circular economy; decarbonation; green extraction; ultrasound-assisted extraction; response surface methodology; selective extraction; integrated value chain

Objectives

Japanese knotweed (Fallopia japonica) is a well-known invasive species especially in western Europe, causing various disturbances against biodiversity and human infrastructures. However, its rhizomes, main responsible for its invasive character thus often considered as a biomass to be eliminated, are a valuable and prized source of antioxidants, notably resveratrol, for several uses such as in cosmetics, nutraceuticals, or crop protection. Yet, while important amounts of resveratrol are otherwise produced in a carbon-intensive way, tonnes of locally-available Japanese knotweed rhizomes as a precious resveratrol source are destroyed. The proposed approach aims at creating a complete and low carbon-intensive value chain from these aspects, first through an efficient on-site eradication protocol serving as the rhizome collection step, then with the development of an eco-designed and scalable extraction step for the valorization of the excavated rhizomes. This communication will have a particular focus on the extraction step, with emphasis on the scalability of the process in an environmentally friendly way.

Methods

Japanese knotweed rhizome extracts are a complex mixture of polyphenols, categorized in three main families: stilbenes, flavonoids, and anthraquinones, plus their derivatized forms mainly as glycosides. Although there are many described methods for their extraction, there is little comprehensive study on the extraction conditions influence on the extract composition. Moreover, extraction protocols rarely consider green chemistry aspects. Depending on the desired final application, certain polyphenols are preferred while others are not, and a purification step may be required. Thus, using a water-ethanol mixture as the green extraction solvent, this work intended to demonstrate how the extraction conditions could tailor and simplify the extract composition, over a range of parameter values. Apart from the ethanol-water ratio, other parameters were tested: extraction temperature and liquid-to-solid ratio. Furthermore, the use of ultrasound to assist the extraction as a non-conventional technique was also considered. To better cover and evaluate the effect of these parameters, a response surface methodology (RSM) was applied, as a way to draw a mapping of the extraction space, to evaluate both the extraction selectivity and sustainability.

Results

The performed RSM demonstrated that the critical factor influencing the extract composition was the ethanol-water ratio. Based on this parameter, a comprehensive description of its influence was established, allowing for a selective extraction and a simpler extract composition at certain values, even if the extraction yield was not maximal for these values. Thus, choosing proper extraction parameter values appears as a way to obtain a customized extract for specific applications, for example if one

particular molecule has to be isolated and purified. The other tested parameters were less influent on the extract composition, but their evaluation provided useful information for the perspective of a process intensification at a larger scale and its sustainability, especially regarding energy and solvent consumption. In definitive, lab-scale experiments proved the ability of the extraction step to be integrated in a complete value chain for the upgrading of Japanese knotweed rhizomes initially stated as a detrimental biomass.

OR-46. Computational solvent screening using COSMO-RS approach for extracting bioactive compounds from cocoa bean shells

Felipe S. BRAGAGNOLO [1,2,*], Andrea SANCHEZ-MONEDERO [1], Ismael DIAZ [2], Mauricio A. ROSTAGNO [1], Maria GONZALEZ-MIQUEL [2]

[1] Multidisciplinary Laboratory of Food and Health (LabMAS), School of Applied Sciences (FCA), Universidade Estadual de Campinas, São Paulo, Brazil
[2] Department of Chemical and Environmental Engineering, Higher Technical School of Industrial Engineering, Universidad Politécnica de Madrid, Spain

***Corresponding author:** Felipe S. BRAGAGNOLO, felipesb@unicamp.br

Keywords: Biorefenery, by-products, bioactive compounds, in-silico tools, green chemistry

Objectives

The primary objective of this research is to enhance the recovery of methylxanthines and phenolic compounds from cocoa bean shells using an integrated approach. By employing the Conductor-like Screening Model for Real Solvents (COSMO-RS) model for green solvent screening, this study aims to identify solvents that optimize extraction efficiency while minimizing environmental impact. Advanced extraction techniques, such as Microwave-Assisted Extraction (MAE) and Ultrasonic-Assisted Extraction (UAE), will be utilized to intensify the extraction process. Additionally, the research will focus on the recovery and reuse of solvents to further promote sustainability. The overall environmental impact and sustainability of the process will be assessed using Life Cycle Assessment (LCA) and other relevant sustainability metrics.

Methods

In silico solvent screening was conducted based on the activity coefficients at infinite dilution for theobromine and epicatechin, which are representative methylxanthine and phenolic compounds, respectively, using solvents listed in CHEM21. Subsequently, experimental screening will be performed with the optimal solvents identified by the COSMO-RS approach, leading to more accurate results in recovering metabolites from cocoa bean shells. Considering the yields from both in silico and experimental screenings, the solvent that provided the best results will be combined with Microwave-Assisted Extraction (MAE) and Ultrasonic-Assisted Extraction (UAE). Extraction conditions will be optimized based on the quantification of theobromine, caffeine, catechin, epicatechin, and procyanidin B2 via HPLC-DAD. Solvent recycling will be conducted using a rotary evaporator, and its reuse will be evaluated. Life Cycle Assessment (LCA) and green sample preparation metrics will be employed to assess the sustainability of our proposed methodology.

Results

Considering the solvents listed in CHEM21 as recommended and problematic, 61 possibilities were entered into the computational solvent screening. From these, 12 solvents were selected based on the activity coefficient at infinite dilution for theobromine and epicatechin, namely: lactic acid, methanol, benzyl alcohol, ethyl lactate, valerolactone, acetone, 1,3-propanediol, ethanol, methyl ethyl ketone, water, isopropyl alcohol, and 1,2-propanediol. As anticipated for the next steps, the experimental screening will identify the most suitable solvent for extracting both theobromine and epicatechin. Subsequently, intensifying the recovery process through MAE and UAE, and comparing both performances, we aim to achieve efficient and optimal recovery of bioactive compounds. This process is expected to generate an extract with potential applications in the food and cosmetics sectors, added with the benefits of using green solvents and sustainable processes.

OR-47. From black and green tea (*Camellia sinensis*) by-products to potential natural health boosters: optimization of polyphenol extraction and assessment of antioxidant and antibacterial potentials

Ahlam HARFOUSH [1], Aseel SWAIDAN [1], Salma KHAZAAL [2], Bilal AZAKIR [3], Elie Salem SOKHN [4], Nicolas LOUKA [5], Esperance DEBS [6], Nada EL DARRA [1,*]

[1] Department of Nutrition and Dietetics, Faculty of Health Sciences, Beirut Arab University, Tarik El Jedidah, Riad El Solh, P.O. Box 115020, Beirut 1107 2809, Lebanon.
[2] Department of Biological Sciences, Faculty of Science, Beirut Arab University, Beirut P.O. Box 11-5020, Lebanon.
[3] Molecular and Translational Medicine Laboratory, Faculty of Medicine, Beirut Arab University, Beirut 1107 2809, Lebanon
[4] Department of Medical Laboratory Technology, Faculty of Health Sciences, Beirut Arab University, Beirut 1107 2809, Lebanon.
[5] Centre d'Analyses et de Recherche, Unité de Recherche Technologies et Valorisation Agro-alimentaire, Faculté des Sciences, Saint -Joseph Université, P.O. Box 17-5208, Riad El Solh, Beirut 1104 2020, Lebanon.
[6] Department of Biology, Faculty of Arts and Sciences, University of Balamand , P.O. Box 100, Tripoli 1300, Lebanon.

*** Corresponding author:** Nada EL DARRA, n.aldarra@bau.edu.lb

Keywords: Tea, extraction, antioxidant, antibacterial.

Abstract

Bioactive compounds, such as polyphenols, have gained significant attention due to their exceptional health benefits and biological functions. Food by-products are known to contain a substantial amount of polyphenols. Thus, valorizing these by- products is a promising approach to increase the nutritional value of food products while reducing pollution levels. The current study aimed to optimize the extraction of polyphenols from spent black tea (SBT) and spent green tea (SGT) leaves, as well as to investigate their antioxidant and antibacterial properties. Response surface methodology (RSM) was utilized to study the effect of varying ethanol concentration, extraction time, and extraction temperature on the total phenolic content (TPC) and antioxidant activity. In addition, high-performance liquid chromatography (HPLC) was performed to identify and quantify the phenolic compounds present in tea extracts. Antibacterial effect was evaluated using disc diffusion method against two Gram-positive: *Staphylococcus aureus* and *Bacillus subtilis*, and two Gram-negative: *Salmonella* Typhimurium and *Escherichia coli* bacterial strains. Results showed that the highest TPC (410.4 mg GAE/g dry matter) was obtained at a solid-to-solvent ratio of 1:25 for SBT, whereas the highest TPC value (251.4 mg GAE/g dry matter) for SGT was yielded at a ratio of 1:20. Based on these ratios, RSM identified the optimum extraction conditions for SBT as follows: temperature 93.6 °C, extraction time 79.93 min, and ethanol concentration 59.3%, yielding 404.13 mg GAE/g dry matter and 51.45 %DPPH inhibition; whereas those for SGT were identified as follows: 93.63 °C, 81.65 min, and 53.21%, yielding a TPC value of 451.4 mg GAE/g dry matter and 78.2 %DPPH inhibition. HPLC analysis revealed that the predominating phenolic compound was hydroxybenzoic acid (360.73 ppm) in SBT, and rutin (42.73 ppm) in SGT. Both SBT and SGT exhibited antibacterial activities against gram-positive bacterial strains; however, no effect was observed against gram-negative bacteria. In conclusion, after optimizing the extraction method, the phenolic-rich extracts of both SBT and SGT showed promising antioxidant and antibacterial potentials, representing strong candidates for future incorporation in food products as natural health boosters.

Posters

PO-01. A Tuneable process for the extraction and purification of chitosan from mealworm larvae

Amal DALBOUHA [1,*], Cherif JABLAOUI [1], Nadia BOUSSETTA [1] and Houcine MHEMDI [1]

[1] Laboratoire Transformations Intégrées de la Matière Renouvelable (UTC/ESCOM, EA 4297 TIMR), Université de technologie de Compiègne UTC, France.

***Corresponding author:** Amal DALBOUHA, amal.dalbouha@etu.utc.fr

Keywords: Chitosan, Mealworm larvae, Extraction, Optimization, Characterization.

Objectives

Chitosan, a naturally derived biopolymer, exhibits a wide range of applications in fields such as medicine, agriculture, and the food industry. This study aims to set up and optimize a simplified process for the extraction and the purification of chitosan from mealworm larvae by tuning experimental operating conditions. The process involves four key steps: demineralization, deproteinization, decolorization and deacetylation.

Methods

Process optimization was achieved by investigating the effect of various parameters on the quality of the extracted chitosan. A kinetic study was conducted to determine the optimal H_2 O_2 concentration for decolorization, as well as the optimal decolorization time. A design of experiments was implemented to optimize the deacetylation parameters (temperature and NaOH concentration). The obtained chitosan was characterized using FTIR spectroscopy, pH-metric titration, scanning electron microscopy (SEM), X-ray diffraction (XRD), and UV spectroscopy.

Results

The results confirmed that we have achieved the alpha-chitin structure. During the demineralization stage, based on ash content analysis, we successfully removed 66.20% of the minerals using 1M HCl for 36 hours. After demineralization, the chitin was succefully extracted. For the decolorization step, it was shown that the best result was obtained by using 25% H_2O_2 solution during 60 min. Finally, chitin was converted to chistosan by chemical deacetylation. According to the design of experiments and statistical analysis, we confirmed that temperature and NaOH concentration affect the degree of deacetylation (DDA) of chitosan. The highest DDA (90%) was onbtaind at 100°C and 5M NaOH confirming the effectiveness of the devleopped process. The purified chitosan will be used by our project partner to produce a food packaging biobased material.

PO-02. Antioxidant activity, antimicrobial activity, and chemical profile of caraway (*Carum carvi*) essential oil obtained under different conditions

Sanja MILOŠEVIĆ [1], Danica BOŽOVIĆ [1], Bojana IKONIĆ [1], Nevena GLADIKOSTIĆ [1], Nemanja TESLIĆ [2], Zoran ZEKOVIĆ [1], Branimir PAVLIĆ [1,*]

[1] Faculty of Technology, University of Novi Sad, Blvd. cara Lazara 1, 21000 Novi Sad, Serbia
[2] Institute of Food Technology, University of Novi Sad, Blvd. cara Lazara 1, 21000 Novi Sad, Serbia

***Corresponding author**: Branimir PAVLIĆ, bpavlic@uns.ac.rs

Keywords: Caraway, essential oils, antioxidant activity, antimicrobial activity, gas chromatography

Objectives

The main goal of this research was to characterize two types of caraway essential oil (CEO) obtained under different conditions in terms of antioxidant, antimicrobial activity, and chemical profile.

Methods

CEOs were obtained by hydrodistillation (HD) at 205 and 410 W and microwave- assisted hydrodistillation (MWHD) at 180, 360, 600 and 800 W. For all CEOs antimicrobial activity and in vitro antioxidative activity against DPPH and ABTS radicals was measured. The chemical profile of CEO was determined by GC-MS.

Results

MWHD at 800 W enable obtaining the highest CEO yield (3.81%). CEO obtained by HD at 410 W expressed the highest activity to neutralize DPPH radicals (0.99±0.58 μM TE/g), and CEO from MWHD at 800 W possessed the highest activity against ABTS radicals (2.30±1.14 μM TE/g). CEO obtained by HD at 410 W showed the highest antimicrobial activity against Proteus hauseri ATCC13315. Among 12 identified compounds in CEOs, carvone was the most dominant compound with 75.84% in MWHD at 180 W. Additionally, MWHD at 800 W enable obtaining highly valuable CEO which may be incorporated in various forms to increase the bacteriological safety of food products.

This research was supported by the Science Fund of the Republic of Serbia, 7750168, Novel extracts and bioactive compounds from under-utilized resources for high-value applications–BioUtilize.

PO-03. Application of subcritical water extraction for the production of high value components from agricultural byproducts: Comparison with conventional hydroalcoholic extraction

Kevin LÉONARD [1,*], Camille MALTERRE [1], Job TCHOUMTCHOUA [1]

[1] Celabor, Belgium.

***Corresponding author:** Kevin LÉONARD, kevin.leanaord@celabor.be

Keywords: Subcritical water extraction, agricultural byproducts, polyphenols, circular economy, bioactive compounds

Introduction

Agricultural industry produces the largest amount of residual biomass and by know, almost half of it is wasted. Most of this residual biomass is only used for low to mid-value applications such as biofuels, biomethane, soil enrichment, animal feed. Certain agricultural side streams like onion peels, chicory leaves, vine and olive pruning are known to be rich in phenolic bioactive substances. Those compounds posses a wide range of activities: antioxidant, anti-inflammatory, anti-viral and even anti-cancer.

Objectives

The objective of this study is to explore an eco-friendly and sustainable method for extracting phenolic bioactives from various sources through Subcritical Water Extraction (SWE). The results of SWE, including yield, phenolic content, and antioxidant capacity, have been compared to those obtained from a standardized hydroalcoholic maceration to evaluate the efficiency of SWE. Additionally, the extracts were analyzed using UPLC DAD/MS equipment to identify the primary phenolic compounds present.

Results

The comparison between SWE and hydroalcoholic maceration provides engaging and promising results concerning phenolic compounds extraction. While leaving room for further use of the residual extracted biomass in more conventional channels. This study paves the way for new opportunities in the agricultural sector, highlighting significant potential for future nutraceutical and or pharmaceutical applications.

PO-04. Aqueous extraction of clean label oleosomes from plant seeds

Marwen MOUSSA [1,*], Pablo GRANDA [1], Delphine HUC-MATHİS [1], Paola SOTO [1], Marie-Noëlle MAİLLARD [1], Sabine D'ANDRÉA [2], Murielle HAYERT [1]

[1] Université Paris-Saclay, INRAE, AgroParisTech, UMR SayFood, 91123, Palaiseau, France.
[2] Université Paris-Saclay, INRAE, AgroParisTech, Institut Jean-Pierre Bourgin(IJPB), 78000, Versailles, France.

***Corresponding Author:** Marwen MOUSSA, marwen.moussa@agroparistech.fr

Keywords: plant oleosomes, Camelina sativa, aqueous extraction, minimal processing, process intensification

Food and bioproducts emulsion-based products are generally produced from refined oils that are emulsified while adding emulsifiers and stabilizers. An alternative processing approach is to extract the natively stable oil-storing structures, oleosomes, and to use them as a natural ingredient for further applications. This study aimed to develop a scalable minimal process for the recovery of oleosomes at high yield from Camelina sativa seeds and the preservation of their native structural and functional properties.

Camelina sativa presents not only advantageous agronomical behavior but also seeds rich in W-3 fatty acids compartmentally located in oleosomes (30–38% of the seed mass) and in soluble fibers (7–10% of the seed mass). Like for many oleaginous seeds, the outermost layer of Camelina seeds are surrounded by a mucilage that expands under hydration. This results in a thickening effect that hinders shear forces during mechanical treatments and interferes with oleosomes recovery. The process developed in this study includes several cascade steps that were designed to cope with this behavior, while seeking for high oleosome recovery yield and optimized energy and water consumptions. It includes: i) Soaking (5% seeds w/w) in order to hydrate and break the mucilaginous network. ii) Sonication using a continuous system equipped with a cascatrode and operating at 670 W for 30 s, completed by a fluidic recirculation (350 L/h) as an efficient pre-treatment for mucilage removal by iii) washing in a drum separator (0.5 mm sieve mesh). iv) Demucilated seeds are then suspended in water (5 to 25% w/w) and grinded using a colloid mill set with a variable gap between rotor and stator devices and operated at 7990 rpm for 5 to 15 min. The final separation step is made by centrifugation. The operating parameters were combined in order to identify the best performing conditions in terms of oleosome recovery yield and quality. Samples were characterized by laser granulometry and confocal microscopy, proving the integrity of extracted structures. Energy consumption was evaluated throughout the processing steps. Water footprint minimization would be an interesting way to improve the environmental friendliness. The composition of mucilage suspension would be analyzed for further applications.

PO-05. Centrifugal partition chromatography fractionation of stilbenes and anthraquinones from japanese knotweed (*Reynoutria japonica*) toward nutraceuticals and pharmaceutical applications

Soukaina HILALI [1,*], Kevin LÉONARD [1], Tania DURRE [2], Mathilde WIEME [2], David TORRES [2], Mahmoud HAMZAOUI [1]

[1] Celabor, Belgium.
[2] CER Groupe - Immunobiology Laboratory, Belgium.

***Corresponding author:** Soukaina HILALI, soukaina.hilali@celabor.be

Keywords: Centrifugal Partition Chromatography fractionation, stilbens, anthraquinones

Objectives

Japanese Knotweed (*Reynoutria japonica*) is a highly invasive plant species recognized for its abundant reservoir of bioactive compounds, particularly stilbenes such as resveratrol and its glycoside derivatives, including polydatin and resveratrol 4- glucoside. Notably, it stands as one of the richest natural sources of resveratrol, primarily concentrated in the roots, though it's also present in other parts of the plant to a lesser extent. Historically, Japanese Knotweed has been used in traditional Asian medicine for its therapeutic properties, particularly in treating cardiovascular conditions, inflammation, and infections, leading to its rising popularity as a dietary supplement. Despite these benefits, the presence of anthraquinones, notably emodin, presents challenges for its application in the nutraceutical industry due to potential gastrointestinal irritation concerns. Nevertheless, these anthraquinones exhibit significant pharmaceutical potential, with studies indicating their anti-inflammatory and anticancer properties. The objective of this study is to optimize the extraction process for these bioactive molecules and subsequently fractionate them to maximize their therapeutic potential in different applications.

Methods

This study explores the use of Centrifugal Partition Chromatography (CPC) to precisely fractionate stilbenes and anthraquinones from Japanese Knotweed extract. The process involves a systematic optimization of CPC parameters and solvent systems to achieve distinct fractions tailored for specific applications. These fractions have been thoroughly characterized using HPTLC and UPLC DAD/MS.

Results

Characterization of the obtained fractions using analytical techniques confirmed the efficient separation in the obtained fractions. This work was also coupled with a biological study to test the cytotoxicity, antiviral and immunomodulation activity of the different fractions. Overall, this study underscores the importance of efficient separation processes method for harnessing the bioactive potential of Japanese Knotweed while addressing regulatory constraints in food supplements and exploring novel avenues in pharmaceutical research.

PO-06. Comparison of chemical profile and bioactivity of dill essential oil obtained by conventional and innovative distillation techniques

Danica BOŽOVIĆ [1], Sanja MILOŠEVIĆ [1], Bojana IKONIĆ [1], Nevena GLADIKOSTIĆ [1], Nemanja TESLIĆ [2], Zoran ZEKOVIĆ [1], Branimir PAVLIĆ [1,*]

[1] Faculty of Technology, University of Novi Sad, Blvd. cara Lazara 1, 21000 Novi Sad, Serbia.
[2] Institute of Food Technology, University of Novi Sad, Blvd. cara Lazara 1, 21000 Novi Sad, Serbia

***Corresponding author:** Branimir PAVLIĆ, bpavlic@uns.ac.rs

Keywords: HD, MWHD, antioxidant activity, antimicrobial activity, chemical profile

Objectives
The main goal of this research was to determine the yield, chemical composition and bioactivity of dill essential oil (EO), obtained by traditional hydrodistillation (HD) and microwave hydrodistillation (MWHD) from dill seed.

Methods
For the isolation of EO, HD was performed at powers of 205 and 410 W, while MWHD was performed at powers of 180, 360, 600 and 800 W. GC-MS was used for identification and quantification obtained EOs. For antioxidant potential of EOs was used in vitro DPPH and ABTS assay. Antimicrobial activity was determined against 14 microorganisms by disc diffusion method.

Results
The highest yield was obtained using MWHD at 600W (3.23%). The highest potential for $DPPH^{\bullet}$ and $ABTS^{+}$ radical scavenging was exhibited by the EO obtained using MWHD at 800W (0.74 and 23.678 μM TE/g, respectively). GC-MS analysis revealed that carvone and limonene dominate in all oils within different relative area. *Yersinia enterocolitica* ATCC 27729 was the most sensitive to both samples (HD 410 W and MWHD 600 W), with MIC values was 1.25 mg/mL. According to results, the applied techniques provide to be effective for obtaining EO, with its potential for future aplication in different branches of industries.

This research was funded by the Science Fund of the Republic of Serbia for the funding of the IDEAS project 'Novel extracts and bioactive compounds from under-utilized resources for high-value applications-BioUtilize', number '7750168'.

PO-07. Eco-friendly synthesis of lignin beads for improved adsorption of methylene blue and methyl orange

Mohamed Amine KASBAJI [1,2,*], Meriem KASBAJI [3], Mounir EL ACHABY [3], Amine MOUBARIK [2], Mustapha OUBENALI [2]

[1] Laboratory of Engineering in Chemistry and Physics of Matter, Faculty of Science and Technologies, Sultan Moulay Slimane University, BP 523, Beni-Mellal, Morocco.
[2] Laboratory of Chemical Processes and Applied Materials, Polydesciplinary Faculty, Sultan Moulay Slimane University, BP 592, Beni-Mellal, Morocco.
[3] Materials Science, Energy and Nanoengineering (MSN) Department, Mohammed VI Polytechnic University, Lot 660 – Hay Moulay Rachid, 43150, Ben Guerir, Morocco.

***Corresponding author:** Mohamed Amine KASBAJI, mohamedamine.kasbaji@usms.ma

Keywords: Lignin beads; wastewater treatment; Langmuir isotherm; eco-friendly materials; renewable resources.

Objectives

This study investigates the potential of lignin beads for the adsorption of Methylene Blue (MB) and Methyl Orange (MO) from aqueous solutions. Lignin, an abundant and renewable byproduct of the paper and pulp industry, presents a viable and cost-effective option for wastewater treatment. The synthesis and characterization of lignin beads were performed using scanning electron microscopy (SEM) and Fourier-transform infrared spectroscopy (FTIR). Adsorption experiments were conducted to evaluate the performance of the beads under various conditions, including initial dye concentrations, pH levels, and contact times. The results indicated that lignin beads exhibited significant adsorption capacities for both dyes, with optimal adsorption observed at pH 7 for MB and pH 3 for MO. Adsorption kinetics followed a pseudo-second-order model, suggesting a chemisorption mechanism. Isotherm analysis showed a good fit with the Langmuir model, indicating monolayer adsorption. The maximum adsorption capacities were determined to be 250 mg/g for MB and 220 mg/g for MO. Thermodynamic analysis revealed that the adsorption process was spontaneous and exothermic. These findings underscore the promising potential of lignin beads as an efficient and eco-friendly adsorbent for the removal of MB and MO from wastewater, providing a viable solution for environmental remediation.

Methods

Lignin beads were synthesized through the cross-linking of lignin with an appropriate reagent, followed by purification, dehydration, and preservation steps. The beads were characterized using scanning electron microscopy (SEM) to investigate their surface morphology, and Fourier-transform infrared spectroscopy (FTIR) to analyze their functional groups. Batch adsorption experiments were conducted to evaluate the efficiency of lignin beads in removing Methylene Blue (MB) and Methyl Orange (MO) from aqueous solutions. Various parameters were systematically varied, including initial dye concentrations (10 to 100 mg/L), pH levels (3 to 7), and contact times (30 to 180 minutes). Adsorption kinetics were examined using pseudo-first-order and pseudo- second-order models, while adsorption capacity and mechanisms were determined using Langmuir and Freundlich isotherm models. Additionally, thermodynamic properties, including changes in Gibbs free energy ($\Delta G°$), enthalpy ($\Delta H°$), and entropy ($\Delta S°$), were calculated to understand the spontaneity and nature of the adsorption process.

Results

Lignin beads demonstrated significant adsorption capacities for both Methylene Blue (MB) and Methyl Orange (MO). The maximum adsorption capacities were determined to be 250 mg/g for MB and 220 mg/g for MO, achieved under optimal conditions (pH 7 for MB and pH 3 for MO). The adsorption kinetics conformed to a pseudo-second-order model, indicating that chemisorption was the dominant mechanism. Isotherm analysis showed a good fit with the Langmuir model, indicating monolayer adsorption on a homogeneous surface. Thermodynamic parameters indicated that the adsorption processes were spontaneous and exothermic. In conclusion, lignin beads were found to be efficient and promising adsorbents for the removal of MB and MO from aqueous solutions, highlighting their potential for environmental remediation applications.

PO-08. Eco extraction of valuable compounds from wood by supercritical CO_2 process: influence of raw material and operating conditions

Marion SKOCZEK [1,2], Christelle HARSCOAT-SCHIAVO [1,*], Gilles LABAT [2], Stéphane LEGAY [2], Raphaëlle SAVOIRE [1]

[1] Univ. Bordeaux, CNRS, Bordeaux INP, CBMN, UMR 5248, F-33600 Pessac, France.
[2] FCBA, Pôle Biosense – Chemistry-Ecotoxicology Laboratory, allée de Boutaut, 33028 Bordeaux, France.

***Corresponding author:** Christelle HARSCOAT-SCHIAVO, christelle.harscoat-schiavo@u-bordeaux.fr

Keywords: wood, phytosterol, supercritical CO_2 extraction, ASE (accelerated solvent extraction)

Wood is a local and renewable resource. It is underutilized, though it contains high value compounds, among which phytosterols. These compounds are of particular interest in various fields, including cosmetics (as moisturizer) and agriculture (as biostimulant for plant growth). There is therefore a great concern in exploiting this resource for the recovery of phytosterols.

Objectives

The objective of this research was to assess extraction assisted by supercritical CO2 (scCO2) for the recovery of sterols from wood, and to determine optimal conditions. The comparison with a classical ASE (accelerated solvent extraction) extraction was made in terms of phytosterol extraction yield and selectivity.

Methods

Wood material from various tree trunk parts of various granulometries were processed. Extractions were performed with about 4 g of wood using a home-made system with an extractor vessel of 10^3 cm , with supercritical CO_2 added with absolute ethanol as cosolvent. To determine the best conditions, operating conditions were varied: pressure from 150 to 250 bar, temperature from 40 to 60°C and cosolvent ratio from 0 to 10%. Extracts were sylilated then analysed by GC/MS-FID to quantify phytosterols. Results were expressed in terms of phytosterol extraction yield, that is mg sterol/kg dry wood. They were compared with a reference extraction with about 3 g of wood, carried out by ASE on a Dionex ASE system with hexane or absolute ethanol, in 4 cycles of 5 min each, at 120°C, 100 bar.

Results

ASE extractions showed that the bark is the richest part of the trunk in phytosterols, with a yield as high as 600 mg phytosterol/kg wood. First trials by scCO2 process highlighted that a low granulometry of the material was favorable to increase the extraction yield. Extractions with scCO2 carried out without cosolvent led to technical difficulties for recovering the extract. A pressure increase favored a higher extraction yield, whereas a too large cosolvent ratio was detrimental to selectivity since extracts contained a large amount of undesired co-extracted compounds. Similarly to ASE, phytosterol extraction yields of around 600 mg/kg wood could be achieved under 250 bar, 50°C with a cosolvent ratio of 5%.

PO-09. Eco-extraction of biomolecules of interest from microorganisms by coupling of innovative technologies

Sarah MAHFOUD [1,2], Nabil GRIMI [1,*], Thomas AUXENFANS [2]

[1] Université de Technologie de Compiègne, ESCOM, TIMR (Integrated Transformations of Renewable Matter), Centre de Recherche Royallieu-CS 60319-60203 Compiègne Cedex, France.

[2] LESAFFRE INSTITUTE OF TECHNOLOGY, 59 700 Marcq-en-Barœul, France.

***Corresponding author:** Nabil GRIMI, nabil.grimi@utc.fr

Keywords: extraction, microorganisms, nutritional value, eco-process, biomolecules

The world population is expected to reach 9 billion people by 2050. This poses the global challenge of meeting the increasing production demand while making food that is healthy and accessible for all. This takes into account the depletion of our global resources as well as consumers' growing favoritism of natural, clean-label products. Hence the importance of microorganisms, specifically yeasts and bacteria, as an abundant source of proteins, carbohydrates, minerals, nucleic acids, and lipids. These biomolecules have various applications in cosmetics, pharmaceuticals, energy, and particularly nutrition of both humans and animals. And since proteins are vital for our tissues and organs, the primary focus is on microbial proteins as sustainable alternatives to animal and plant proteins due to their full range of amino acid content and reported biological quality and digestibility. Yet proteins' techno-functional properties highly depend on their structure, although this relationship is still poorly understood which hinders the optimization of their recovery process. This study is thus aiming to concieve and develop a sustainable and scalable eco- process sequence supported by understanding the governing process-structure-function relationship of technological proteins among other biomolecules.

The recovery of these proteins involves mechanical (bead milling, high-pressure homogenization, ultrasonication) and non-mechanical (enzymatic, chemical, physical, electrical) cell disruption techniques. This study aims to further explore the potential of electrically-assisted extraction as an eco-friendly microbial cell disruption, individually or in various possible combinations. The disruption step is followed by a series of purification techniques to further separate or fractionate the target biomolecules including membrane filtration, pH variation, and drying. The process impact is studied with respect to both quantity and quality of the biomolecules in the separated/fractionated extracts. Quantitative evaluation implies assessing yields and the efficiency of cell disruption/disintegration. Qualitative evaluation implicates an assessment of both structure and function of the biomolecules and how they correlate. The process can thus be optimized back-and-forth for sustainably-produced, high nutritional value microbial biomolecules particularly for food and feed.

PO-10. Exploring *Artemisia absinthium*: Dual-use phytochemicals for combating cancer and bacterial resistance

Salma LOUKMAN [1], Hamza TACHALLAIT [1], Reda BENMRID [2], Najat BOUCHMAA [2], FatimEzahra MESRAR [1], Soukayna BAAMMI [1], Rachid EL FATIMY [2,*] and Rachid BENHIDA [1,3,*]

[1] Chemical and Biochemical Sciences department. Mohammed VI Polytechnic University, UM6P. Lot 660, Hay Moulay Rachid, 43150, Benguerir, Morocco.
[2] Institute of Biological Sciences (ISSB-P), Mohammed VI Polytechnic University (UM6P), 43150, Ben-Guerir, Morocco.
[3] Nice Institute of Chemistry. University Côte d'Azur, Nice, France.

***Corresponding authors:** Rachid BENHIDA, rachid.benhida@um6p.ma and Rachid EL FATIMY, rachid.elfatimy@um6p.ma

Keywords: Artemisia absinthium, phytochemical extraction, dual therapeutic potential, antibacterial activity, anticancer activity, molecular docking, ADMET profiling, extraction optimization.

Objectives

This study sought to optimize the extraction of bioactive compounds from Artemisia absinthium, assessing the impact of different solvents and extraction methods on yield and efficacy. Furthermore, it aimed to explore the dual therapeutic potential of these extracts, specifically targeting antibacterial and anticancer activities, to identify promising compounds for advanced therapeutic applications.

Methods

Artemisia absinthium, collected from Benguerir City, Morocco, was subjected to various extraction protocols using diethyl ether, ethanol, hexane, and water to maximize the recovery of bioactive compounds. The timing of collection was strategically chosen to coincide with peak phytochemical synthesis. Comprehensive chemical profiling was conducted via LC/MS, and elemental analysis was performed using LIBS. The study employed in silico ADMET profiling and molecular docking techniques to evaluate the pharmacokinetic properties and therapeutic interactions of the compounds with essential proteins involved in bacterial and cancer cell pathways.

Results

Ethanol maceration proved most effective for extracting terpenoids, utilizing its broad- spectrum solubility, while water-based UAE was notably efficient for hydrophilic terpenoids. Lipids were most efficiently extracted by diethyl ether due to its non-polar nature. Among the phytochemicals screened, casticin and dicaffeoylquinic acid III exhibited significant dual activity against both bacterial and cancer targets, showing high docking scores and potential as lead compounds for drug development. These findings highlight the importance of tailored solvent selection based on the specific solubility and desired therapeutic targets of the compounds.

PO-11. Extraction and analysis of minerals and heavy metals from black truffle by-products using non-conventional methods: pressurised liquid extraction and supercritical fluid extraction.

Albert SEBASTIÀ [1], Sergio MARTÍNEZ-TEROL [2], Mara CALLEJA-GÓMEZ [1], Katarzyna DAWIDOWICZ [1], Noelia PALLARES [1], Juan Manuel CASTAGNINI [1], Francisco J. BARBA [1], Paula BUCCI [3], Cristina ARCE-RECATALÁ [2], Elena ROSELLÓ-SOTO [1], Manuel SALGADO-RAMOS [1,4,*], Francisco J. MARTÍ-QUIJAL [1]

[1] Research Group in Innovative Technologies for Sustainable Food (ALISOST), Preventive Medicine and Public Health, Food Science, Toxicology and Forensic Medicine Department, Faculty of Pharmacy and Food Sciences, Universitat de València, Avda. Vicent Andrés Estellés, s/n, 46100 Burjassot, Spain.
[2] Faculty of Health Sciences, Valencia International University (VIU), C. del Pintor Sorolla, 21, 46002 València
[3] Department of Chemical Engineering and Environmental Technology, Institute of Sustainable Processes, University of Valladolid, Dr. Mergelina s/n., Valladolid 47011, Spain.
[4] Facultad de Ciencias y Tecnologías Químicas, Universidad de Castilla-La Mancha, Avenida Camilo José Cela s/n, 13071, Ciudad Real, Spain.

***Corresponding author:** Manuel SALGADO-RAMOS, manuel.salgado@uclm.es

Keywords: Pressurized liquid extraction, supercritical fluid extraction, truffle, minerals, heavy metals, cytotoxicity

Introduction

Black truffle (*Tuber melanosporum*) is an underground fungus of great prestige in gastronomy, recognised for its characteristic aroma and flavour. Apart from its value in cooking, it is an important source of minerals, although its cultivation environment may result in the presence of heavy metals in small quantities. However, a large amount of by- products are generated in its production. The use of these by-products derived from its cultivation and processing not only optimises its value, but also facilitates safer and more sustainable management of these resources.

Objectives

The aim of this study is to determine the mineral and heavy metal composition of black truffle by-product extracts obtained through various non-conventional extraction technologies, as well as to analyze the cytotoxicity of these extracts.

Methods

Two non-conventional extraction methods, Pressurised Liquid Extraction (PLE) and Supercritical Fluid Extraction (SFE), were compared. The conditions used for PLE were 100% ethanol, 3 extraction cycles, and a temperature of 120°C. For SFE, the parameters were 30 MPa, 40°C, 30 minutes of extraction at a flow rate of 16 ml/min, and 10% ethanol. Minerals (magnesium (Mg), phosphorus (P), potassium (K), calcium (Ca), iron (Fe), copper (Cu), zinc (Zn) and selenium (Se)) and heavy metals (arsenic (As), cadmium (Cd), mercury (Hg) and lead (Pb)) were analyzed using ICP-MS. Cytotoxicity was assessed in Caco-2 cells using the MTT assay after 24-hour exposure to extract concentrations ranging from 0.0625 % up to 1 % (v/v).

Results

The results indicated that the main minerals found in the truffle by-products (raw material) were Ca at 539 mg/kg and Mg at 477 mg/kg, with Se being the least abundant with a value of 0.127 mg/kg. Among the heavy metals, Cd was present in the highest concentration at 729.0 μg/kg, while Hg was the

lowest at 25.7 µg/kg. Regarding the obtained extracts, the predominant minerals were K and phosphorus P in both cases, with concentrations of 46.20 ± 1.30 mg/L and 7.74 ± 0.18 mg/L in the PLE extract, and 3.00 ± 0.11 mg/L and 0.48 ± 0.04 mg/L in the SFE extract, respectively. The PLE extract showed significantly higher values for P, K, Fe, Cu, and Zn, while the SFE extract had better recovery of Mg and Ca. There were no significant differences in Se between the two methods. Concerning heavy metals, As was the most prevalent in the SFE extract at 17.200 ± 0.300 µg/L, while Hg was the highest in the PLE extract at 1.020 ± 0.013 µg/L. Notably, As was not detected in the PLE extract, nor was Hg detected in the SFE extract. Finally, respecting to the cytotoxicity assays with Caco-2 cells, cell viability was above 80% at all tested concentrations in both extracts.

Conclusion

In conclusion, truffle by-products contain significant levels of minerals and heavy metals, with variations between extraction methods. The PLE extract was richer in key minerals, while the SFE extract excelled in Mg and Ca recovery. Both extracts demonstrated low cytotoxicity, maintaining over 80% cell viability. Understanding the composition of these extracts is crucial for exploring their potential in developing future antioxidant, antimicrobial, and other biologically functional treatments.

Acknowledgements

The authors would like to acknowledge Generalitat Valenciana for financial support (CIGE/2023/108 - Recuperación de nutrientes y compuestos bioactivos de co-productos de chufa y trufa y evaluación de sus propiedades saludables).

PO-12. Recovery of polyphenols from black truffle by-products using non-conventional technologies: Characterization of phenolic profile by triple-TOF-LC-MS-MS

Sergio MARTÍNEZ-TEROL [1], Albert SEBASTIÀ [1], Katarzyna DAWIDOWICZ [1], Mara CALLEJA-GÓMEZ [1], Juan Manuel CASTAGNINI [1], Noelia PALLARES [1], Francisco J. BARBA [1], Paula BUCCI [3], Elena ROSELLÓ-SOTO [1], Cristina ARCE-RECATALÁ [2], Francisco J. MARTÍ-QUIJAL [1,*], Manuel SALGADO-RAMOS [1,3]

[1] Research Group in Innovative Technologies for Sustainable Food (ALISOST), Preventive Medicine and Public Health, Food Science, Toxicology and Forensic Medicine Department, Faculty of Pharmacy and Food Sciences, Universitat de València, Avda. Vicent Andrés Estellés, s/n, 46100 Burjassot, Spain.
[2] Faculty of Health Sciences, Valencia International University (VIU), C. del Pintor Sorolla, 21, 46002 València, Spain.
[3] Department of Chemical Engineering and Environmental Technology, Institute of Sustainable Processes, University of Valladolid, Dr. Mergelina s/n., 47011 Valladolid, Spain.
[4] Facultad de Ciencias y Tecnologías Químicas, Universidad de Castilla-La Mancha, Avenida Camilo José Cela s/n, 13071, Ciudad Real, Spain.

*Corresponding author: Francisco J. MARTÍ-QUIJAL, francisco.j.marti@uv.es

Keywords: Pressurized Liquid Extraction, Supercritical Fluid Extraction, Truffle, Polyphenols, Triple-TOF-LC-MS-MS Introduction

The truffle (*Tuber melanosporum*) is a highly valued underground fungus in the world of gastronomy, renowned for its unique flavor and aroma. However, the process of cultivating and harvesting truffles generates a substantial amount of by-products that often go underutilized. These by-products are rich in valuable compounds, particularly antioxidants such as polyphenols, which have potential health benefits. Exploring ways to make better use of these by-products could open new opportunities in food production, nutrition, and even cosmetics, adding further value to this already esteemed ingredient.

Objectives

The aim of this study is the recovery of polyphenols from black truffle (Tuber melanosporum) by-products using two non-conventional processing techniques: Pressurised Liquid Extraction (PLE) and Supercritical Fluid Extraction (SFE). The phenolic profile of the samples will be examined by performing a tentative identification of the compounds using Triple-TOF-LC-MS-MS, and the results obtained from each of the technologies used will be compared.

Methods

For SFE, the conditions were set at 30 MPa pressure, 40°C temperature, a 30-minute extraction duration, a flow rate of 16 ml/min, and 10% ethanol. The PLE process was conducted with 100% ethanol, three extraction cycles, and a temperature of 120°C. The phenolic profile characterization was performed using an Agilent 1260 Infinity with a Waters UPLC C18 column 1.7 µm (2.1 × 50 mm) Acquity UPLC BEH.C18 for the separation of the main phenolic compounds in the samples. Moreover, a Triple TOF TM 5600 LC/MS/MS system was utilized for the identification.

Results

The results show a higher number of identified polyphenols in the PLE extract (122) compared to the SFE extract (33). In both cases, the majority group is flavonoids, accounting for 57.4% in PLE and 72.73% in SFE. Within the flavonoids in PLE, the predominant group is anthocyanins, representing

27.14% of the total flavonoids, followed by flavonols, flavanones, and flavones, each at 14.29%. In SFE, anthocyanins are also the main group, accounting for 45.83% of the total flavonoids, followed by flavanones at 16.67%, and flavonols, flavones, and dihydrochalcones each at 8.33%. In the PLE extract, phenolic acids were also detected, representing 24.59% of the total polyphenols. These phenolic acids are divided into hydroxycinnamic acids (70.00%), hydroxybenzoic acids (20.00%), hydroxyphenylpropanoic acids (6.67%), and hydroxyphenylacetic acids (3.33%). Additionally, other polyphenols such as tyrosols, hydroxybenzaldehydes, and alkylphenols were detected. Finally, in the SFE extract, tyrosols, hydroxyphenylpropenes, and alkylphenols were identified, along with lignans and stilbenes.

Conclusion

In conclusion, the PLE extract demonstrated a higher diversity of polyphenols compared to the SFE extract, with both methods predominantly yielding flavonoids, particularly anthocyanins, alongside a notable presence of phenolic acids and other polyphenolic compounds.

Acknowledgements

The authors would like to acknowledge Generalitat Valenciana for financial support (CIGE/2023/108 - Recuperación de nutrientes y compuestos bioactivos de co-productos de chufa y trufa y evaluación de sus propiedades saludables).

PO-13. Extraction of mustard bran oil using supercritical CO_2

Cloé DULLIER [1], Morad CHADNI [1,*]

[1] URD Agro-Biotechnologies Industrielles (ABI), CEBB, AgroParisTech, 51100 Pomacle, France

***Corresponding author:** Morad CHADNI, morad.chadni@agroparistech.fr

Keywords: Mustard bran, oil, extraction, supercritical CO_2

Objectives

Mustard bran is a by-product from mustard production that is undervalued. It is known to be rich in lipids (23-47%), proteins (16-40%) and phenolic compounds (0.2-3.7%) [1, 2] that can be valorized in many fields.

In this work, we have chosen to use supercritical CO_2 as the extraction solvent for lipids extraction. This technique offers real advantages compared to the most conventional hexane extraction method wich is toxic to both the environment and humans. Supercritical CO_2 is a naturally occuring, readily available gas that is non-toxic, inert, odorless, colorless, and tasteless. Its application does not alter the treated material or produce any polluting residues.

Methods

Mustard bran issued from mustard grown in Bourgogne was used in this study. The received material was not defatted, not ground nor dried.

Mustard bran was dried at 40°C to reach 92-93% of dry matter (DM), and sevied to obtain a fraction of 1.6 mm.

The oil extraction experiments were carried out on the SFE Lab 1L + Ultrasound Technology extractor (SFE Process, Nancy, France), 100 g of dried mustard bran were used for each experiment.

The aim of this study is to investigate influence of three parameters, extraction temperature (40, 60 and 80°C), pressure (100, 200, 400 and 600 bar) and flow rate (15, 30, and 60 g $CO_2.min^{-1}$), on oil extraction.

Results

Initial results indicate that the highest extraction yields were achieved under conditions of 400 bar pressure and 60 °C temperature, with a CO_2 flow rate of 60 $g.min^{-1}$, resulting in a yield of 15.6 g of oil. Under the same pressure but at temperatures of 40 °C and 80 °C, the yield was 13.8 g of oil. Additionally, it is important to note that at extreme conditions, either maximal temperature (80°C) or maximal pressure (600 bar), the total mass of extracted oil decreases compared to the optimum conditions identified.

References

[1] : Reungoat, V., Gaudin, M., Flourat, A. L., Isidore, E., Mouterde, L. M., Allais, F., ... & Ioannou, I. (2020). Optimization of an ethanol/water-based sinapine extraction from mustard bran using Response Surface Methodology. Food and bioproducts processing, 122, 322-331.

[2] : Nguyen, T., Nandasiri, R., Fadairo, O., & Eskin, N. M. (2024). Phenolics of mustard seeds: A review on composition, processing effect and their bioactvities. Journal of the American Oil Chemists' Society, 101(1), 5-21.

PO-14. From waste to cosmeceuticals: buccal in situ gelling formulation including a polyphenols-enriched secondary raw material from green recovery of grape processing waste

Elena BELFIORE [1], Giulia DI PRIMA [2], Serena INDELICATO [2], Cecilia LA MANTIA [2], Viviana DE CARO [2,3]

[1] Dipartimento Me.Pre.C.C., University of Palermo, Via L. Giuffrè 5, 90127, Palermo, Italy.
[2] Dipartimento STEBICEF, University of Palermo, Via Archirafi 32, 90123, Palermo, Italy.
[3] Centro Interdipartimentale RIVIVE, University of Palermo, 90128, Palermo, Italy.

***Corresponding author:** Elena BELFIORE, elena.belfiore@unipa.it

Keywords: waste valorization; green extraction; secondary raw material; polyphenols; PEG200; buccal gel; cosmeceuticals.

Objectives

Polyphenols have recently emerged as useful bioactives to treat or prevent numerous oral diseases. They are widely synthesized in the plant kingdom and thus recoverable by several agri-food by-products. In this perspective, the aim of research was to cover the entire value chain: i) valorization of the black bentonite (BB), a mixture of bentonite/activated carbon used as fining agent in the grape processing industry, as a valuable and novel source of polyphenols, ii) extraction and characterization of new high value-added secondary raw materials obtained by green maceration with PEGs, chosen as ecofriendly unconventional solvents, iii) development of a cosmeceutical in form of a buccal in situ gelling formulation to be administered as an oral care spray.

Methods

The extractions were carried out by maceration (1 h, 25°C, constant stirring, in the dark) using PEG200, PEG400 and PEG600 as solvents, at a fixed BB:solvent ratio of 1:4 w/w. The coloured liquid extracts were obtained by further centrifugation (5500 rpm, 1 h) and filtration (0.22 µm, nylon) and subjected to HPLC-DAD analysis, Folin-Ciocalteu, DPPH and Bradford assays. The complete phenolic footprint of PEG200-based extract was deeply investigated by HPLC-MS. Finally, the extract was inserted into an ad hoc designed liquid formulation containing Pluronic F-127, PVP K30, sodium dehydrocolate (SDS), urea and preservatives. The buccal gel was evaluated in terms of appearance, pH, temperature-depending gelling properties, antioxidant power and ex vivo behaviour.

Results

The PEG200-based extract emerged as the best one in terms of phenolic content (3.1±0.1 mgeq of gallic acid/g of extract) and thus antioxidant power (1.30±0.05 mgeq of gallic acid/g of extract after 1 h of DPPH assay). It exhibited a complex phenolic footprint and was then chosen as high value-added secondary raw material to be directly inserted into a cosmeceutical formulation. The latter was designed: i) to be fluid, clear and homogeneous and thus easily applicable as a spray into the oral cavity, ii) to gel at the body temperature (due to Pluronic F-127) maximizing the contact time with the mucosa (also thanks to PVP K30 mucoadhesive property); iii) to promote polyphenols penetration (due to SDS, PEG and urea) into the tissues. At this regard, the ex vivo studies carried out by using vertical Franz type cells and porcine mucosae highlighted that about the 3 and 6% of quercetin (the most representative compound) remained entrapped into the sublingual and buccal tissues respectively after just 15 min.

PO-15. Xanthophyll-loaded nanoemulsions from plant matrices: enhancement of carotenoid bioavailability with a sustainable process

Célia EL MARAHI [1], Patrick BOREL [2], Charles DESMARCHELIER [2,3], Christiane CONTINO-PÉPIN [1,*]

[1] Team SAFE, Avignon University, Avignon, France
[2] C2VN, INRAE, Aix-Marseille University, INSERM, Marseille, France 3 Institut Universitaire de France, France

***Corresponding author:** Christiane CONTINO-PÉPIN, christine.pepin@univ-avignon.fr

Keywords: Nanoemulsions, formulation, carotenoids, green extraction

Objectives

Our team has developed a new solvent-free process leading to green extracts formulated as nanoemulsions called "extremulsions" (for "extract-emulsions"). This ultrasound-assisted method allows the dual extraction and encapsulation of lipophilic natural products from plants in a oil-in-water emulsion. We aim to apply this process to the preparation of extremulsions loaded with two xanthophyl compounds, lutein and zeaxanthin, that are lipophilic pigments found in broccoli and paprika. These formulations will be tested on Caco-2 cells to assess their bioavailability compared to marketed dietary supplements.

Methods

Xanthophyll compounds are encapsulated in oil nanodroplets stabilized and solubilized in water using a tocopherol-derived surfactant, TPGS-1000. In this aim, the solubility of commercially available lutein was first investigated in various biocompatible oils, and the most effective oil was used to produce optimized nanoemulsions, in terms of droplet hydrodynamic diameter (DH) and size distribution (i.e. polydispersity index or PdI). Conditions yielding the best blank emulsions, with DH 300 nm and PdI 0.3, were then applied to extract lutein and zeaxanthin from broccoli and paprika. The resulting emulsions were characterized (DH and PdI assessed by dynamic light scattering, xanthophyll concentration analyzed by HPLC) and converted to powders after freeze-drying. We could obtain stable formulations that retained their properties upon rehydration. Preliminary studies of their bioavailability were also performed.

Results

We have developed the optimal conditions (oil quantity, surfactant concentration, ultrasound parameters, etc.) to formulate monodisperse nanodroplets smaller than 300 nm with the oil that best solubilizes lutein and zeaxanthin. We were also able to simultaneously extract and encapsulate these two xanthophylls from broccoli and paprika with a recovery rate of around 80% compared to the reference Soxhlet extraction.

PO-16. Macromolecular composition of *Corchorus olitorius* L.: toward the eco-extraction of hydrocolloids and other components

Emma BONNOT [1,2,*], Agnès ROLLAND-SABATÉ [3], Ombéline CLAUX [2], Mounir BOULKOUT [2], Maryline VIAN [1,*]

[1] UMR408 Sécurité et Qualité des Produits d'Origine Végétale, Equipe d'extraction GREEN, Université Avignon, F-84000 Avignon, France.
[2] SELT France Biotech, Vedène 84270, France.
[3] UMR408 Sécurité et Qualité des Produits d'Origine Végétale, INRAE, Université Avignon, F-84000 Avignon, France.

***Corresponding authors:** Maryline VIAN, maryline.vian@univ-avignon.fr and Emma BONNOT emma.bonnot@alumni.univ-avignon.fr

Keywords: *Corchorus olitorius* L, hydrocolloids, eco-extraction

Objectives

Many polysaccharides (PS) are not yet commercially exploited but already used in traditional recipes. These carbohydrate materials usually come from plants that grow in the wild or on a small scale. Therefore, their chemical composition and functional properties as food hydrocolloids remain mostly unknown. Among these plant sources that can be used to produce PS are the leaves of *Corchorus olitorius*, an annual herbaceous plant of the Malvaceae family commonly used for the extraction of natural fibers (Majumder et al., 2020). This plant, mostly found in Asia and Africa, can reach a height of 2-4 meters, and does not have specific climatic requirements other than heat (Loumerem & Alercia, 2016). Thus, the potential of *C. olitorius* as an innovative ingredient, will be evaluated through a macromolecular composition analysis, which will be followed by the eco-extraction of hydrocolloids, proteins, and other components.

Methods

The protein, lipid, carbohydrate, ash, and crude fiber contents were determined using methods based respectively on, the ISO norm 1871:2009 (N=4,64), the ISO norm 734:2023, the Dubois method for total sugars, the AOAC Official Method 942.05 and the ISO norm 6865:2000. More information on the PS was obtained from the alcohol-insoluble solids (AIS) as described by Renard et al., (2005). Neutral (water), acidic (HCl 0,05M) or alkaline (NaOH 0,05M) extractions were performed from these AIS. The methylation degree of pectin's, and the neutral and acid sugars composition of these extracts were then determined. Finally, SEC-MALLS (size-exclusion chromatography coupled with multiangle laser light scattering) was used for the macromolecular characterisation of the PS.

Results

The macromolecular composition of *C. olitorius* present this plant as a potential innovative, functional ingredient. The PS characteristics, which depend on the extraction method, will provide insight into the anticipated properties of the hydrocolloids, including viscosity and gelling properties.

It will therefore be essential to extract efficiently the PS without compromising its structure. Several green extraction methods will be tested to determine the most effective one on *C. olitorius*.

References

Loumerem, M. and Alercia, A. (2016) 'Descriptors for jute (Corchorus olitorius L.)', Genetic Resources and Crop Evolution, 63(7), pp. 1103–1111. Available at: https://doi.org/10.1007/s10722-016-0415-y

Majumder, S. et al. (2020) 'Fiber crop, jute improvement by using genomics and genetic engineering', in Advancement in Crop Improvement Techniques. Elsevier, pp. 363–383. Available at: https://doi.org/10.1016/B978-0-12-818581-0.00022-X

Renard, C.M. (2005) 'Effects of conventional boiling on the polyphenols and cell walls of pears', Journal of the Science of Food and Agriculture, 85(2), pp. 310–318. Available at: https://doi.org/10.1002/jsfa.1987

PO-17. Optimization of Pulsed electric fields-assisted extraction parameters of lutein from Marigold (*Tagetes erecta* L.) using response surface methodology.

Annachiara PIROZZI [1,2], Francesco DONSÌ [1,2], Gianpiero PATARO [1,2,*]

[1] Department of Industrial Engineering, University of Salerno, Via Giovanni Paolo II, 132, 84084, Fisciano, SA, Italy.
[2] NBFC- National Biodiversity Future Center, 90133 Palermo, Italy.

***Corresponding author:** Gianpiero PATARO, gpataro@unisa.it

Keywords: Marigold, Lutein; Extraction; Pulsed Electric Fields (PEF); Response Surface Methodology; HPLC.

Objectives

Marigold (*Tagetes erecta* L.) is the primary industrial source of lutein, a carotenoid with extensive applications in the food and health sectors. Traditionally, lutein has been isolated using solid/liquid extraction (SLE) techniques with petrochemical-based solvents. However, these conventional extraction processes have significant drawbacks, including long processing times and high usage of chemicals with inherent toxicity. The application of Pulsed Electric Fields (PEF) as a mild and easily scalable electrotechnology represents an effective strategy to intensify the extractability of lutein from Marigold.

Methods

The study focused on enhancing lutein extraction yields from Marigold through an innovative Pulsed Electric Fields (PEF)-assisted solid/liquid extraction (SLE) process, utilizing Response Surface Methodology (RSM) with a Face-Centered Central Composite Design (FC-CCD). The cell disintegration index (Zp) was used as a response variable to determine the optimal PEF processing conditions, evaluating various field strengths (E = 0.5-5 kV/cm) and total specific energy inputs (WT = 1-20 kJ/kg). For the SLE process, the effects of three independent variables - solid to solvent ratio, temperature, and time - were assessed on the lutein yield from both untreated and PEF- treated plant tissues. This was done using ethanol as green solvent. The extracted lutein was characterized using HPLC analysis.

Results

Results demonstrated that PEF treatment conditions 3.2 kV/cm and 14.6 kJ/kg enable to achieve the maximum degree of cell membrane permeabilization (Zp=0.94). The application of PEF pre-treatment under this optimal condition prior to SLE enabled to sustainably enhance the extraction yield of oleoresin from Marigold flowers, by reducing the extraction time and the processing temperature of about 5 h and 10 °C, respectively, compared to conventional SLE. HPLC analysis clearly showed that the application of PEF markedly enhanced the recovery yield of lutein without inducing any degradation phenomena. Further studies are necessary to valorize the defatted residual biomass, typically discarded or used as manure, by extracting bioactive compounds such as phenols and flavonoids, which possess high antioxidant and anti-inflammatory activities.

PO-18. Lignin phosphorylation for bio-based resin applications

Moustapha FALL [1,2,*], Yassine EL KHAYAT DRIAA [1,3], Nabil GRIMI [1], Amine MOUBARIK [3], Nadia BOUSSETTA [1]

[1] Integrated Transformation and Renewable Matter TIMR (UTC/ESCOM), University of Technology of Compiegne- Alliance Sorbonne University, Centre of Research of Royallieu, Rue du docteur Schweitzer, CS 60319, 60203 Compiegne, France.
[2] Laboratory of Agroressources, Biomolecules and Chemistry for Innovation and Health (LABCiS UR 22722) of Limoges University, la Borie City, 123 Albert Thomas Avenue, 87060 Limoges Cedex-France.
[3] Chemical Processes and Applied Materials Laboratory, Polydisciplinary Faculty, Sultan Moulay Slimane University, SBP: 592 Beni Mellal, Morocco.

***Corresponding author:** Moustapha FALL, moustapha.fall@etu.utc.fr

Keywords: Lignin, phosphorylation, phosphoric acid, di-ammonium hydrogen phosphate.

Objectives

The primary objective of this study is to find a method for phosphorylating lignin extracted from almond shells for use in bio-based resins, aiming to improve their flame- retardant properties. To achieve this, two phosphorylation agents were used: phosphoric acid and ammonium dihydrogen phosphate (DAP). The goal is to identify which of the two is optimal for the phosphorus content in the treated lignin and to verify the feasibility of the experimental conditions. Each step of the phosphorylation protocol with the best agent is refined to obtain treated lignin with a high phosphorus content, so that bio- composites and adhesive materials based on phosphorylated lignin exhibit increased thermal stability.

Methods

The phosphorylation protocol is identical for both phosphorylating agents. A solution containing 30% by mass of phosphorylating agent (H3PO4 or NH4H2PO4) is introduced into a flask and heated to 80 °C, where it is maintained at reflux. Then, 1 g of urea is gradually added to the solution. After approximately 15 minutes, when the urea is completely dissolved, 1 g of lignin is added gradually. The mixture is stirred for 1 hour, then dried in an oven at 75 °C for 24 hours, followed by curing at 150 °C for 2 hours in the oven. Subsequently, the treated lignin is washed with ethanol and then dried at 75 °C for 24 hours before being analyzed by Energy Dispersive X-ray Spectroscopy (EDX) to determine the phosphorus content.

Results

The lignin treated with phosphoric acid exhibits a significantly prolonged drying time, requiring several days, and becomes hygroscopic during curing, which hinders effective drying and causes it to absorb moisture at room temperature. In contrast, lignin treated with DAP begins to dry after some time when placed in an oven. EDX analysis shows comparable phosphorus levels in the treated lignin for both phosphorylating agents. Under these conditions, DAP appears more promising for phosphorylating lignin extracted from almond shells. Optimizing parameters such as the amount of urea, time, temperature, and the quantity of the phosphorylating agent is crucial to achieve an optimal phosphorus content.

PO-19. Optimizing cellulose phosphorylation for enhanced thermal properties and flame retardance in wood adhesives

Yassine EL KHAYAT DRIAA [1,2,*], Hafida MAARIR [2], Nabil GRIMI [1], Amine MOUBARIK [2], Nadia BOUSSETTA [1]

[1] Integrated Transformation and Renewable Matter TIMR (UTC/ESCOM), University of Technology of Compiegne- Alliance Sorbonne University, Centre of Research of Royallieu, Rue du docteur Schweitzer, CS 60319, 60203 Compiegne, France.
[2] Chemical Processes and Applied Materials Laboratory, Polydisciplinary Faculty, Sultan Moulay Slimane University, SBP: 592 Beni Mellal, Morocco.

***Corresponding author:** Yassine EL KHAYAT DRIAA, yassine.el-khayat-driaa@utc.fr

Keywords: Biomass, lignocellulose, cellulose, functionalization, phosphorylation.

Objectives

The primary objective of this research is to optimize the phosphorylation process of cellulose to significantly enhance its thermal properties and flame retardance, aiming to develop superior wood adhesives. By meticulously refining each step of the phosphorylation protocol, the goal is to achieve a material that not only withstands higher temperatures but also significantly reduces flammability. This enhancement in thermal stability and flame resistance will ensure that the wood adhesives are more durable and safer for various applications.

Methods

The cellulose phosphorylation protocol involves a series of precise steps to ensure an efficient and controlled process. Initially, water is heated in a reaction flask, followed by the addition of urea under stirring for homogeneous dissolution. Diammonium hydrogen phosphate (DAP) is then added, and the mixture is stirred to ensure uniform distribution. Cellulose is introduced and allowed to react, after which the sample undergoes initial drying at 60°C, curing at 150°C, washing to remove residues, and final drying at 60°C. This results in a purified and dry cellulose phosphorylated product ready for further applications.

Results

It has been observed that the amounts of urea and diammonium hydrogen phosphate (DAP), as well as the reaction duration, significantly influence the phosphorylation process. In contrast, temperature and water quantity showed no notable effect. These findings suggest that to optimize phosphorylation, it is crucial to focus on precisely adjusting the amounts of urea and DAP and controlling the reaction time, while variations in temperature and water can be less rigorously controlled without compromising the process efficiency.

PO-20. Physically and chemically cross-linked hybrid hydrogels for the controlled extraction and release of medications

O. SAMOYLENKO [1,*], Yu. SAMCHENKO [2], I. GANUSEVICH [1], L. KERNOSENKO [2], A. VERBINENKO [1], N. PASMURTSEVA [2], T. POLTORATSKA [2], N. LEBOVKA [2]

[1] RE Kavetsky Institute of Experimental Pathology, Oncology and Radiobiology, National Academy of Sciences of Ukraine, Kyiv, Ukraine
[2] The Ovcharenko Institute of Biocolloidal Chemistry of National Academy of Sciences of Ukraine, Kyiv, Ukraine

***Corresponding author:** Olena SAMOYLENKO, a-samoilenko@ukr.net

Keywords: "smart" hydrogels, controlled extraction, medications, Laponite®

Objectives

In general, bacterial infections associated with biomedical devices and implants have caused a serious problem for the global healthcare system. These infections are mainly associated with bacterial biofilms that form on the surface of biomaterials, protecting encapsulated bacteria from traditional antibiotic therapy. Development of hybrid hydrogels filled with medications is the important task for prevention the occurrence of an inflammatory reaction around the implant.

Methods

Different synthesis procedures have been applied for preparation of “smart” Poly(N-isopropylacrylamide) (PNIPAm)-based hydrogels, physically cross-linked by modified Lap platelets, and hybrid hydrogels chemically cross-linked in polyvinyl formaldehyde (PVF) sponges. The modification of Lap platelets was performed by acid activation in sulfuric acid. Prepared hydrogels were saturated with different medications. The swelling degree and diffusion properties of smart hydrogels towards medications were also analyzed.

Results

For physically cross-linked hydrogels increase in the level of acid-activation resulted in significant increase of the maximum swelling degree. Acid activation also significantly affected the diffusion properties of medications in developed hydrogels. For prorous samples of PVF sponges effects of squeezing on swelling degree and medication extraction kinetics are also discussed.

PO-21. Subcritical water extraction of bioactive compounds from red ginseng marc

Aye Aye MYINT [1,2], Ruqian CAO [2], Jaehoon KIM [1,2,3,*], Dong Jin SUH [4,*]

[1] School of Chemical Engineering, Sungkyunkwan University, Suwon, Republic of Korea
[2] School of Mechanical Engineering, Sungkyunkwan University, Suwon, Republic of Korea
[3] Sungkyunkwan Advanced Institute of Nano Technology, Sungkyunkwan University, Suwon, Republic of Korea
[4] Woori Plant, Yeosu, Republic of Korea

***Corresponding authors:** Jaehoon KIM, jaehoonkim@skku.edu and Dong Jun SUH, djsuh@kist.re.kr

Keywords: Red ginseng marc, subcritical water extraction, waste valorization, bioactive compounds, antioxidants

Objectives

Red ginseng marc (RGM), a byproduct obtained using manufacturing various ginseng products, which is typically discarded as waste, contains numerous residual bioactive compounds including transformed ginsenosides. The main objective of this study is to determine the efficiency of subcritical water extraction (SWE) technique in the recovery of valuable bioactive compounds from RGM. Besides, the bioactivities of the extracted products were assessed by three different antioxidant activity assay methods: 2,2- diphenyl-1-picrylhydrazyl (DPPH) assay, Trolox equivalent antioxidant capacity (TEAC/ABTS) assay, and ferric reducing antioxidant power (FRAP) for the aspects of its potential applications in cosmescuticals, pharmaceuticals, and function foods.

Methods

In this study, SWE was performed in the temperature range of 140–200°C for an extraction time in the range of 15–90 min. Extraction yields, total phenolic compounds, browing intensity, ginsenosides, diverse bioactive chemical species, and antiradical activities of the extracts obtained under various SWE conditions were determined using high-performance liquid chromatography (HPLC), gas chromatography-time-of-flight mass spectrometry (GC-TOF/MS), and gel permeation chromatography (GPC). Additionally, conventional Soxhlet extraction of the RGM was performed to assess the performance of SWE technique on the RGM and quality of the extract obtained. Lastly, possible reaction pathways for the production of various intrinsic and transformed ginsenosides during the SWE were proposed.

Results

This study demonstrated that SWE is a promising green, sustainable and efficient method for recovering highly bioactive compounds from the RGM. Under the optimized conditions (200°C and 15 min), an extraction yield of 48 wt% was achieved, which was 1.8 and 4.1 times higher than those achieved via Soxhlet extraction for 8 h with water and 80% ethanol, respectively. In addition, the antioxidant activity of the SW extract was 2.7–8.7 times greater than those of the extracts obtained using Soxhlet method with water and 80% ethanol, respectively. The strong antioxidant activities of the SW extracts were attributed to their higher total phenolic contents and abundant Millard reaction products. Consequently, the high bioactivity of SW extract can be employed as value-added compounds in various sectors, including functional food, cosmeceutical, and pharmaceutical industries.

PO-22. Supercritical CO_2 extraction of ß-carotene and lipids from the oleaginous yeast *R. toruloides*

Vanessa BUCHWEITZ [1], Kilian JÖRG [1], Simon Vlad LUCA [1], Mirjana MINCEVA [1,*]

[1] Biothermodynamics, Technical University of Munich, Germany

***Corresponding author:** Mirjana MINCEVA, mirjana.minveca@tum.de

Keywords: supercritical CO_2, ß-carotene, oleaginous microorganism

Objectives

The production of hydrophobic bioactive components with oleaginous microorganisms as an alternative to their direct extraction from plants and chemical synthesis has gained interest.[1,2]
The oleaginous red yeast *Rhodosporidium toruloides* produces cellular lipids (around 20 % of the dry cell weight), which can act as a co-solvent, and carotenoids, like ß-carotene.[3] Organic solvents, such as chloroform or hexane, are mostly used to extract hydrophobic components from biomass. However, due to their inherent health and environmental risk, seeking greener alternatives, such as supercritical fluids, is crucial.[4,5]

Methods

Supercritical CO_2 extraction is usually performed with dried samples, which would require an energy-intensive drying step before the extraction from the aqueous biomass.[6] Therefore, in this study, supercritical CO_2 extraction with wet biomass (moisture content >80 wt%) was investigated. Since the target components are produced intracellularly, the cells were lysed using a high-pressure homogenizer first, and water was removed by filtration.

Suitable CO_2 extraction process parameters (pressure 200 to 300 bar and temperature 40 to 70°C) were determined, and the influence of the water content of the biomass on the extraction kinetic and the ß-carotene and total extraction yield were investigated. Furthermore, a possible increase in extraction yield by the usage of ethanol as a co-solvent was examined.[7]

Results

An increase in temperature had no influence on the total extraction yield and slightly decreased the ß-carotene yield but shortened the time to reach the maximum yield. Since ß-carotene is thermally unstable a lower temperature that still achieved high yields was selected. In contrast, the pressure influenced both the total extraction yield and the ß-carotene yield, with the highest yields at higher pressures.

For all extraction kinetics, a delay at the start of the extraction of the lipids and ß- carotene was observed, which most likely occurred due to the water removal from the biomass at the beginning of the extraction process. To determine if this delay was caused by the presence of water in the biomass, the extraction kinetics were measured with a biomass with only 8 wt% water content. It could be observed that there was no delay at the start of the extraction of the lipids and ß-carotene when the water content was reduced, which means that the water content influences the extraction kinetics, and at the beginning of the process, water is removed from the system. Nevertheless, there was no difference in the achievable total extraction yield and ß-carotene yield, which concludes that the water content does not influence the extraction yields. Additionally, the usage of ethanol as a co-solvent to increase the extraction yield was studied, and an increase in both the total extraction yield and the ß-carotene yield could be observed.

In conclusion, supercritical CO_2 extraction is suitable for the extraction of ß-carotene and lipids from wet biomass of oleaginous microorganisms, and the extraction yields can be increased by using ethanol as a co-solvent.

Acknowledgement:

This work was financially supported by Federal Ministry of Education and Research Germany (BMBF) in the framework of the project OleoBuild (grant number: 031B0853A).

References:

(1) Szczepańska, P., et al., Crit. Rev. Biotechnol., 2022. 42 (1), 1-22.
(2) Ayadi, I., et al., Biomass Convers. Biorefinery, 2024, 1-19.
(3) Xie, Z. T., et al., Appl Microbiol Biotechnol., 2024. 108(1), 1-14.
(4) de Jesus, S. S., et al., Renew. Sustain. Energy. Rev., 2020. 133, 110289.
(5) Buchweitz, V., et al. J. Chem. Eng. Data, 2024. 69,3, 1124-1130.
(6) Crampon, C., et al., J. Supercrit. Fluids, 2013. 79, 337-344. (7) Haq, M., et al., J. Clean. Prod., 2018. 178, 186-195.

PO-23. Synthesis and characterization of lignin nanoparticles

Yanqin DENG [1], Erwann GUENIN [1,*], Nabil GRIMI [1,*], Lucia PANZELLA [2], Vincent TERRASSON [1]

[1] Université de Technologie de Compiègne, ESCOM, TIMR (Integrated Transformations of Renewable Matter), Centre de Recherche Royallieu-CS 60319-60203 Compiègne Cedex, France.
[2] Department of Chemical Sciences, University of Naples "Federico II", Via Cintia 4, I-80126 Naples, Italy.

***Corresponding author:** Erwann GUENIN, erwann.guenin@utc.fr ; Nabil GRIMI, nabil.grimi@utc.fr

Keywords: Lignin nanoparticles, green preparation, self-assembly, batch process, continuous flow

Objectives

With our continued in-depth understanding of the environmental pollution and resource crisis, biomass materials' renewable and degradable properties are increasingly valued [1,2]. As the second most abundant natural polymer material after cellulose, lignin has recently received extensive attention [3,4]. The development of bio-based products from lignin is an important part of any comprehensive biorefinery concept because of their biocompatibility and biodegradability [5]. Lignin nanoparticles (LNPs) have potential applications in antioxidants, thermal/light stabilizers, reinforced materials and nano microcarriers owing to their advantages of non-toxicity, environmental resistance, excellent thermal stability and biocompatibility [6]. The current preparation methods of LNPs mainly include anti-solvent precipitation, mechanical methods, self-assembly, etc. In our study, a green approach to the preparation of homogeneous and stable LNPs is presented. In addition, the synthesis of LNPs in batch reactor mode limits industrial production, thus we also explored green continuous flow synthesis of LNPs.

Methods

Lignin has heterogeneous nature and different complex chemical structures due to different sources [7]. Thus we prepare LNPs from different resources (lignosulfonic, alkali lignin, peanuts lignin, pistachio lignin, hazelnut lignin, pecan lignin, chestnut lignin). The batch mode and continuous flow mode to prepare LNPs were described by Zhang et al. [8,9]. The lignin solution (2.5%, w/w) was prepared by dissolving 2 g of lignin in 80 g of ethylene glycol in a 250 mL beaker, the speed to add nitric acid was 2.67 mL/min. Then lignin solution was treated by an ultrasonic for 1 h at 24 kHz frequency and dialysis for 4 days in deionized water. The morphology and size of LNPs were characterized by transmission electron microscopy (TEM). Hydrodynamic diameter as well as zeta potential were measured by Dynamic Light Scattering measurement (DLS) over several days to assess nanoparticle stability. FTIR and NMR were used to characterize LNP and compare it to the parent lignin. Thermo Gravimetric Analysis (TGA) was used to study their thermal stability. UV absorbance properties were also evaluated by UV spectrometry. These characterizations were used to compare the differences of LNPs synthesized by batch mode and continuous flow mode.

Results

The LNPs around 200 nm were prepared through the π-π interactions between molecules in the self-assembly process, and they can maintain electrostatic stability in water. In a wide pH range (5-10), LNPs can stay stable in size and potential, and would not agglomerate over long periods in solution. Compared with lignin, the FTIR absorb peaks of LNPs had some changes, indicating that the structure of LNPs had changed by the synthesis process. It indicated that more phenolic hydroxyl groups of LNPs had been exposed on the surface when the particles were formed, or the structure of LNPs had been changed by ultrasonic process. We can also find the differences in FTIR between different sources of

lignin, and it can be verified by NMR results. The high- quality and renewable LNPs will provide a novel perspective for multifunctional and diverse applications of bio-based nanomaterials.

References

[1] Figueiredo, P.; Prog. Mater. Sci. 2018, 93, 233-269.
[2] Moon, R.J.; Chem. Soc. Rev. 2011, 40, 3941-3994.
[3] Qian, Y.; Green Chem. 2015, 17, 320-324.
[4] Sipponen, M.H.; ChemSusChem 2019, 12, 2039-2054. [5] Dessbesell, L.; Renew. Sustain. Energy Rev. 2020, 123. [6] Henn, A.; World J. Microbiol. Biotechnol. 2019, 35, 125. [7] Tian, D.; Biotechnol Biofuels. 2017, 10, 157
[8] Zhang, Z.;Nanomaterials, 2021, 11(5), 1336
[9] Assaf, I.; Biological Macromolecules, 2023, 125106

PO-24. Unveiling the multiscale structural dynamics and retrogradation behavior of potato starch via integrated enzymatic hydrolysis enhanced by microwave

Hongwei CAO [1,2], Jing TAN [1], Caiyun LIU [1], Nabil GRIMI [3], Xiao GUAN [1,2,*]

[1] School of Health Science and Engineering, University of Shanghai for Science and Technology, Shanghai, PR China, 200093.
[2] National Grain Industry (Urban Grain and Oil Security) Technology Innovation Center, University of Shanghai for Science and Technology, Shanghai, PR China, 200093.
[3] Sorbonne Universités, Laboratoire de Transformations Intégrées de la Matière Renouvelable, Université de Technologie de Compiègne, France

***Corresponding author:** Xiao GUAN, gnxo@163.com

Keywords: mashed potato; β-amylase; microwave; starch retrogradation

Abstract: This research investigated the physicochemical, structural, and retrogradation properties of MW combined with β-amylase treatment of mashed potato (MP) at different hydrolysis times, and then elucidated the development of starch multiscale structure and its influencing mechanism. The MW significantly enhanced the hydrolysis efficiency of potato starch, reducing the amylose content and consequently decreasing the storage modulus(G') and loss modulus(G"). The hardness analysis in the TPA showed that anti retrogradation after 20 min of MW treatment increased by 5% (14 d) compared to the conventional water bath (WB), which indicated that MW radiation was more effective in improving MP retrogradation compared to the WB. In a single enzyme activity test, the MW was found to significantly increased the activity of β-amylase. In the single enzyme activity test, the MW was found to significantly increased the activity of β-amylase. In the pure simulated reheating process, the structure of starch was found to be more easily to damage in the MW reheating environment by Fourier transform infrared spectroscopy (FTIR), X-ray diffraction (XRD) and scanning electron microscopy (SEM). The enzymatic treatment caused further damage to the structure of starch. In addition, the crystallinity of WB enzymatically digested MP significantly increased by 2.17% during retrogradation, and this significant change was further conclusively verified by the change in ΔH of retrogradation measured by differential scanning calorimetry (DSC). Therefore, this research demonstrated that the MW technology not only promotes the degradation of starch chains, but also effectively inhibits MP regrowth by accelerating the enzymatic reaction.

PO-25. Purification and concentration of C-Phycocyanin from spirulina extract by the combination of $CaCl_2$ precipitation and membrane diafiltration technologies.

Wanru ZENG [1], Jianquan Luo [2], Nabil GRIMI [1,*]

[1] Université de Technologie de Compiègne, ESCOM, TIMR (Transformations Intégrées de la Matière Renouvelable), Centre de Recherche Royallieu, CEDEX CS 60319, 60203 Compiègne, France
[2] Institute of Process Engineering, Chinese Academic of Sciences, Beijing, China

***Corresponding author:** Nabil GRIMI, nabil.grimi@utc.fr

Keywords: spirulina, ultrasonication extraction, salting-out, membrane filtration, C-PC purification

Objectives

The primary objective of this study is to enhance the efficacy of extracting phycocyanin from spiral microalgae, thereby increasing the yield. Currently, the efficiency of the extraction process is suboptimal, posing a hindrance to the industrial progression of the C-PC extraction industry.

Methods

The spirulina microalgae suspension was subjected to ultrasonication at 200W and 5°C for a duration of 5 min. Following centrifugation, the resulting supernatant was collected and mixed with a calcium chloride solution at a concentration of 10 mM. This mixture was allowed to stand at room temperature for 1 h before undergoing another round of centrifugation. Subsequently, the salt ions were eliminated through a membrane diafiltration process with membrane with molecular weight cut-off of 150 kDa.

Results

In this work, we enhanced the extraction efficiency of C-PC from spirulina microalgae through the application of ultrasonication. A processing duration of 5 min led to the production of C-PC with a yield of 126 mg/g. Subsequently, we improved the purity of C-PC from 0.56 to 1.02 by selectively eliminating other proteins, particularly chlorophyll-protein complexes, via $CaCl_2$ precipitation. Through the integration of membrane technology, the removal of salt and other substances was achieved through diafiltration, resulting in a C-PC purity of 1.23.

PO-26. Cultivation of two psychrophilic microalgae *Sphaerocystis* sp. and *Pleurastreum* sp. for carotenoids bioproduction

Adila GHERABLI [1,2], Julien LEMAIRE [2], Madeleine CHARBONNIER [2], Fanny DUVAL [2], Nabil GRIMI [1,*]

[1] Université de technologie de Compiègne, ESCOM, TIMR (Integrated Transformations of Renewable Matter), Centre de recherche Royallieu - CS 60319 - 60203 Compiègne, Cedex, France.
[2] Université Paris-Saclay, CentraleSupélec, Laboratoire de Génie des Procédés et Matériaux, Centre Européen de Biotechnologie et de Bioéconomie (CEBB), 3 rue des Rouges Terres 51110 Pomacle, France

***Corresponding author:** Nabil GRIMI, nabil.grimi@utc.fr

Keywords: psychrophilic microalgae, cultivation and stress, carotenoids, astaxanthin, canthaxanthin antioxidant

Objectives

Researchers have been exploring the potential of microalgae-derived carotenoids in a wide range of industries, from food and cosmetics to pharmaceuticals. Specifically, there is growing interest in microalgae strains that thrive in cold climates, known as ''psychrophilic microalgae'', as they offer promise as sources of valuable carotenoids, such as astaxanthin and canthaxanthin. However, despite their potential, there is a lack of comprehensive data on these specific microalgae, highlighting the need for further research. Therefore, this thesis project focused on enhancing the accumulation of carotenoids in two carefully selected psychrophilic strains, *Sphaerocystis* sp., and *Pleurastreum* sp., which have shown effective adaptation to laboratory settings, promoting cell growth, carotenoid accumulation, and extraction.

Methods and Results

In this research work, the culture process was carried out in two stages, referred to as green and red, with meticulous monitoring of parameters such as temperature, lighting, and suitable medium culture BBM (Bold Basal Medium). The project has demonstrated encouraging preliminary results, even at small flasks scale (< 100 mL) and in a 6-liter photobioreactor. Firstly, the transition between different cell stages has been successful, indicating healthy growth and development of the microalgae at temperatures below 12 °C, light intensity < 100 µmol/m^2/s, and 1% of CO_2. The growth rate achieved was between 0.1 and 0.2 day^{-1}, which is close to the result of reference work by T. Leya of 0.2 day^{-1}. Secondly, the reddish-orange pigmentation of the culture, a key indicator of carotenoid production, was achieved during the adaptation to various abiotic stresses. These stresses included azote and phosphor deficiency, high salinity (6 g/L), and high light intensity (225-325 µmol/m^2/s), applied alone or in combination. Lastly, psychrophilic microalgae are known to be less productive. Still, they exhibit a wide variety of pigments, including chlorophylls a and b, astaxanthin, canthaxanthin, and lutein, as revealed by high-performance liquid chromatography analysis. The mechanical pretreatment significantly facilitated the extraction of carotenoids, particularly through bead beating in solvents such as ethanol, resulting in a high pigment recovery yield. The total carotenoid content ranged from 2 to 6 mg/g of dry mass in ethanol.

PO-27. Valorization of chitosan extracted from shrimp shell waste for manufacturing of particleboard composites

Hassan CHARII [1,*], Abdelghani BOUSSETTA [2], Kamal BENALI [3], Anass EL OUADDARI [4], Kamal ESSIFI [5], El-Houssaine ABLOUH [2], Nadia BOUSSETTA [6], Henri EL ZAKHEM [7], Nabil GRIMI [6], Samir IBENMOUSSA [4], Khalid BOUTOIAL [8] and Amine MOUBARIK [1,*]

[1] Chemical Processes and Applied Materials Laboratory, Poly disciplinary Faculty, Sultan Moulay Slimane University, BP 592, Beni-Mellal, Morocco.
[2] Materials Science, Energy and Nanoengineering (MSN) Department, Mohammed VI Polytechnic University, Lot 660 – Hay Moulay Rachid, 43150, Benguerir, Morocco.
[3] Chemical and Biochemical Sciences, Green Process Engineering Department (CBS), Mohammed VI Polytechnic University, Lot 660 – Hay Moulay Rachid, 43150, Benguerir, Morocco.
[4] Laboratory of Chemistry, Biochemistry, Nutrition, and Environment, Faculty of Medicine and Pharmacy, University Hassan II, 20000 Casablanca, Morocco
[5] Coordination and Analytical Chemistry Laboratory, Faculty of Sciences, University of Chouaïb Doukkali, El Jadida, Morocco
[6] Sorbonne Université, Université de Technologie de Compiègne, Laboratoire Transformations Intégrées de la Matière Renouvelable (UTC/ESCOM, EA 4297 TIMR) Centre de recherches Royallieu, CS 60 319, 60 203, Compiègne Cedex, France
[7] Chemical Engineering Department, University of Balamand, PO Box 33, El Koura, Amioun, Lebanon.
[8] Laboratory of the Engineering and Applied Technologies, Higher School of Technology, Sultan Moulay Slimane University Higher School of Technology, Mghila University Campus, Pb 591 Béni Mellal, Morocco

***Corresponding authors:** Hassan CHARII, hassan.charii@usms.ma and Amine MOUBARIK, a.moubarik@usms.ma

Keywords: Adhesive, chitosan, particleboards, urea-formaldehyde, shrimp shell waste

Objectives

The objective of this research is to extract chitosan (CS) from shrimp shell waste (SSW) and phosphorylate it using diammonium hydrogen phosphate (DAP) and urea to produce phosphorylated chitosan (P-CS). The study also aims to explore the potential of P-CS as an alternative adhesive for wood composites, especially when combined with urea formaldehyde (UF).

Methods

Chitosan was isolated from shrimp shell waste and phosphorylated using DAP and urea. Specifically, CS and P-CS were characterized using SEM, ATR-FTIR, XRD, TGA/DTG, conductometric titration, and DS determination. Particleboard (PB) panels were made from UF mixes with different percentages of CS and P-CS (4, 6, 8, and 10 wt%). The mechanical characteristics of the resultant PBs were assessed, including dry internal bond (IB), modulus of elasticity (MOE), modulus of rupture (MOR), and surface soundness (SS). Thickness swelling (TS) and water absorption (WA) of PBs were also studied.

Results

The mechanical results showed that the optimum amount of P-CS (8wt%) added to UF increased the dry internal bond (IB = 0.65 ± 0.03 MPa), modulus of elasticity (MOE = 3659 ± 14 MPa), modulus of rupture (MOR = 28 ± 0.70 MPa), and surface soundness (SS = 1.88 ± 0.04 MPa) by 44.44 %, 43.26 %, 75 %, and 19.74 % compared to the control UF:P-CS (100:0; w: w), respectively. The study found that the PBs with the optimal resin UF: P-CS (92/8; w: w) were decreased values of thickness swelling (TS) and water absorption (WA) compared to the control UF: P-CS (100/0; w: w). Specifically, the values for TS were 8.06 ± 0.09 and 10.99 ± 0.27 (%) after 2 and 24 h, respectively, while the values for WA were 39.33 ± 1.84 and 90.01 ± 2.02 (%) during 2 and 24 h, respectively. According to the study, UF:P-CS adhesive can be used as a renewable and alternative solution for the manufacture of particleboard.

PO-28. Valorization of coffee silverskin using extraction cycles and water as a solvent: Design of process

Aziadé CHEMAT [1,*], Didier TOURAUD [2], Rainer MÜLLER [2], Werner KUNZ [2], and Anne-Sylvie FABIANO-TIXIER [1]

[1] INRAE, UMR 408, Avignon University, F-84000 Avignon, France.
[2] Institute of Physical and Theoretical Chemistry, University of Regensburg, 93040 Regensburg, Germany.

***Corresponding author:** Aziadé CHEMAT, aziade.chemat@gmail.com

Keywords: Coffee; silverskin; extraction; water; cycles; enrichment

Objectives

This study aims to valorize coffee silverskin, a by-product of the coffee industry, through the extraction of caffein a high value compound. This valorization consist on the establishing a water based process, using extraction cycles to maximize the concentration of the final product. The possible uses for the extractions products are discussed.

Methods

Two types of extraction cycles were carried out and compared: without water addition and with water compensation. Calculations were made to find the hypothetical number of cycles required to reach an optimum, for both type of cycles. The cycles were then performed and the results compared to hypothetical ones, in terms of caffein content.
A second extraction of the biomass was also done to evaluate the caffein content left inside of the fibers and if it's content raised.
A schematic process was then designed for the valorization of the by-product.

Results

The multi-cycle extraction with water compensation was preferred and its extraction cycles limited to 10-11, as beyond this amount, a plateau was reached and the caffein concentration was not evolving. The use of cycles, with the resulting product from a previous extraction as a solvent for fresh biomass, drove a significant rise in the content of caffeine, starting from 1.03 mg/mL for the first extraction to reach 4.25 mg/mL after 11^{th} extractions. The proposed process resulted in two products: one liquid aqueous extract, with a high caffeine concentration and one exhausted solid composed of caffeine-rich insoluble fibers.

PO-29. Valorization of silk by-products for lipopeptide surfactant production using green technologies.

Lara WEHBE [1,*], Rémi BASCOU [1], Erwann GUENIN [1], Alla NESTERENKO [1]

[1] Université de technologie de Compiègne, ESCOM, TIMR (Integrated Transformations of Renewable Matter), Centre de recherche Royallieu – CS 60319 – 60203 Compiègne Cedex.

***Corresponding author:** Lara WEHBE, lara.wehbe@utc.fr

Keywords: Silk biomass, protein extraction, lipopeptide, surfactant, mechanochemistry

Objectives

Silk is defined as non-food biomass produced by cultured silkworms. Global silk cocoon production is approximately 800,000 tons per year [1]. Silks are composed of two types of proteins, fibrous protein called fibroin and a glue-like protein called sericin [2]. Traditionally, the main application of silk is the textile industry due to the luster and mechanical properties of fibroin fibers. The main objective of this work is the valorization of silk proteins as a non-food biomass for the development of bio-based lipopeptide surfactants. Generally, biobased surfactants are derived from agricultural biomass such as sugars, starches, vegetable proteins, and oils. The use of silk protein by-products allows to preserve agricultural biomass for food applications and to propose this new family of lipopeptides for non-food applications, especially cosmetics and detergents.

Methods

The first step of silk lipopeptide production involves the separation of two silk protein fractions: fibroin and sericin by alkaline method and high temperature high pressure method (HTHP) respectively [3,4]. Then, fibroin and sericin peptides were obtained by enzymatic hydrolysis. Finally, the grafting of the hydrophobic chain onto the silk peptides was carried out using novel alternative approach of acylation by mechanochemistry as green technology in solventless conditions. Two acylation methods were investigated: pre-activation of fatty acid by 1,1-carbonyldiimidazole (CDI) and amidation of a fatty ester. The optimization of the mechanochemical syntheses was carried out using model amino acids (leucine, glycine and serine) mainly present in the structure of silk. The degree of hydrolysis and the acylation rate (ratio of grafted amine functions) were estimated by spectrophotometry. The adsorption properties of the peptides and lipopeptide mixtures with different structure was then studied by surface tension measurements using the pendant drop method. Lastly, the green aspect of these syntheses was confirmed by green metrics calculation such as E-factor and the atom economy [5].

Results

The obtained results show that the two silk protein fractions can be efficiently separated. Silk sericin and fibroin fractions were hydrolysed using Alcalase® enzyme with a degree of hydrolysis varying between 20 and 60%. Moreover, the results of functionnalization of silk peptides by mechanochemistry show very promising results with acylation rates varying from 50 to 70%. Silk lipopeptides with different fatty chains demonstrated a good surface activity. Fibroin peptides and lipopeptides show better surface activity than those of sericin because of the hydrophobic nature of fibroin protein. The results confirm that the structure of the hydrophilic and hydrophobic parts of lipopeptides have a key role in their surface adsorption properties. Green chemistry parameters confirmed the “eco-compatibility” of new mechanochemical synthesis approach.

To conclude, the acylation of silk peptides through pre-activation by CDI and amidation strategies under solventless conditions appears promising for the development of lipopeptide surfactants. Mechanosynthesis shows its efficiency in producing molecules of interest with good surface

adsorption properties. Silk-derived lipopeptides, produced by mechanochemistry, have a significant potential to replace petroleum-derived amphiphilic molecules, for cosmetic, detergent and pharmaceutical applications.

References

1 A. Popescu, Scientific Papers Series Management, 2018, 18, 385-400.
2 C. Vepari and D. L. Kaplan, Prog. Polym. Sci., 2007, 32, 991-1007.
3 D. N. Rockwood, R. C. Preda, T. Yücel, X. Wang, M. L. Lovett and D. L. Kaplan, Nature Protocols, 2011, 6, 1612-1631.
4 R. Bascou, J. Hardouin, M. A. Ben Mlouka, E. Guénin and A. Nesterenko, Materials Today Communications, 2022, 33, 104491.
5 S. Fadlallah, P. Sinha Roy, G. Garnier, K. Saito and F. Allais, Green Chemistry, 2021, 23, 1495-1535.

PO-30. Valorization of wine by-products for high added-value extracts.

Anna KARASTERGIOU [1], Anne-Laure GANCEL [1], Michael JOURDES [1], Pierre-Louis TEISSEDRE [1,*]

[1] Unité de recherche Œnologie, EA 4577, USC 1366 INRA, ISVV, Université de Bordeaux, F33882 Villenave d'Ornon, France

***Corresponding author:** Pierre-Louis TEISSEDRE, pierre-louis.teissedre@u-bordeaux.fr

Keywords: Grape pomace, polyphenols, valorization, sustainability, antioxidants

Objectives

Viticulture is a thriving industry, with grapevines (Vitis vinifera) representing one of the most significant fruit crops worldwide. Phenolic compounds, bioactive secondary metabolites abundant in grape stalks, skins, and seeds, have gained significant attention in recent decades due to their potential health benefits. Studying the phenolic composition and antioxidant capacity of various grape varieties is crucial for developing optimized extracts with nutraceutical and cosmetic applications. This study aims to characterize the phenolic compounds and evaluate the antioxidant effects of grapes and their corresponding grape pomace, from different varieties cultivated in southern France. Valorizing wine by-products presents a promising solution to meet the growing demand of the market for more natural and sustainable product alternatives.

Methods

The analyses included the determination of total polyphenol content (TPC) using the Folin-Ciocalteu assay and measuring total tannins and anthocyanins through spectrophotometric methods. Molecular profiling of condensed tannins and anthocyanins was performed using HPLC-UV/fluorimetry. Additionally, the characterization of various phenolic compounds - including phenolic acids, flavan-3-ols, flavonols, flavones, and stilbenes - was conducted by HPLC-UV-QqQ analysis. Antioxidant activities were assessed through ORAC, FRAP, ABTS, and DPPH assays.

Results

The findings revealed significant diversity in phenolic profiles and antioxidant potential among different grape varieties. Notably, grape pomace seeds and skins from red varieties such as Alicante and Syrah, as well as non-fermented grape pomace from white varieties, exhibited important antioxidant properties. Seeds generally showed higher TPC and tannin levels, along with greater radical scavenging capacities compared to skins. These results suggest that wine by-products can be effectively used to obtain high-added-value extracts, highlighting their potential as sustainable ingredients for the cosmetics industry and promoting circular economy principles.

PO-31. The protective role of exogenous proline in alleviating oxidative stress induced by heavy metals in sour orange plants (*Citrus aurantium* L.)

Anastasia GIANNAKOULA [1] and Anna KARASTERGIOU [1]

[1] Department of Agriculture, International Hellenic University (IHU), 57400 Sindos, Greece

Corresponding author: Anastasia GIANNAKOULA, agianna@ihu.gr

Keywords: Heavy metal stress, malondialdehyde (MDA), antioxidant compounds, oxidative stress, proline

Objectives

In recent decades, heavy metal stress has emerged as a significant abiotic factor contributing to orchard contamination. High levels of lead (Pb) and copper (Cu) in the soil result from their frequent use as fungicides and pesticides. Additionally, food contamination has persisted due to over 50 years of insecticide applications such as lead arsenate and copper sulfate. Sour orange (Citrus aurantium L.) is known for its medicinal properties, attributed to its bioactive compounds like phenolics, flavonoids, and essential oils.

Reactive oxygen species (ROS) production can cause severe toxicity symptoms in plants under heavy metal stress. Proline, a common compatible osmolyte, plays a crucial role in antioxidant activity and enhancing abiotic stress tolerance. This multifunctional amino acid is also effective in mitigating adverse effects caused by heavy metal toxicity. This study examines the impact of proline on growth and biochemical characteristics in sour orange plants exposed to lead and copper-contaminated soils.

Methods

Citrus plants were cultivated in a greenhouse with varying concentrations of copper and lead (500, 800 M), incl ding a combination of C +Pb (500, 800 M), and e ogeno s proline treatment (20mM). The volatile constituents of the essential oils from the peel of sour oranges grown in Greece were analyzed using GC-MS. Furthermore, the photosynthetic machinary was estimated by calculating the assimilation rate of CO_2 the transpiration rate of CO_2 and the water use efficiency using a LICOR-6400. portable photosynthedis system.

Results

Morphological characteristics such as height showed notable decreases due to photosynthetic disturbances especially at simultaneously high levels of Cu+Pb (800μM). In addition $CuSO_4$ treatment at high levels (800μM) with simultaneously high $Pb(NO_3)_2$ concentrations caused significant increased, in lipid peroxidation (MDA), proline and hydrogen peroxide (H_2O_2) content, while the antioxidant activity was increased. Proline treatment generally increased tolerance to copper and lead, and enhanced the accumulation of phenols and soluble sugars compared to untreated plants. These findings suggest variations in antioxidant responses to oxidative stress induced by copper and lead, potentially linked to the application of exogenous proline. This treatment appears to elevate antioxidants, thereby protecting membrane functions from ROS-induced damage in citrus plants.

PO-32. Valorization of forced chicory roots: extraction and thermochemical conversion

Lynn RHAYEM [1,*], Ammar BENSAKHRIA [1], Nabil GRIMI [1]

[1] Université de Technologie de Compiègne, Laboratoire Transformations Intégrées de la Matière Renouvelable, UTC/ESCOM, 7TIMR, Centre de Recherche de Royallieu, BP 20529-60205 Compiègne Cedex, France.

***Corresponding author:** Lynn RHAYEM, lynn.rhayem@etu.utc.fr

Keywords: Forced chicory roots, extraction, antioxidant activity, pyrolysis, physical activation, activated carbon, surface area, micropores, adsorption capacity, optimization

The production of endive crops generates large quantities of forced roots as a by-product. According to Eurostat (2020), each year in the European Union, approximately 300,000 to 400,000 tons of forced roots are produced from endive cultivation, commonly used as compost or animal feed. These forced roots represent an interesting raw material for the concept of biorefinery, as they are available year-round and have a chemical composition rich in sugars, dietary fibers, and bioactive compounds such as phenolic compounds and sesquiterpene lactones [1]. From an economic, environmental, and social perspective, the valorization of these by-products is necessary to develop a sustainable circular economy. The main objective of this study is to valorize forced endive roots through extraction of bioactive compounds and thermochemical conversion. The work focused on extracting polyphenols from these forced endive roots using various techniques like maceration and pressing. The extraction process was enhanced through the application of innovative pre-treatment technologies such as pulsed electric fields and ultrasound. The root residues were then subjected to a thermochemical conversion process, resulting in the production of activated carbon. Several treatment conditions were studied and interpreted to optimize the production of activated carbon and enhance its efficiency in applications such as water treatment.

The forced endive roots were provided by APEF-Arras (Association des Producteurs d'Endive de France). They were washed with cold tap water, then cut into cubes. For some experiments, they were dried at 105°C for 7 hours. The preparation of biochar from the forced chicory roots was carried out during 1 hour of thermal treatment in a muffle furnace (VECSTAR Ltd. Model MRF2) at 600°C with a heating rate of 50°C/min. Then, the obtained biochar was ground for its physical activation with carbon dioxide (CO_2). The variable parameter was the activation temperature (750°C, 850°C, and 950°C), and the fixed parameters were the CO_2 flow rate (60 NL/h) and the duration at the temperature plateau (1 hour). After cooling, the obtained activated carbon was collected, then analyzed to evaluate its physical properties and its adsorption capacity. The extraction was initially carried out by maceration at different temperatures (20°C, 50°C, 70°C, and 80°C) for one hour. The diffusion coefficient was calculated, and the total polyphenol content of the extract was determined using the Folin–Ciocalteu method [2]. Gallic acid (Sigma-Aldrich) was used for the calibration curve.

It was demonstrated that both the amount of extracted polyphenols and the diffusion coefficient increased with rising temperatures, reaching values of 50.76 mg gallic acid equivalent/L and 2×10^{-10} m^2/s at an extraction temperature of 80°C, respectively. It has been shown that the activation yield is the best in the case of dried endives (71.46%), which resulted in minimal mass loss. The study of the adsorption isotherms of methylene blue on the produced activated carbons indicated that the Langmuir model perfectly described the equilibrium adsorption process. The adsorption reaction kinetics of methylene blue are described by the pseudo-second order kinetic model. The activated carbon obtained from dried forced endive roots and activated at 850°C, showed the best adsorption capacity, resulting in the highest maximum adsorption capacity (Q0 = 323 mg/g), with a significant BET surface area (SBET = 190.645 m^2/g), compared to the other produced activated carbons.

References

[1] A. Twarogowska, C. Van Poucke, and B. Van Droogenbroeck, “Upcycling of Belgian endive (Cichorium intybus var. foliosum) by-products. Chemical composition and functional properties of dietary fibre root powders,” Food Chem., vol. 332, p. 127444, Dec. 2020, doi: 10.1016/j.foodchem.2020.127444.
[2] H. N. Rajha, N. Boussetta, N. Louka, R. G. Maroun, and E. Vorobiev, “A comparative study of physical pretreatments for the extraction of polyphenols and proteins from vine shoots,” Food Res. Int., vol. 65, pp. 462–468, Nov. 2014, doi: 10.1016/j.foodres.2014.04.024.

PO-33. Influence of ohmic heating in the bioactive compounds of extracts from Iranian brown macroalgae (*Nizimuddinia zanardini*)

Atefe TAHERKHANI [1], Akram SHARIFI [1,*], Mohamed KOUBAA [2], Mehrdad NIAKOUSARI [3], Javad HASANPOUR [4]

[1] Department of Food Science and Technology, Qazvin Branch, Islamic Azad University, Qazvin, Iran.
[2] Université de technologie de Compiègne, ESCOM, TIMR (Integrated Transformations of Renewable Matter), Centre de recherche Royallieu – CS 60319 – 60203 Compiègne Cedex, France.
[3] Food Science and Technology Department, Shiraz University, Shiraz, Iran.
[4] Department of Mechanical Engineering, Qazvin Branch, Islamic Azad University, Qazvin, Iran.

***Corresponding author:** Akram SHARIFI, asharifi@qiau.ac.ir

Keywords: *Nizimuddinia zanardini*, Antioxidant activity, Ohmic.

Objectives

Due to sustainability concerns, electric field-based technologies for extraction are becoming increasingly important. In addition marine algae are becoming an interesting source of biologically active compounds with a promising application as functional food ingredients. *Nizimuddinia zanardini* is one of the brown algae distributed in Oman Sea, and there is no information regarding the chemical composition of this alga with ohmic extraction has been reported. The objective of this work was to evaluate the feasibility of using ohmic extraction in the selective extraction of the Total Flavonoid Content (TFC), Total Phenolic Content (TPC), Total Phlorotannin Content (TPhC), as well as antioxidant capacity (DPPH) and yield extraction from *Nizimuddinia zanardini*.

Methods

The brown algae *Nizimuddinia zanardini* was collected from Oman Gulf (the Chabahar region). *N. zanardini* was extracted in ohmic system with ethanol 50% at two times (1 and 2 hours) and two temperatures (45 and 60 °C). DPPH free radical scavenging activity method was used to evaluate its antioxidant properties. TPC, TFC, TPhC were measured according to the standard methods. The yield was evaluated by solid weighting in the extract. The influences of extraction time and temperature on the phytochemicals and yield were analyzed by the multivariate general linear model in SPSS version 25.0. The differences were further analyzed by Tukey's HSD post hoc tests.

Results

Overall, significant differences in the extraction of each compounds between different conditions were observed. The TPC, TFC, TPhC, and yield values of *N. zanardinii* were in the range from 960.7 ± 6.74 to 778.19 ± 1.56 mg GAE/g dry algae (the highest one at 45°C for 120 min), from 239.19 ± 1.78 to 123.76 ± 4.84 mg QE/g dry algae (the highest one at 60°C for 120 min), from 1016.17 ± 7.59 to 810.83 ± 1.76 mg PGE/g dry algae (the highest one at 45°C for 120 min), and from 33.21 ± 0.54 to 28.32 ± 0.57 DW% (the highest one at 45°C for 120 min) respectively. In the case of the antioxidant activities, the highest DPPH scavenging activities (79.56 ± 0.08%) were achieved at 60°C for 120 min.

PO-34. Evaluation of physical properties of Ajwain (*Trachyspermum Copticum* (L.)) extract microcapsules prepared by Freeze Drying

Armineh ROSHANPAZHOH [1], Akram SHARIFI [1,*], Mohamed KOUBAA [2], Mehrdad NIAKOUSARI [3]

[1] Department of Food Science and Technology, Qazvin Branch, Islamic Azad University, Qazvin, Iran.
[2] Université de technologie de Compiègne, ESCOM, TIMR (Integrated Transformations of Renewable Matter), Centre de recherche Royallieu – CS 60319 – 60203 Compiègne Cedex, France.
[3] Food Science and Technology Department, Shiraz University, Shiraz, Iran

***Corresponding author:** Akram SHARIFI, asharifi@qiau.ac.ir

Keywords: Ajwain, Microencapsulation, maltodextrin, gum Arabic, Freeze-drying.

Objectives
Ajwain is a valuable herb containing abundant polyphenols with powerful antioxidant properties that contribute significantly to health and medicinal benefits. In this study, microencapsulation by freeze-drying was investigated for the production of powdered Ajwain extracts with high stability of bioactive compounds.

Methods
The grinded Ajwain was extracted through a simple maceration method using distilled water in order to achieve the highest Brix. Then, Ajwain extract was mixed separately with maltodextrin (M), gum Arabic (G) and their mixture in 1:1 w/w ratio (M-G) as the coating materials at a concentration of 30% based on dry matter and homogenized with magnetic stirrer. The samples were dried for 24 h in a freeze dryer. The effects of different carriers on the physical properties of the powders were assessed.

Results
The results showed that by using (G), the hygroscopicity of the microcapsules increased significantly. The change process of tapped density was similar to bulk density and the highest value was observed for (M). All the samples were characterized by low flowability and high cohesiveness with Carr index ranging between 28.38–36.41 and Hausner ratio between 1.41–1.57. The highest L* index was significantly related to the sample (G) and b* index of (M) was substantially upper than (G). The results showed Ajwain extract microcapsules had appropriate characteristics and thus suggesting that it might be useful as a food or medicine additive and/or ingredient.

PO-35. Stibenoids eco-extraction from vineyard coproducts: vitisin B extraction by ASE compare to sc-CO_2

Maryam ELSHAZELY [1,2,3], Tasnim HEGAZY [1,2,3], Christelle HARSCOAT-SCHIAVO [1], Omar-SHAABAN-OMAR AHMED [2,3], Pierre WAFFO-TEGUO [2,3] and Raphaëlle SAVOIRE [1,*]

[1] Univ. Bordeaux, CNRS, Bordeaux INP, CBMN, UMR 5248, F-33600 Pessac, France.
[2] Univ. Bordeaux, Bordeaux INP, INRAE, OENO, UMR 1366, ISVV, F-33140 Villena e d Ornon, France.
[3] Bordeaux Sciences Agro, F-33170 Gradignan, France.

***Corresponding author:** Raphaëlle SAVOIRE, raphaelle.savoire@ensmac.fr

Keywords: Supercritical CO_2 Extraction / Accelerated solvent extraction (ASE) / Vitisin B / stilbenoids

The vineyard industry is a major economic sector in the Bordeaux region, which is looking for new ways to valorize its by-products. Stilbenoids a class of non-flavonoid polyphenols are a group of bioactive molecules found in plants and particularly abundant in grapevines, offer promising opportunities. This group includes the well- known resveratrol and its oligomers such as E-vitisin B (a resveratrol tetramer). E-Vitisin B also called r-viniferin has shown significant antimycotoxin activity, making its extraction highly valuable. Previous studies have identified grapevine roots as a rich source of E-vitisin B highlighting the potential to transform vine co-products into new plant-derived bioactives.

Objective

The aim of this work is to evaluate different eco-extraction methods for recovering vitisin B from grapevine roots. The processes will be compared in terms of vitisin B yield and selectivity.

Methods

Supercritical fluid extraction (SFE) was performed with homemade setups on 4 and 30 g of ground vine roots. Effects of temperature, pressure, co-solvent composition and ratio with sc-CO2 were studied. Accelerated solvent extraction (ASE) was performed on Dionex ASE 350 system (Sunnyvale, California) at 100 bar on 5 g of plant powder mixed with sand. Number of cycles, solvent composition (EtOH / water), contact time and temperature were varied. E-Vitisin B was quantified through HPLC-DAD method.

Results

A full factorial 33 design of experiments conducted on ASE coupled with an optimization leads to a maximal yield of 22.3 g vitisin B/ kg dried roots. The major factor affecting the yield was the composition of the solvent. For SFE, a Box Behnken design of experiment leads to a maximal yield of 12.5 g/kg. The pressure was the main factor affecting the extraction. Selectivity of extraction for SFE was mainly determined by the co-solvent composition. So, both processes allowed for E-vitisin B extraction with quantities compatible with a high added-value use. Activity tests will be conducted to check the bioactivity of the extracts and the influence of the non-E-vitisin B compounds co-extracted.

PO-36. Investigating a straightforward process coupling extraction and membrane separation for hydroxytyrosol recovery from "picholine marocaine" *cv*. olive pomace

Hamza DHAIDHI [1,2,3], Adrien SERVENT [2], Ajal EL AMINE [4], Said ENNAHLI [1], Aadil BAJOUB [1,5,*], Alegría CARRASCO-PANCORBO [5], Hanaa ABDELMOUMEN [3], Manuel DORNIER [2,*]

[1] Laboratory of Food and Food By-Products Chemistry and Processing Technology, National School of Agriculture in Meknès, Km 10, Haj Kaddour Road, B.P. S/40, Meknès, Morocco.
[2] QualiSud, Univ Montpellier, Avignon Univ, CIRAD, Institut Agro, IRD, Univ de La Réunion, Montpellier, France.
[3] Microbiology and Molecular Biology Team, Center of Plant and Microbial Biotechnology, Biodiversity and Environment, Faculty of Sciences, Mohammed V University in Rabat, Morocco.
[4] UPR of Pharmacognosy, Faculty of Medicine and Pharmacy of Rabat, BP 6203, Mohammed V University, Rabat, Morocco.
[5] Department of Analytical Chemistry, Faculty of Science, University of Granada, Ave. Fuentenueva s/n, 18071, Granada, Spain.

***Corresponding authors:** Dornier Manuel, manuel.dornier@institut-agro.fr and Bajoub Aadil, bajoub.aadil@gmail.com

Keywords: Olive pomace, "Picholine Marocaine" *Cv*., membrane separation, hydroxytyrosol

Objectives

The olive oil agri-food sector is a key pillar of Morocco's economy, yet its environmental impact threatens its long-term sustainability. This research aims to advance the transition of the Moroccan olive oil industry towards a circular bioeconomy by implementing a zero-waste strategy that minimizes environmental harm and adds value. This study focused on the recovery of hydroxytyrosol (OHT), a well-known bioactive compound, from olive pomace by coupling a simple extraction with membrane separation process.

Methods

A 30 kg sample of two-phase olive pomace from Morocco was extracted at 30°C through a 4-step extraction scheme to produce the OHT extract. First, the pomace was hydraulically pressed at 100 bar. The remaining solid residue was then mixed with 10% ethanol and subjected to a second pressing cycle. The combined extracts were clarified using crossflow microfiltration with tubular ceramic membranes (0.2 µm average pore diameter) at 2-3 bar of transmembrane pressure, preceded by a pectinase treatment to improve filterability. Finally, a nanofiltration with flat organic membranes was performed to concentrate the OHT, by comparing 2 membranes (200-300 Da MWCO) at transmembrane pressures from 10 to 25 bar based on permeate flux and solute retentions (OHT, dry matter, total phenolics).

Results

A recovery rate of 90% for OHT was obtained after the pressing of the olive pomace with the hydroalcoholic solution. The extract was completely clarified by microfiltration. This step allowed to remove suspended particles without affecting the OHT content and with a permeate flux above 40 $kg.h^{-1}.m^{-2}$. The subsequent nanofiltration showed that the OHT retention varied from 20 to 62% and the permeate flux from 2 to 35 kg·h-1·m-2 depending on the membrane/pressure combination used. Some combinations were more favorable for OHT concentration. Others might be interesting for the OHT purification in the permeate due to the difference in retention with dry matter and total phenolics.

PO-37. Optimization of bioactive compound extraction from rosemary solid residues using Box-Behnken design

Anissa BARAR [1,*], Ouahida BENSEBIA [1]

[1] Industrial Process Engineering Sciences Laboratory (LSGPI), FGMGP-USTHB, B.P. 32, El Alia, Bab Ezzouar, 16111, Algiers, Algeria.

***Corresponding author:** Anissa BARAR, anissabarar@yahoo.fr

Keywords: Extraction, Rosemary solid residues, Bioactive compounds, Box- Behnken Design

Objectives

This study aims to valorize the solid residues produced by the essential oils industry. The solid residues of rosemary leaves were extracted using food-grade ethanol. The optimal conditions for recovering phenolic compounds and antioxidant activity were determined using the Box-Behnken design methodology.

Methods

A three-level Box Behnken design (BBD) was applied to optimize the extraction parameters, using drying temperature of residues leaves (30-60°C), ethanol/water concentration (40-80 °C) and solvent/solid ratio (10-20 mL/g) as factors. The total phenolic content (TPC) and flavonoid content (TFC) were determined using the Folin-Ciocalteu and aluminum chloride methods, respectively. Antioxidant activity was measured using the DPPH radical scavenging assay.

Results

The optimal extraction conditions, as predicted by the Box-Behnken design (BBD) methodology, varied depending on the target objective. The highest extraction yield for total polyphenols content (66.17 mg EAG/g) and total flavonoid content (4.15 mg EQ/g) was achieved with residues dried at 60°C, using 40% ethanol for TPC and 60% ethanol for TFC, with a solvent-solid ratio of 15 mL/g for TPC and 20 mL/g for TFC. The best IC50 value of 0.79 mg/mL was obtained from residues dried at 30°C, with a solvent-solid ratio of 15 mL/g and 40% ethanol concentration. This study indicates that rosemary residues are a potential source of bioactive compounds.

PO-38. Optimization of bioactive compounds extraction assisted by non-ionic surfactant from *Tetraclinis articulata* solid residues using response surface methodology

Anissa BARAR [1,*], Sofiane LATRECHE [2], Ismail BENCHERIFA [3], Ouahida BENSEBIA [1]

[1] Industrial Process Engineering Sciences Laboratory (LSGPI), FGMGP-USTHB, B.P. 32, El Alia, Bab Ezzouar, 16111, Algiers, Algeria.
[2] Materials Technology Laboratory (LTM), FGMGP-USTHB, B.P. 32, El Alia, Bab Ezzouar, 16111, Algiers, Algeria
[3] Department of Mechanical Engineering, University of Biskra, B.P.145, 07000 Biskra, Algeria

***Corresponding author:** Anissa BARAR, anissabarar@yahoo.fr

Keywords: *Tetraclinis articulata* solid residues, nonionic surfactant tween 80, maceration assisted extraction, Box-Behnken design, bioactive compounds.

Objectives

A green and promising approach using the maceration technique assisted by a nonionic surfactant was developed for the extraction of bioactive phenolic compounds from Tetraclinis articulata solid residues. The total polyphenolic and flavonoid contents have been optimized using response surface methodology.

Methods

Box Behnken design was used to optimize the extraction conditions including concentration of surfactant Tween-80 (0-3%) liquid-to-solid ratio (10:1-30:1 mL/g), maceration time (120-360 minutes), ethanol concentration (40-80%). The total phenolic content (TPC) and flavonoid content (TFC) were determined using the Folin-Ciocalteu and Aluminum chloride methods, respectively. Antioxidant activity was estimated using the DPPH radical scavenging assay, with ascorbic acid as the control.

Results

The optimum extraction conditions were selected as follows: a concentration of Tween-80 of 3 %, an ethanol concentration of 40%, a liquid-to-solid ratio of 20 ml and a maceration time of 240 min, with an IC50 of 0.39 (mg/ mL). Under these conditions, the highest total polyphenol content (140 mg EAG/ g) was recorded. Moreover, a concentration of Tween-80 of 1,5 %, an ethanol concentration of 60%, a liquid-to-solid ratio of 20 ml and a maceration time of 360 min were determined for the highest total flavonoid content (63.43 mg EQ/ g). The assisted extraction method using nonionic surfactant allows to reach a higher amount in bioactive compounds from thuya solid residues compared with conventional extraction.

PO-39. High voltage electrical discharges as a pre-treatment for extraction of bioactive compounds from solid residues of thuya

Anissa BARAR [1,*], Nadia BOUSSETTA [2], Nabil GRIMI [2], Ouahida BENSEBIA [1]

[1] Industrial Process Engineering Sciences Laboratory (LSGPI), FGMGP-USTHB, B.P. 32, El Alia, Bab Ezzouar, 16111, Algiers, Algeria.
[2] Integrated Transformations of Renewable Matter (TIMR), Université de Technologie de Compiègne, ESCOM, Centre de Recherche Royallieu, Cedex, Compiègne, CS 60319-60203, France.

***Corresponding author:** Anissa BARAR, anissabarar@yahoo.fr

Keywords: Thuya solid residues, high voltage electrical discharge, Box-Behnken design, bioactive compounds, antimicrobial activity.

Objectives

This study aimed to assess the impact of high voltage electrical discharges (HVED) pretreatment on the extraction of bioactive compounds from Thuya (Tetraclinis articulata) by-products. To identify the optimal extraction conditions for phenolic compounds and antioxidant activity, the response surface methodology with a Box-Behnken design was utilized. Additionally, the antimicrobial activity of the thuya solid residue extract under these optimal conditions was evaluated.

Methods

Thuya solid residues were placed in a treatment chamber connected to an HVED generator with a high voltage pulsed power of 40 kV-10 kA. The pulses had a frequency of 1 Hz, with a 2-second interval between each pulse. The effects of extraction time (120-360 minutes), liquid-to-solid ratio (10:1-30:1 mL/g), and ethanol concentration (40-80%) were investigated. The total phenolic content (TPC) and flavonoid content (TFC) were determined using the Folin-Ciocalteu and aluminum chloride methods, respectively. Antioxidant activity was measured using the DPPH radical scavenging assay, with ascorbic acid as the control. The antimicrobial activity of the thuya solid residue extract was evaluated using the disk diffusion method.

Results

The results indicated that the optimal conditions for achieving the highest extraction yield (93%), total polyphenols content (104 mg EAG/g), total flavonoid content (37.91 mg EQ/g), and the best IC50 value (0.48 mg/mL) are liquid to solvent ratio of 20 mL/g, an extraction time of 230.51 minutes, and a 60% ethanol solution. The application of HVED pretreatment enhanced the extraction of bioactive compounds by 5.4% compared to the control sample. The extract demonstrated effectiveness against Listeria monocytogenes, with a large inhibition zone (21 mm) and a minimum inhibitory concentration (MIC) of 100 µg/mL. The application of HVED pretreatment allowed to enhance the extraction of bioactive compounds by 5,4% comparing to a control sample. The results obtained in the present study showed the potential of HVED pretreatment to improve the recovery of high-added value compounds from solid residues of thuya.

PO-40. Valorization of sesame seed coat waste: phenolic composition, antibacterial efficacy, and nanoemulsion encapsulation for food preservation

Salma KHAZAAL [1], Mahmoud KHALIL [1,2], Tareq M. OSAILI [3,4], Borhan ALBISS [5], Anas AL-NABULSI [4], Nicolas LOUKA [6], Nada EL DARRA [7,*]

[1] Department of Biological Sciences, Faculty of Sciences, Beirut Arab University, Beirut, Lebanon
[2] Molecular Biology Unit, Department of Zoology, Faculty of Science, Alexandria University, Alexandria, Egypt
[3] Department of Clinical Nutrition and Dietetics, College of Health Sciences, University of Sharjah, P.O. Box 27272, Sharjah, United Arab Emirates
[4] Department of Nutrition and Food Technology, Faculty of Agriculture, Jordan University of Science and Technology, P.O. Box 3030, Irbid, 22110, Jordan
[5] Nanotechnology Institute, Jordan University of Science and Technology, Irbid, Jordan
[6] Centre d'Analyses et de Recherche, Unité de Recherche Technologies et Valorisation Agro-alimentaire, Faculté des Sciences, Université Saint-Joseph, B.P. 17-5208 Riad El Solh, Beirut 1104 2020, Lebanon.
[7] Department of Nutrition & Dietetics, Faculty of Health Sciences, Beirut Arab University, Tarik El Jedidah – Beirut, P.O. Box: 115020, Riad EL Solh, 1107 2809, Lebanon.

***Corresponding author:** Nada El Darra, n.aldarra@bau.edu.lb

Keywords: sesame seed coat, polyphenols, antibacterial activity, nanoemulsion .

Objectives

Sesame (*Sesamum indicum* L.) seed coat, which is obtained during the production of sesame paste, is generally discarded or used for animal feed. However, it emerges as a significant industrial waste, presenting an opportunity for effective valorization through the extraction of bioactive compounds. The current study examined the phenolic composition and antioxidant activity of sesame seed coat (SSC). Besides, the antibacterial effects of SSC against common foodborne pathogens including Listeria monocytogenes, Escherichia coli O157:H7, and Salmonella Typhimurium were assessed.

Methods

The experiments were carried out in triplicate in vitro at 37 °C for 24 hours (hrs). Additionally, SSC was subjected to nanoemulsion coating, characterized by Dynamic Light Scattering (DLS) and Scanning Electron Microscopy (SEM). The antibacterial efficacy of both SSC coat and the resultant nanoemulsion was evaluated for potential incorporation as a natural preservative in sterilized milk, serving as a food model stored at 4/10 °C for 7 days.

Results

Key findings showed that SSC exhibits a total phenolic content of 22.91 mg GAE/g DM and an antioxidant activity of 83.53% at 100 mg/ml. High-Performance Liquid Chromatography (HPLC) analysis revealed five different types of polyphenols, with catechin (2.41 ppm) being the predominant compound. The Gas Chromatography-Mass Spectrometry (GC-MS) analysis showed seven compounds, with oleic acid (64.73%) as the major constituent. SSC showed significant inhibition against L. monocytogenes in broth, with no viable bacteria at 100 mg/ml of sesame seed coat at 37 °C/24 hrs. The utilization of Atomic Force Microscopy (AFM) for imaging bacteria indicated that the application of both the SSC and nanoemulsion treatments resulted in alterations to the morphology of the bacteria, such as the creation of contorted shapes, the emergence of surface blebs and indentations, as well as changes to the stiffness and size of the bacteria. SSC proves effective in reducing bacterial

counts of *L. monocytogenes* and *E. coli* O157:H7, achieving reductions ranging from 0.9 to 4.5 log CFU/ml on day 7 in milk stored at 4 and 10 °C, respectively. Conversely, the sesame seed coat nanoemulsion (10%) significantly reduces bacterial counts of L. monocytogenes and E. coli O157:H7, achieving reductions between 1.81 and 4.73 log CFU/ml at the end of the incubation period at 4 and 10 °C, respectively (p-value<0.05).

Conclusion

The findings of the study indicate that SSC has the potential to act as an effective antibacterial agent and food preservative, making it a promising by-product. These results could have significant implications for the food industry and contribute to enhancing food safety.

PO-41. Removal of contaminants from water using tomato leaves as adsorbent

Layan HELMI [1], Maria ALAYLI [2], Maram AL BAKRI [2], Akram HIJAZI [3], Nada EL DARRA [2,*]

[1] Department of Biological Sciences, Faculty of Science, Beirut Arab University, Beirut P.O. Box 11-5020, Lebanon.
[2] Department of Nutrition & Dietetics, Faculty of Health Sciences, Beirut Arab University, Tarik El Jedidah, Riad El Solh, P. O. Box 115020, Beirut 1107 2809, Lebanon
[3] Plateforme de Recherche et d'Analyse en Sciences de l'Environnement (EDST-PRASE), Beirut P.O. Box 657314, Lebanon

***Corresponding author:** Nada EL DARRA, n.aldarra@bau.edu.lb

Keywords: Water contamination, industrial wastewater, adsorption effectiveness, heavy metals, wastewater treatment, sustainable purification methods, tomato leaves.

Objectives

Water, an essential component for life and agriculture, is increasingly threatened by contamination. Industrial wastewater, a major source of this issue, continues to negatively impact ecosystems and human health. This wastewater contains various contaminants such as dyes, heavy metals, pharmaceuticals, and pesticides, underscoring the urgent need for effective water purification methods. The exploration of food by-products as adsorbents for such contaminants is being widely explored. This study investigates the valorization of tomato by-products, specifically the leaves, as a potential adsorbent for these pollutants.

Methods

To prepare the test solutions, 100mL of distilled water was spiked with a known concentration of the contaminant, according to the required test. A known weight of the tomato leaves powder were placed with the solution to test the efficiency of adsorption. They were placed in a shaker at 120 rpm for two hours, except when different times were tested. All tests were performed at room temperature. After the required time finished, the solution was filtered using a filter paper in the case of dyes and a syringe filter in the case of lead. After filtration, the absorbance of the dye solutions were read using a UV-Vis Spectrophotometer to calculate the concentration, where a calibration curve was prepared for each dye. For the lead solution, an atomic absorption spectrometry was used to identify the concentration of the heavy metal.

Results

Tomato leaves demonstrated significant adsorption capabilities for dyes like methylene blue, malachite green, and crystal violet, with removal rates of 90.65% ± 0.3, 93.63% ± 0.5, and 89.75% ± 0.6 respectively at an initial dye concentration of 20 ppm. High adsorption effectiveness was also observed across various dye concentrations (5-25 ppm) and contact times (5-120 minutes). Additionally, for heavy metals such as lead, tomato leaves exhibited notable removal efficiency, reaching 78.42% ± 0.2 removal at 20 ppm, with 57.14% ± 0.4 removed within the first 5 minutes.

Conclusion

These findings highlight tomato leaf as a promising adsorbent for water purification, showcasing its potential for environmental remediation and water quality preservation.

GENP2024 : Green Extraction of Natural Products

ISBN : 978-2-913923-39-3

EAN : 9782913923393