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TECNOLOGIA DEL FARMACO
UNIVERSITÀ DEGLI STUDI DI TORINO

GENP2016

GREEN EXTRACTION OF NATURAL PRODUCTS

II EDITION - TURIN, 31 MAY - 1 JUNE 2016

Website: http://e20.unito.it/genp2016/

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GENP2016 - Green Extraction of Natural Products

II Edition

Book of Abstracts

Turin, Italy

31st May - 1st June 2016

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Welcome

Dear Colleagues, Friends and Students,

Welcome to **GENP 2016**!

This is a unique occasion to bring all the main players and experts in the Green Extraction of Natural Products field together under one roof. The near equal numbers of participants from Industry and Academia should create the best conditions under which to bridge the gap between the two worlds. The high level and multidisciplinary nature of the communications at the conference will shed light on fresh potential as we discuss the efficiency, process intensification, scale up potential, investment costs and environmental impact of new technologies. We aim to work at the interfaces between several research, practice and production disciplines while hearing the viewpoints of chemists, engineers, biologists and biotechnologists. One conference to unite all minds, one conference to find them, one conference to bring them all and in the interface bind them!

GENP 2016 will build on the experience shared and context created in Avignon during the first Edition, which was chaired by Prof. Farid Chemat.

The outstanding speakers at GENP 2016 will be the highlights of an unforgettable conference. New extraction and purification technologies, green solvents, process engineering, biomass valorisation, sustainable protocols and the circular economy are just some of the themes due to be explored.

The conference is located at the Cavallerizza Reale in the heart of Turin, Italy's first Capital. The site evokes the atmosphere of a magic city that has evolved as a stratification of cultures, people and civilisations. The city is the subject of numerous testimonials to a past that stretches back over 2000 years. The original settlement, Taurasia, was populated with the descendants of Gauls and Celtic-Ligures and became a military citadel under the ancient Romans. Augustus then gave it the name of Augusta Taurinorum, in 28 B.C.. The University, founded in 1404, attracted brilliant minds from all over Europe; Erasmus of Rotterdam, one of the geniuses of Renaissance Humanism, graduated here. Torino was also much loved by Montesquieu as well as by French politician and intellectual Charles de Brosses, who once defined it as "*the loveliest city in Italy and, as far as I'm concerned, of Europe*".

Dear participants, enjoy the Conference and the city. Thanks indeed for coming!

On behalf of the organizing committee

Giancarlo Cravotto

GENP 2016 Conference Chair

Program

May 31st

8.00 **Registration and welcome**

9.30 **Opening Ceremony and meeting presentation**

CHAIR: CRAVOTTO G. and MARTINA K.

10.00 **PL1** - Farid CHEMAT (University of Avignon, France)

"Green extraction of natural products. Past and present panacea"

10.40 **PL2** - Andrew P. ABBOTT (University of Leicester, UK)

"Deep eutectic solvents as novel media for natural product extraction"

11.20 **Coffee Break**

CHAIR: BILY A. and CHRISMAN R.

11.50 **K1** - Diels LUDO (Antwerp University - VITO, Belgium)

"Functionalized bioaromatics from lignin"

12.20 **O1** - Daniele NAVIGLIO (University of Naples Federico II, Italy)

"The extractor Naviglio in food and beverage productions"

12.40 **O2** - Francesco DONSÍ (University of Salerno, Italy)

"Sustainable valorization of agri-food residues by high pressure homogenization processing to unlock high-added value intracellular components"

13.00 **O3** - Gianpiero PATARO (University of Salerno, Italy)

"PEF-assisted green solvent extraction of high-added value compounds from agri-food by-products"

13.20 **K2** - Christof KERSCH (NATECCO_2, Germany)

"Phytoextractions using supercritical CO_2"

13.50 **LUNCH**

CHAIR: CHEMAT F. and STRUBE J.

15.00 **PL3** - Amin CHAANIN (VitaPlant AG, Switzerland)

"The controlled production of plant raw material for the phyto industry"

15.40 **O4** - Bahar ALIAKBARIAN (University of Genoa, Italy)

"Extraction of antioxidants from spent coffee grounds using microwave-assisted and high pressure and temperature extractions"

16.00 **O5** - Peggy VAUCHEL (University of Lille 1, France)

"Valorization of chicory by-product by ultrasound-assisted extraction of antioxidant polyphenols: extraction yield and energy consumption optimization"

16.20 **O6**- Edinson YARA-VARÓN (University of Avignon, France)

"Extraction of bioactive compounds using a green solvent from forestry biomass"

16.40 **O7** - Nadia MULINACCI (University of Florence, Italy)

"Recovery and fractionation of phenolic compounds from rosemary leaves by US and MW-assisted extractions"

17.00 **O8** - Alexandra NĚSIĆ (University of Belgrade, Serbia)

"Microwave-enhanced extraction of Alginate from Sargassum seaweeds: from coastal beach wastes to raw film for agricultural applications"

17.20 **O9** - Sivakumar MANICKAM (University of Nottingham, Malaysia)

"Extraction of antioxidant polysaccharides from *G. lucidum* using ultrasound: scale-up potential"

17.40 **SG1** - Sahar Ben YOUSSEF (University of Sfax, Tunisia) "Green extraction procedures of lipids from seeds of different Tunisian date palm verities"

SG2 - Paolo CIRILLO (RI-LAVO S.r.l., Itay) "New Ultrasound applications"

18.00 **1st POSTER SESSION**

June 1st

8.40 **University and Local Authorities welcome**

CHAIR: MANICKAM S. and ABBOTT A.

9.00 **PL4** – Benjamin LIGHTBURN (Mazza Innovation Ltd., Canada)

"An Introduction to the green, clean and economical Mazza extraction technology"

9.40 **PL5** - Pablo JULIANO (CSIRO Food and Nutrition, Australia)

"Advances in megasonic processing to enhance recovery of quality oils"

10.20 **O10** - Vincent RAPINEL (University of Avignon - Celsius Sarl, France)

"Liquefied gases: new alternative solvents for eco-extraction of natural products"

10.40 **O11** - Alexei LAPKIN (University of Cambridge, UK)

"Feasibility of using 2,3,3,3-tetrafluoropropene (R1234yf) as a solvent for solid-liquid extraction of biopharmaceuticals"

11.00 **Coffee Break**

CHAIR: JULIANO P. and ALIAKBARIAN B.

11.30 **K3** - Antoine BILY (Naturex, France)

"An industrial perspective on the use of natural deep eutectic solvent in extraction"

12.00 **O12** - Khalef LEFSIH (University of Bejaia, Algeria)

"Water-soluble pectin from *Opuntia ficus indica*: optimization of microwave-assisted extraction and preliminary characterization"

12.20 **O13** - Francisco J. BARBA (University of Copenhagen, Denmark)

"High Pressure tailor made processes: a useful tool to improve extractability and functionality of healthy compounds"

12.40 **O14** - Magali JACOTET-NAVARRO (University of Avignon, France)
"Life Cycle Assessment (LCA) as a tool for green extraction of natural products."

13.00 **O15**- Roberto ROSA (University of Modena and Reggio Emilia, Italy)
"Cradle to the grave Life Cycle Assessment of microwave assisted *vs.* conventional extraction for the obtainment of highly pure curcumin"

13.20 **O16** - Ray CHRISMAN (USA)
"A new process offers a cost competitive route to the production of polymer grade ethylene glycol from glucose"

13.40 **LUNCH**

15.00 **2nd POSTER SESSION**

CHAIR: LAPKIN A. and BARBA F.

15.40 **PL6** - Jochen STRUBE (Clausthal University of Technology, Germany)
"Production of plant extracts - Green challenges"

16.20 **O17** - Barbara SGORBINI (University of Turin, Italy)
"Green strategies for the analysis of the plant volatile fraction"

16.40 **SG3** - Katya CARBONE (CREA-FRU, Italy)
"Fruit processing by-products as potential bio-factories for green synthesis of metallic nanoparticles"

17.00 **Awards ceremony**

17.15 **Final Remarks & Closing**

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Plenary Lectures

PL1 - GREEN EXTRACTION OF NATURAL PRODUCTS: PAST AND PRESENT PANACEA

Farid Chemat

GREEN (Groupe de Recherche en Eco-Extraction des produits Naturels)

UMR408, INRA, Université d'Avignon et des Pays de Vaucluse

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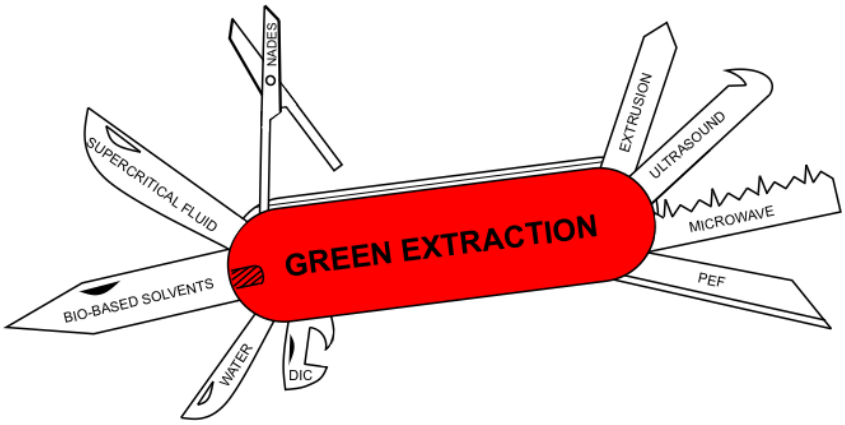
Abstract

This presentation will introduce a new and innovative area in the frontiers of chemistry, biology and processing: green extraction with special emphasis on natural products. Green extraction is a part of the sustainable development concept; its history, concept, principles and fundamentals will be described. We will pay special attention to the strategies and the tools available to make biorefinery greener. The representation will present the innovative research in this area these past five years in term of innovative techniques (microwave, ultrasound, pulse electric field...) and alternative solvents (ionic liquids, sub and supercritical fluid, agrosolvents, water...) applied to this new area green extraction of natural products with special examples applied to biorefinery concept.



A general definition of green chemistry is the invention, design and application of chemical products and processes to reduce or to eliminate the use and generation of hazardous substances. In relation of green extraction of natural products, this definition can be modified as follows: "*Green Extraction is based on the discovery and design of extraction processes which will reduce energy consumption, allows use of alternative solvents and renewable natural products, and ensure a safe and high quality extract/product*". The listing of the "six principles of Green Extraction of Natural Products" should

be viewed for industry and scientists as a direction to establish an innovative and green label, charter and standard, and as a reflection to innovate not only in process but in all aspects of solid-liquid extraction. The principles have been identified and described not as rules but more as innovative examples to follow discovered by scientist and successfully applied by industry.

References

[1] Chemat F., Strube J. Green Extraction of Natural Products. Theory and practice, Wiley-VCH, Weinheim, 11 chapters, 384 pages, 2015, 978-3-527-33653-1

[2] Chemat F, Abert-Vian M, Cravotto G. Green Extraction of Natural Products: Concept and Principles, International Journal of Molecular Sciences, 2012, 13(7): 8615-8627

PL2 - DEEP EUTECTIC SOLVENTS AS NOVEL MEDIA FOR NATURAL PRODUCT EXTRACTION

Andrew P. Abbott

Department of Chemistry, University of Leicester, Leicester, LE1 7RH, UK

Abstract

Deep eutectic solvents, DESs, are mixtures of quaternary ammonium salts and hydrogen bond donors such as urea or glucose [1, 2]. There are in excess of 10^5 binary liquid mixtures possible and an almost limitless number of ternary and quaternary mixtures. Inclusion of hydrophobic and hydrophilic groups in the same liquid enables novel solvent properties to be blended. The concept of producing active ingredients as part of the DES will be explained using a variety of pharmaceutical and natural product examples. Many quaternary ammonium salts and hydrogen bond donors are naturally occurring and some food grade examples are demonstrated in this presentation. DESs have been used for a wide variety of applications including metal processing, biocatalysis, leather processing and material preparation [3]

References

[1] A. P. Abbott, G. Capper, D. L. Davies, R. Rasheed and V. Tambyrajah. Novel Solvent Properties of Choline Chloride/ Urea Mixtures, Chem. Commun., 2003, 70

[2] A. P. Abbott, D. Boothby, G. Capper, D. L. Davies, R. Rasheed and V. Tambyrajah. Deep Eutectic Solvents Formed Between Choline Chloride and Carboxylic Acids, J. Am. Chem. Soc. 2004, 126, 9142

[3] E. L. Smith, A. P. Abbott and K. S. Ryder. Deep Eutectic Solvents (DESs) and their applications, Chem. Rev. 2014, 114, 11060-82

PL3 - THE CONTROLLED PRODUCTION OF PLANT RAW MATERIAL FOR THE PHYTO INDUSTRY

Amin Chaanin

VitaPlant AG, Uttwil, Switzerland

Abstract

For the security of supply and delivery of high quality standards in raw plant materials, controlled cultivation becomes increasingly more important. Guideline standards such as GACP (Good Agriculture and Collection Practice) progressively take a more central position in the production and procurement of raw materials for the phyto industry. A change from wild collection into controlled cultivation is an ongoing procedure and many plants species are already grown commercially. For many years, VitaPlant has been working with the establishment of controlled cultivation of different plant species under the fulfillment of the GACP guidelines.

The establishment of controlled cultivation takes place in several steps: Sourcing and screening of genotypes which fulfill the requirements of the customers, development of reliable propagation methods, implementing techniques for the pilot cultivation of selected genotypes and finally the conducting of large scale production.

In *Petasites hybridus* (Butterbur) a global sourcing program in the natural distribution areas was performed to identify suitable plants for the production of foliage material. In total, several hundred accessions were collected originating from the natural distribution areas. In a next step a reliable *in vitro* protocol was developed and used for the propagation of selected genotypes and pilot experiments were carried out at different locations. Today, registered Petasites varieties produced by VitaPlant are grown commercially year after year in an area of more than 30 ha in locations that differ considerably from their natural habitat which is often by streams and forest edges.

Another example is the establishment and controlled production of *Vitex agnus-castus* seeds (Monk's pepper or Chaste tree). This shrubby plant is naturally distributed through the Mediterranean. The main issues with wild collections are the contamination with aflatoxins as well as the low content of active compounds. Firstly, for the controlled cultivation, different genotypes were tested at VitaPlant and then a selection for the controlled cultivation was made. Currently, several hectares have been established with a high planting density and the production and delivery of raw material in the required quality according to the European Pharmacopoeia Monograph has been ensured.

For another example, a similar program has been started with *Cimicifuga racemosa* (Black cohosh). An efficient *in vitro* method has already been established for the mass propagation of selected genotypes. Firstly, pilot cultivations have been started in the experimental fields at VitaPlant and they then will be followed by a large scale production.

A challenge in controlled contract farming is the securing of contractual obligations and the exclusivity of cultivation. The cost however, can be considerably higher than that of wild collecting.

PL4 - AN INTRODUCTION TO THE GREEN, CLEAN AND ECONOMICAL MAZZA EXTRACTION TECHNOLOGY

Benjamin Lightburn, Giuseppe (Joe) Mazza

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https://ca.linkedin.com/in/dr-giuseppe-joe-mazza-5b641345

Abstract

Health conscious consumers are seeking out naturally sourced functional foods and nutraceuticals, especially those that are produced using green technologies that are compatible with organic production and processing systems. This is driving a market demand for nutrients and herbal extracts that is currently estimated at $3.5 billion in North America alone.

The Mazza Innovation Ltd. patented Pressurized Low Polarity Water (PLPW) extraction technology is used to create high quality functional ingredients for applications in health products. Applications of the technology enable manufacturers to produce innovative, high quality products that are also competitive in costs savings compared to existing techniques.

This presentation will address the value proposition of the Mazza process, especially the high quality products at lower operating costs compared to existing solvent-based technology.

PL5 - ADVANCES IN MEGASONIC PROCESSING TO ENHANCE RECOVERY OF QUALITY OILS

Pablo Juliano, Mary Ann Augustin, Raymond Mawson, Xin-Qing Xu, Kai Knoerzer

CSIRO Food and Nutrition, Sneydes Rd, Werribee, VIC 3030, Australia

Abstract

In recent years, the use of high frequency ultrasound for droplet or cell separation from biomass has emerged outside the microfluidics space into the litre to industrial scale applications. The principle for this separation technology (referred to as Megasonics) relies on the differential positioning of individual droplets or particles across a high frequency (0.4 - 2 MHz) ultrasonic standing wave field within the reactor and subsequent biomass material predisposition for separation *via* rapid droplet agglomeration or coalescence into larger entities. Megasonics separation technology is being commercialised by the palm oil industry to minimise oil losses in the palm mill effluent, while providing higher oil recovery. It provides faster oil removal, thereby decresasing the need for extensive centrifugation. It has been recently demonstrated on a litre scale to assist olive oil, coconut oil and milk fat separation, and the size fractionation of milk fat globules. Ultrasound frequencies in the range of 100 – 1000 kHz have the potential to produce quality damaging free radicals in these oil bearing materials. Our investigations in the range 300 – 1000 kHz have found no evidence of quality damage resulting from the megasonic separation process. The ability to use megasonics as an intervention to improve oil recovery from food crops has implications beyond efficiency and differentiation. It also reduces waste, lowers environmental foot print of by-products of oil processing, with flow-on effects for improved food security.

PL6 - PRODUCTION OF PLANT EXTRACTS – GREEN CHALLENGES

Resource Efficient Manufacturing in Regulated Industries

J. Strube[1], M. Tegtmeier[2], H.-J. Hagels[3], R. Ditz[1]

[1] Institute for Separation and Process Technology, Clausthal University of Technology, D-38678 Clausthal-Zellerfeld, strube@itv-tu-clausthal.de

[2] Schaper & Brümmer GmbH & Co.KG, 38259 Salzgitter-Ringelheim

[3] Boehringer-Ingelheim Pharma GmbH & Co.KG, 55216 Ingelheim

Abstract

Plants come back into focus also of the chemical industry in a time of increasing impact for sustainability and environmental compatibility. Plant constituents substitute basic chemicals of petrochemical origin more and more. Plant material is green and sustainable, but chemical production parameter like a-polar solvents have to be overcome in order to gain true renewable and sustainable life cycles. The first step for preparing the desired natural constituents is always an extraction. Today the central aim for research and development of extraction procedures are careful, efficient and successful processes. Only this will guarantee the necessary high quality and attractive economic efficiency in the use of plant extracts. In the last years, process design as well as optimization of existing processes is supported by modelling the unit operations. Therefore, model parameters must be determined in lab scale [1].

On the other hand, new principles in the research are generated, which allow a rapid screening of possible conditions for extraction in the view of basic proceedings, solvents, temperatures and pressures. So, also the complex character of plant extracts is considered, which is determined by the multicomponent mixture existing of the group of the interesting constituents and also the side-fractions. The extraction process has to guarantee that side-fractions are not critical in the following use of the plant extract. For that, all problematic fractions should not be extracted, which will be achieved by selective extractions [2, 3]. Concepts and cost structures for further product purification will be discussed with regard to innovative resource efficient manufacturing technologies [4].

An efficient concept for total process development and evaluation will be presented based on QbD (Quality by Design) principles according to FDA/EMA demands of biologics – which may prove benefits if applied to botanicals as well. Integration of PAT (Process Analytical Technology) in process development and manufacturing will be shown and discussed.

In addition, the activities of the German Dechema/ProcessNet working group on “plant-based extraction – products and processes” are presented and thereby opened for any participation. [5]

References

[1] S. Both, I. Koudous, U. Jenelten, J. Strube. Model-based equipment-design for plant-based extraction processes-considering botanic and thermodynamic aspects, C. R. Chimie (2013). http://dx.doi.org/10.1016/j.crci.2013.11.004

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[5] http://www.processnet.org/en/Plant+based+Extracts+_+Products+and+Processes-p-222.html

Keynotes

K1 - FUNCTIONALIZED BIOAROMATICS FROM LIGNIN

Diels Ludo

Antwerp University, Flemish Institute for Technological Research (VITO), Business Unit Separation and Conversion Technology, Mol, Belgium

Abstract

The lignocellulose fraction of biomass is an attractive starting point for the production of value-added bio-aromatics, primarily for use as building blocks within the chemical and material industry. Depolymerization of lignin typically results in complex mixtures comprising a wide array of phenolics, bearing a variety of oxygen-based functionalities and covering a broad range of molecular weights. However, in many cases, valorization of these lignols can only be pursued from well-defined fractions. VITO's research activities in this context primarily focus on the development and demonstration of membrane separation processes for fractionation and purification of lignins and lignin degradation fragments to enable the use of these molecules in further applications. Thanks to their low energy requirements, mild processing conditions, often unique separation capabilities, straightforward operability, moderate cost-performance ratio and flexibility in equipment design, membranes are indeed a highly attractive technology for use within tomorrow's lignocellulosic biorefineries.

In this presentation, the potential of membranes in the value chain of lignin towards bio-based aromatics will be illustrated through some recent case studies. Whereas standard membrane processes allow to separate and classify lignin degradation products by molecular weight, VITO developed an innovative method to functionalize membranes for more challenging separations. Recent work revealed promising results in the use of membranes with tailored surface properties for affinity-based fine-separation of small lignin fragments from complex hydrolysates.

K2 - PHYTOEXTRACTIONS USING SUPERCRITICAL CO_2

Christof Kersch, M. Tippelt, J. Schulmeyr, N. Igl

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1. Introduction

Usually organic solvents are used to separate substances from a mixture. But equally inert gases are highly effective as solvents after inducing a liquid like density by increasing pressures. Especially for the separation of sensitive ingredients out of plant material for food or cosmetics supercritical CO_2 is used at NATECO$_2$.

2. Summary

Technology

To generate supercritical CO_2, liquid CO_2 is pressurized and warmed up. Then the supercritical solvent is loaded with soluble substances after flowing through the extractor, which is charged with plant material. Subsequently the pressure is reduced and the phytoextracts precipitate into separators.

Advantages

The process is suited for sensitive products by applying moderate temperatures in an oxygen free atmosphere without using any organic solvents. Additionally tailor-made extracts can be produced with sophisticated fractionation systems or adjacent technologies like counter-current columns or particle generation techniques.

Applications

Nowadays breweries often use hop extract for the aromatization of beer. To concentrate the bitter substances the CO_2 extraction technology is the method of choice. Also cocoa is degreased, protein concentrates are defatted, tea or coffee are decaffeinated, oils are bleached, vitamins are enriched, omega-3 fatty acids are gained or carotenoids are gathered in big scale - just to mention a few potentials of supercritical CO_2

3. Conclusions

The CO_2– technology is used worldwide for the refinements of natural products. The great spectrum of applications, trends, challenges and advanced prospects of the respective processes will be presented in the lecture.

K3 - AN INDUSTRIAL PERSPECTIVE ON THE USE OF NATURAL DEEP EUTECTIC SOLVENT IN EXTRACTION

Antoine Bily

Naturex

Abstract

Deep eutectic solvents arising from natural building blocks — the so-called NADES — have attracted a lot of attention from researchers due to their great promise as natural alternatives to conventional solvents. Their industrial implementation for extraction purpose, however, is still in its infancy. In this context, the recent development of commercial eutectic extracts and actives for the cosmetic sector paves the way to a new generation of botanical liquids. Here some eutectic extracts are presented compared to their standard hydroglycerinated counterparts in terms of chemical profiles and cosmetic benefits. We show that the eutectigenesis-assisted extraction process preserves biologically-important molecules, delivering greater concentrations of actives, richer compositions, and higher biological performances.

Oral Presentations

O1 - THE EXTRACTOR NAVIGLIO IN FOOD AND BEVERAGE PRODUCTIONS

Daniele Naviglio

Department of Chemical Sciences, University of Naples Federico II, via Cintia 4 – Monte S. Angelo Complex – 80126 Naples – ITALY, naviglio@unina.it

Abstract

For over a decade the Naviglio Extractor is a good alternative to solid-liquid extraction techniques such as maceration and percolation, as has been amply demonstrated that it is able to provide the same quality (if not more) of the extracts obtained with the traditional extraction techniques, also provides additional benefits such as significant reduction in the time of the extraction process (Ten days of maceration extraction equals one hour of extraction by the Naviglio extractor equal conditions) and a more efficient extraction. The principle on which it bases its functioning (Naviglio's Principle) is studied in graduate courses in Herbal Techniques of several Italian Universities. Currently, the Naviglio extractor is widely used in many fields of research and production (herbal, nutritional supplements, omeoterapici, cosmetics, beverages). In the food sector, in particular, the Naviglio Extractor has been shown to be a viable alternative to macerate for production of lemon liquor (limoncello) and similar liquors; and production of bitter elixir of juniper; rapid aging of wines, brandies and liqueurs; extraction of lycopene from tomato processing waste. Finally, more unconventional applications of the Naviglio extractor have recently been found as the rapid hydration of dried vegetables and their simultaneous aromatization, cleaning washers for the production of cork stoppers, cleaning the rubber polymer, the tanning of leather.

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O2 - SUSTAINABLE VALORIZATION OF AGRI-FOOD RESIDUES BY HIGH PRESSURE HOMOGENIZATION PROCESSING TO UNLOCK HIGH-ADDED VALUE INTRACELLULAR COMPONENTS

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Abstract

Residues from agri-food industry, including both by-products and wastes, often represent an environmental burden, either in terms of the impact of their use in low-added value products, or of their disposal. However, they also represent an opportunity to bring economic and social benefits, because still rich in valuable intracellular compounds, which can be efficiently valorized if adequately recovered.

The present work addresses the use of wet milling by High Pressure Homogenization (HPH), as an efficient process to unlock high-added value intracellular components from plant-based agri-food residues through the mechanical disruption of vegetable cells. HPH processing has been tested in the valorization of different agri-food residues, such as tomato peels, coffee residues, and rice bran, primarily to increase the bioaccessibility of antioxidant compounds.

The vegetable matrices were suspended in water, pre-milled and screened at 600 μm. Subsequently, the vegetable suspensions were processed by HPH, at pressures ranging between 100 and 200 MPa and for 1 - 5 passes.

The results show that HPH processing causes a statistically significant increase in both supernatant antioxidant activity (related to the antioxidant compounds released in the aqueous phase upon HPH processing) and surface antioxidant activity (related to the antioxidant still bonded to the surface of cell debris).

However, HPH-processed suspensions exhibited also other interesting features, suggesting their possible use not only as functional ingredients but also as thickening agents or stabilizers: (a) the content of soluble protein significantly increased, with a consequent higher surface activity of the vegetable suspension; (b) the fine cell debris resulting from the mechanical disruption of vegetable cells caused a measurable increase in the viscosity of the suspension.

In conclusion, HPH processing of agri-food residues enables to unlock the high-added values components still remaining in the vegetable cells using a purely physical process and water as solvent, contributing to pursue the concepts of sustainable valorization and total use of the residue.

O3 - PEF-ASSISTED GREEN SOLVENT EXTRACTION OF HIGH-ADDED VALUE COMPOUNDS FROM AGRI-FOOD BY-PRODUCTS

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Abstract

Extraction of valuable compounds from biomaterials of microbial, algae or plant origin is a crucial step for the use of these compounds in the food and pharmaceutical industries. In particular, the recovery of valuable compounds (natural colorants or nutraceuticals) from food wastes and by-products, which have been matter of concern by the agri-food industry due to their environmental impact, is gaining increasing interest in recent years. However, traditional extraction methods are generally very time-consuming and require large quantities of solvents in order to achieve sufficiently high extraction yield of target compounds from agri-food products. Moreover, these methods may do not comply with criteria of green chemistry concept, since they may require application of toxic organic solvents for the selective recovery of target compounds. Consequently, demand is increasing for extraction techniques that improve yield, shorten the extraction time and reduce the use of organic solvents.

Recently, the interest in Pulsed Electric Fields (PEF) treatment of plant material before extraction with solvent, has considerably grown, due to its ability to induce a selective permeabilization of the cytoplasmatic membranes (electroporation), facilitating the release of intracellular compounds from the cells.

This manuscript gives an overview of the PEF-assisted solvent extraction, and presents the experimental results on the recovery of antioxidants and pigments (phenolic compounds and anthocyanins) from red fruits and vegetables by-products, which can be used as potential food additives and/or nutraceuticals. The effects of PEF process parameters (field strength, total specific energy input), as well as individual green solvents (water, ethanol) or their mixtures on the extraction yield of the compounds of interest is discussed.

O4 - EXTRACTION OF ANTIOXIDANTS FROM SPENT COFFEE GROUNDS USING MICROWAVE-ASSISTED AND HIGH PRESSURE AND TEMPERATURE EXTRACTIONS

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Abstract

Coffee-based beverages are one of the food industrial products consumed worldwide. The *International Coffee Organization* estimated a global production of coffee of $8.6 \cdot 10^6$ tons during the year 2015/2016 and consequentially generation of huge amount of wastes. As well as coffee products, spent coffee grounds are rich source of antioxidants, which can found several applications in food and cosmetic areas. In spite of the increasing interest on antioxidant recovery from spent coffee grounds, currently few studies compared the extraction methods with promising perspective for industrial processing. Then, the overall objective of this study was to examine the effects of two non-conventional techniques, Microwave-Assisted Extraction (MAE) and High Pressure and Temperature Extraction (HPTE), and the solvent role for the extraction of antioxidants from exhausted coffee collected from common vending –machines. Extract quality was evaluated on the basis of total phenolic and total flavonoids content and the antiradical power exhibited. Results showed that antioxidant-rich extracts (60 mg GALLIC EQUIVALENT/g (HPTE), 41 mg GALLIC EQUIVALENT ACID/g (MAE)) could be obtained by using a mixture of ethanol/water as the extraction solvent. In conclusion, the non-conventional techniques selected, mainly High Pressure and Temperature Extraction, are able to perform antioxidant recovery using green solvents, providing extraction yields that are higher than classical extraction process in which methanol is used.

O5 - VALORIZATION OF CHICORY BY-PRODUCT BY ULTRASOUND-ASSISTED EXTRACTION OF ANTIOXIDANT POLYPHENOLS: EXTRACTION YIELD AND ENERGY CONSUMPTION OPTIMIZATION

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Abstract

Cichorium intybus L. var. *sativum* (Asteraceae family) is widely cultivated in Europe for its roots which are used to produce a beverage (coffee substitute). During processing of chicory roots, several wastes are generated including chicory grounds (obtained after roasting of green slices, crushing into grains and extraction with hot water). Every year, about 15,000 tons of chicory grounds are generated in department of Nord (France). The interest in reusing such wastes increases for economic and environmental considerations. Chicory grounds still contain compounds of interest, including antioxidant polyphenols, which could be valorized by a suitable extraction process.

Ultrasound-assisted extraction (UAE) of antioxidant polyphenols from chicory grounds was studied in order to propose a suitable valorization of this food industry by-product using a green process. Several extraction experiments were carried out varying the main influencing parameters: temperature (20-60°C), ethanol content in the solvent (0-60% (vol.) in ethanol-water mixtures) and ultrasound power (0-100 W). During each experiment, kinetics of extracted polyphenols, antioxidant activity of the extract, as well as energy consumption were followed. On the base of this set of experiments, a global model was built, permitting the following and the prediction of the yield of extracted polyphenols, of the antioxidant activity of the obtained extracts and the energy consumption during the extraction process, as a function of extraction time, temperature, solvent composition and ultrasounds power (in the studied experimental field). Different simulations at different technological restrictions were performed to illustrate the potentiality of the model to find the optimal conditions for obtaining a given yield within minimal process duration or with minimal energy consumption. The advantage of ultrasound assistance was clearly demonstrated both for the reduction of extraction duration and for the reduction of energy consumption.

O6 - EXTRACTION OF BIOACTIVE COMPOUNDS USING A GREEN SOLVENT FROM FORESTRY BIOMASS

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Abstract

The ability to be used as an alternative solvent of a *cis*-rich pinane (*cis*/*trans*: 7/3), which was obtained for the first time through an environmentally friendly catalytic hydrogenation of α/β-pinenes or turpentine oil under neat conditions over Pd/C catalyst, was investigated and compared to *n*-hexane for the extraction of several bioactive compounds. Experimental evaluation as well as a simulation of the *cis*-rich pinane extraction capacity have been undertaken. The predictive approach was performed using the simulation software COSMO-RS (Conductor like screening model - realistic solvatation), which uses a statistical thermodynamics approach based on the result of quantum chemical calculation, for comprehension of dissolving mechanism. COSMO-RS simulation showed that the bio-based solvent is a good solvent to solubilize carotenoids, oil from rapeseed and aromas from caraway seeds. The experimental results indicated that the highest carotenoid yield was obtained with the *cis*-rich pinane with 95.4 % of the maximum carotenoid content in carrots while *n*-hexane was only able to extract 78.1% of them. Concerning to the extraction of oil from rapeseed, both solvents showed similar extraction yields and no differences were observed in the fatty acid profiles. In relation to the aromas, the characterization of the essential oil extracted by the two solvents showed a similar composition, where carvone (64%) was the main component followed by limonene (34%). No selectivity of the solvents was observed for any of the two major compounds. The results indicate that the bio-based solvent could be a promising solvent for *n*-hexane substitution.

Keywords: Green solvent, pinane, eco-extraction, bioactive compounds, COSMO-RS.

O7 - RECOVERY AND FRACTIONATION OF PHENOLIC COMPOUNDS FROM ROSEMARY LEAVES BY US AND MW-ASSISTED EXTRACTIONS

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Abstract

Rosemary (*Rosmarinus officinalis* L) is a very rich source of bioactive phenols, such as mono- and di-terpenes, phenolic acids, and flavonols, which are primarily responsible for this plant's high bioactivity. It is well known the use of rosemary extracts in gastronomy and traditional medicine for centuries. Consequently, the interest of scientific community in attaining these bioactive extracts by mean of efficient extraction processes is increased. In this work, we investigate the most efficient and selective conditions for the recovery of phenols from dried rosemary leaves under UAE (ultrasound-assisted extraction) and MAE (microwave-assisted extraction) using suitable solvents for food applications. All the extracts were then analysed by the combination of HPLC-DAD-MS TOF techniques.

Compared to a more traditional solid-liquid extraction process, the phenols yield was dramatically increased (more than three times) by MAE and UAE in ethanol and acetone. In term of selectivity, a remarkable high content of rosmarinic acid was obtained in ethanol under UAE.

Even more impressive was the selectivity of UAE using *n*-hexane affording the highest content of carnosic acid (up to 13% on dried extract). Among these non-conventional energy sources, the high-intensity ultrasound showed to be fast, efficient and selective technique for phenolic recovery of rosemary, affording fractions with a high content of rosmarinic and carnosic acids.

Regarding rosemary extract, nervous beneficial effects emerge from the traditional use and the scientific evidences. Here some results will be shown on *n*-hexane extract by UAE. This sample, selectively rich in terpenoids mainly in carnosic acid, was able to reduce neuropathic hypersensitivity and protect nervous tissues after *in vivo* administration in a rat model of peripheral neuropathy.

O8 - MICROWAVE-ENHANCED EXTRACTION OF ALGINATE FROM SARGASSUM SEAWEEDS: FROM COASTAL BEACH WASTES TO RAW FILM FOR AGRICULTURAL APPLICATIONS

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Abstract

Sargassum algae are brown free-floating seaweeds found worldwide in temperate and tropical regions, providing shelter and food for many animal species. In recent years, their wide diffusion has gone out of control, providing dense clumps of rotting weeds, yielding for toxic waste alongside urban beaches [1]. Nevertheless, these harmful brown seaweeds represent a valid source of Sodium Alginate (SA), a well known biodegradable and biocompatible polysaccharide, widely used in food, pharmaceutical and biomedical applications, due to its stabilizing and gelling properties [2]. In this work, new eco sustainable and cost-effective extractive methods, based on microwave-assisted extraction (MAE), have been used to obtain alginate by *Sargassum* seaweed wastes, in order to use it as polymeric matrix of mulching films in agricultural applications. As a matter of fact, MAE method has been successfully applied to extract several biologically active compounds from natural resources, using less energy, time and solvent volume, in this way resulting a more eco-sustainable process [3]. The extraction was performed following both the conventional protocol and a mild MAE.

Dried *Sargassum* seaweeds particles were exposed to controlled microwave energy in a specific equipment, where power density, duration, thermal regime of working air and exposure uniformity were optimized. Treated substrate was then subjected to sodium alginate extraction process, using hot water as solvent. At the same time, raw algae underwent to conventional extractive procedures. SEM analysis confirmed that MAE treatment induced seaweeds cell wall breaking, thus promoting the availability of SA, whereas FTIR-ATR analysis assessed the extraction of the polymer in both cases.

Finally, preliminary results related to SA extraction, evidenced that MAE technique could represent both a valid method to obtain sodium alginate in a cost-effective way and an environmentally friendly approach finalized to the upgrading of coastal beach waste materials.

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O9 - EXTRACTION OF ANTIOXIDANT POLYSACCHARIDES FROM *G. LUCIDUM* USING ULTRASOUND: SCALE-UP POTENTIAL

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Abstract

Utilisation of ultrasound to assist the extraction of active ingredients from natural resources has many advantages over other conventional techniques. Besides reducing the extraction time and solvent usage which are considered to be the major limitations of any extraction process, lesser energy consumption, higher yield of production, and higher preservation of bioactivity of the separated biomolecules due to lower extraction temperatures could be added as merits of ultrasonic extraction. Historically among a large number of mushroom species that have been reported for pharmaceutical applications, *Ganoderma lucidum* is considered to be one of the most precious mushroom species. The fruiting body, mycelia, and spores of *G. lucidum* contain approximately 400 bioactive compounds. Diverse groups of these bioactive compounds such as triterpenes, polysaccharides, proteins, nucleotides, nucleosides, metals etc. with different pharmaceutical activities have been isolated from this species. Among these bioactive compounds, polysaccharides have attracted the attention of many researchers due to their potential ability to treat many diseases. In particular, polysaccharides could be used as anti-oxidants to prevent the cellular damages caused by the free radicals resulted from oxidation reactions during the process of energy generation in the living organisms. Looking into the potential of this polysaccharide, ultrasonic assisted extraction (UAE) has been adopted which was then compared with other conventional methods of extraction such as soxhlet (SE) and hot water extraction (HWE). The characteristics and antioxidant properties of the extracted polysaccharides were also determined. UAE seems to be more productive and economical over other methods of HWE and SE if the time-factor and energy consumption of the process are taken into consideration. The lower extraction rate observed with the conventional methods (SE and HWE) makes these methods less efficient than UAE for the isolation of polysaccharides.

The UAE of polysaccharides (PS) from *Ganoderma lucidum* was subjected to a scale-up study. 0.25 L extractor was employed to optimize the extraction conditions toward maximum yield of PS. The extracted PS was observed to be reduced by increasing the scale from 1 to 6 L. To intensify the

extraction, axial circulation at different stirring rates was induced and optimized in a 3 L U-tube extractor**.** Although circulation at 300 rpm improved the yield of PS for 3 L, introducing dispersion geometry (conical funnel) and adjusting the radiation distance in a 6 L U-tube extractor further intensified the extraction efficiency. A radiation distance of 4 cm and circulation induced using 600 rpm enhanced the PS as compared to the conventional 6 L extractor. Overall, the scale-up from 0.25 to 6 L was successful and introducing circulation and dispersion geometry intensified the extraction efficiency under similar dissipation of ultrasonic power.

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O10 - LIQUEFIED GASES: NEW ALTERNATIVE SOLVENTS FOR ECO-EXTRACTION OF NATURAL PRODUCTS

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Abstract

Over the last decades, the research for new alternative volatile solvents has revived the interest on liquefied gases as extraction solvents. In the first place, many studies about vegetable extraction using supercritical CO_2 were performed, nevertheless the high working pressure limited considerably industrial applications. Therefore, more recently, new studies have been focused on the development of extraction processes involving liquefied gases at lower pressure.

In this work, the use of low pressure liquefied gases as substitution solvents was investigated for the extraction of natural compounds. For this purpose, a new method was developed at laboratory scale (1 liter). As an example, the applicability of liquefied n-butane was evaluated for the extraction of carrot oleoresin, caraway essential oil and sunflower oil from the corresponding raw materials. In a first step, the relative solubilities of lipophilic targeted components from carrot, caraway and sunflower in liquefied butane and hexane were assessed using a predictive approach (COSMO-RS simulations). This approach was completed by experimental study. For all extractions, the performances of liquefied butane were compared with those of hexane in terms of extraction yield and final extract composition.

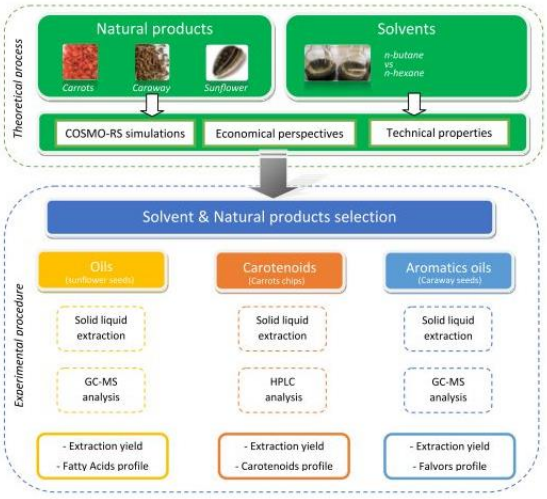

Figure 1 - Experimental diagram for the comparison of *n*-hexane vs *n*-butane for the extraction of natural products

O11 - FEASIBILITY OF USING 2,3,3,3-TETRAFLUOROPROPENE (R1234YF) AS A SOLVENT FOR SOLID-LIQUID EXTRACTION OF BIOPHARMACEUTICALS

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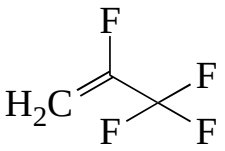
Abstract

Tetrafluoropropene (R1234yf) is the most likely replacement for tetrafluoroethane (R134a), a widely used refrigerant, propellant and solvent, characterised by very high global warming potential. In this study solvation properties of R1234yf were studied experimentally and computationally for solubility of artemisinin, a precursor to the important bio-pharmaceutical API, and extraction of artemisinin from biomass.



The new solvent was found to be comparable or better to many conventional solvents in solubility of artemisinin, especially at low temperatures. It was also found to be more selective towards artemisinin in solid-liquid extraction from biomass. The concentration of artemisinin in the R1234yf primary extract was superior to those reported for ethanol, hexane and toluene and similar to R123a extraction. This should allow for design of more selective separation processes based on the new solvent molecule with a low global warming potential of 4 relative to CO_2.

COSMO-RS calculations of solvation in R1234yf and R134a suggest that the difference in performance is likely to be due to entropic effects.

Reference

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O12 - WATER-SOLUBLE PECTIN FROM OPUNTIA FICUS INDICA: OPTIMIZATION OF MICROWAVE-ASSISTED EXTRACTION AND PRELIMINARY CHARACTERIZATION

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Abstract

Optimization of Microwave-Assisted Extraction (MAE) of water-soluble pectin (WSP) from *Opuntia ficus indica* (OFI) cladodes was performed using Response Surface Methodology (RSM). The effect of extraction conditions, extraction time (X_1), microwave power (X_2), pH (X_3) and solid-to-liquid ratio (X_4) on the extraction yield was examined using an experimental central composite design. The optimum conditions of MAE were as follows: X_1 = 2.15 min; X_2 = 517 W; X_3 = 2.26 and X_4 = 2g/30.6 mL. The maximum obtained yield of pectin extraction was 12.57%. Total carbohydrate content of WSP is about 95.5% including 34.4% of Galacturonic acid. Pectin-related protein represents only the 0.66% of WSP mass. HPSEC and light scattering analyses reveal that WSP is mostly constituted of high molecular pectin and FTIR measurements show that the microwave treatment does not alter the chemical structure of WSP, in which galacturonic acid content and yield are about 34.4% and 4.33%, respectively. Overall, application of MAE can give rise to high quality OFI pectin.

Keywords: Pectin; Microwave-Assisted Extraction; Surface Response Methodology; HPSEC; FTIR; Light Scattering.

O13 - HIGH PRESSURE TAILOR MADE PROCESSES: A USEFUL TOOL TO IMPROVE EXTRACTABILITY AND FUNCTIONALITY OF HEALTHY COMPOUNDS

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Abstract

High Pressure Processing has emerged over the last 20 years as one of the most promising non-thermal technologies for food preservation. This technology consists on applying high pressures (100-600 MPa) at mild temperatures for relatively short treatment times.

Apart from its high potential for microbial inactivation, HP has also been shown as a useful tool to improve healthy aspects from foods, (eg. improving bioavailability and bioaccessibility of foods, as well as reducing allergenicity of food products and the formation of food contaminants during processing).

Moreover, the potential of HP to recover healthy compounds from wastes and by-products from food industry has also been shown. It is of a great importance, as HP kills two birds with one stone and addresses both the use of waste and by-products and societal health, thus greatly contributing for a sustainable food chain from an environmental and economical point of view.

Furthermore, depending on processing conditions, HP can control enzyme activity, thus promoting the development of tailor-made processes which can decrease or increase enzyme activity. Particularly important is to control myrosinase activity from Brassica vegetables. For instance, there is a general consensus that to control the myrosinase activity in order to obtain the desired health beneficial effects in human is of paramount importance.

For this purpose, during the last few decades, several research efforts have been focused on developing new technologies to effectively control the complex glucosinolate-myrosinase system. Recently, some studies have shown the different behaviour of the glucosinolate-myrosinase system after applying HP conditions. Overall, these studies concluded that HP can be a useful technology to effectively control the complex glucosinolate-myrosinase although HP parameters had an impact on the extent of glucosinolate hydrolysis and, thus, on the type and amount of glucosinolate degradation products.

O14 - LIFE CYCLE ASSESSMENT (LCA) AS A TOOL FOR GREEN EXTRACTION OF NATURAL PRODUCTS

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Abstract

Most industrials and academics have now adopted the philosophy of "Green Chemistry" and more particularly in the extraction field where emerged the concepts and principles of "eco-extraction" or "green extraction". The eco- or green processes have to be designed as far as possible considering these aspects which seek to recover a natural and safe extract (principle 6), to reduce the use of organic solvent (principle 2), to decrease energy consumption (principle 3) and extraction time (principle 5). Well-reasoned sourcing (principle 1) and production of by-products with a high added value instead of waste (principle 4) have to be assessed as well [1]. To evaluate the performance of a new eco-process or an existing one which has been improved, some aspects of Life Cycle Assessment (LCA) can be used. LCA is a multi-criteria study which enables to quantify the potential environmental or social impacts of a product or a service during all its life, from the cradle (extraction of raw material) to the grave (end-of-life treatment). For each step of product life cycle, an assessment of total inputs and outputs has to be done, that is energy or non-energy resources for inputs, and emissions into the air, the soil and the water for outputs. Overall, it is difficult to assess the impact and particularly the environmental footprint of a product during its entire life cycle, so industrials generally adopt a "gate to gate" approach considering the step from the exit of field until the exit of factory (Figure 1).

Despite the existence of a huge number of databases directly implemented in LCA software, there is a lack of data especially dedicated to the extraction field and more particularly extraction processes. The specific environmental impact of a process is therefore difficult to estimate and needs a heavy data gathering directly in the production site. However, it is necessary to do this work, particularly if one aims at reducing the environmental impact of a current process and wants to quantify the continuous improvement applied on processes in terms of reduction of impact.

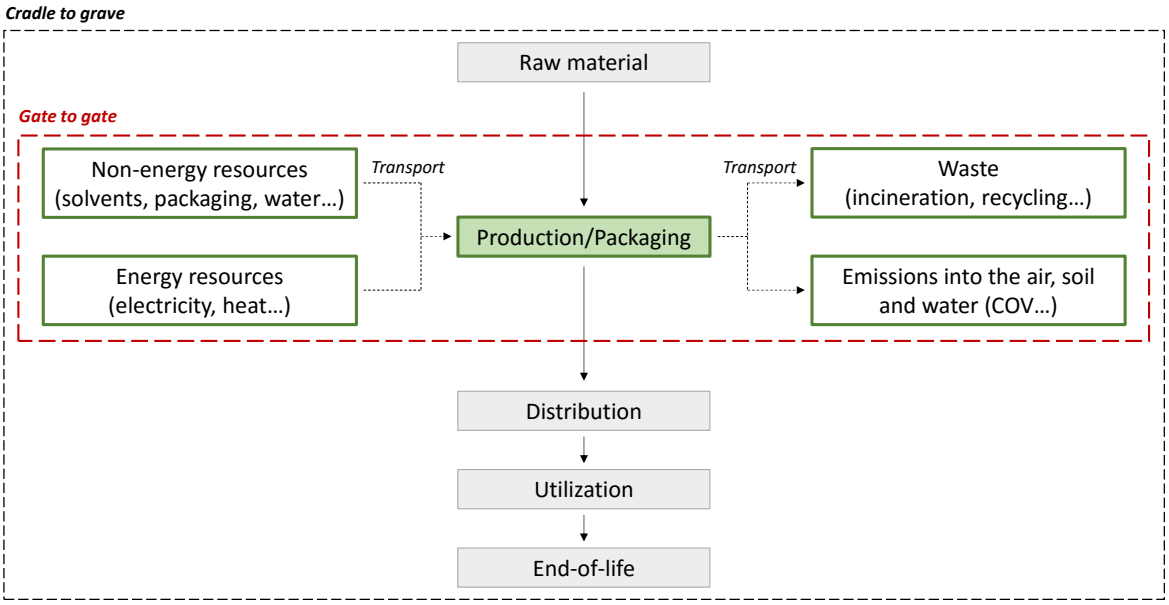

Figure 1. "Gate to gate" approach in Life Cycle Assessment (LCA) methodology.

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O15 - CRADLE TO THE GRAVE LIFE CYCLE ASSESSMENT OF MICROWAVE ASSISTED VS. CONVENTIONAL EXTRACTION FOR THE OBTAINMENT OF HIGHLY PURE CURCUMIN

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Abstract

Microwave assisted extraction of natural compounds is widely recognized as one of the most promising green extraction techniques [1], even if comprehensive comparisons with more conventional procedures are surprisingly scarce, being on the other hand limited to mere considerations concerning extraction time and yield.

In this work, "cradle to the grave" environmental assessments of microwave assisted compared to conventional Soxhlet-based extraction procedures will be presented, for the particular case of curcumin molecule, extracted from *Curcuma longa* L., in view of its important antioxidant, anti-inflammatory and anticancer properties. The Life Cycle Assessment (LCA) methodology was applied, since it allows evaluating the environmental consequences associated with all the stages of the extraction process, thus including the crop production of the Indian plant, the subsequent production of the dried rhizomes, their commercialization, their transport, all the energy consumptions needed, the necessary laboratory facilities and their maintenance, together with the chemicals needed and their disposal treatments. The study was then completed by also comparing the obtained results with those of a particular synthetic strategy of curcumin [2], in order to establish the most environmentally friendly production procedure of this fundamental phytochemical [3].

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O16 - A NEW PROCESS OFFERS A COST COMPETITIVE ROUTE TO THE PRODUCTION OF POLYMER GRADE ETHYLENE GLYCOL FROM GLUCOSE

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Abstract

The use of commodity chemicals from renewable sources is often stymied by the higher costs that are usually required to produce them. The high cost is often a result of lower selectivity conversion chemistries being implemented in batch processes, the combined impact of which results in higher costs. Unfortunately this approach loses the cost advantage of clean low cost renewable starting materials. This presentation will describe the development of a very selective continuous process for the production of ethylene glycol from corn starch derived glucose.

This process could have a significant impact on the demand for renewable raw materials since the market for ethylene glycol is over 33 billion dollars per year and is growing at more than 7% per year. The bulk of ethylene glycol goes into making PET for packaging and fibers which are markets that value the use of renewable materials. This process focuses on the use of corn starch as the raw material, which, as has been pointed out by several researchers, is a major insurance policy for food resources since increases of overall food crop plantings provide additional immediate capacity if there are crop failures in the world.

The presentation will describe in some detail the chemistry and a continuous process that has now been evaluated in literally hundreds of lab scale runs. The new process is expected to be cost competitive for the production of polymer grade, commodity scale, ethylene glycol.

O17 - GREEN STRATEGIES FOR THE ANALYSIS OF THE PLANT VOLATILE FRACTION

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Abstract

The plant volatile fraction is an important biosensor that is not only diagnostic of plant metabolism and changes, but also, an effective marker of other fractions, including non-volatile metabolites. Recently, Maffei and Bicchi defined the plant volatilome "… as the complex blend of essential oils (EOs) and volatile organic compounds (VOCs) fed by different biosynthetic pathways and produced by plants, constitutively and/or after induction, as a defense strategy against biotic and abiotic stress" (1,2). Moreover, plant volatiles can also be responsible for important plant biological properties. The extended role played by the plant volatiles have greatly contributed to increase the interest in their study in particular by adopting methods and technologies able to characterize raw plant materials and to quantify key-aroma and/or biologically active markers (even at trace level).

Sampling of the volatile fraction includes a range of approaches and/or techniques, which produce samples that differently represent the volatiles characterizing a vegetable matrix, e.g. headspace, essential oils and solvent extracts. The choice of a suitable approach to analyze the composition of the volatile fraction of a plant material involves several factors including speed, ease of use, complete automation while avoiding, of course when possible, the use of solvents. In this perspective, green approaches (solvent free) based on High Concentration Capacity Headspace Sampling techniques combined with separative (HCC-HS/GC-MS) and non-separative analysis (HCC-HS/MS) have been developed and applied to the analysis of the plant volatile fraction.

This lecture is a short overview showing some applications of the above HCC-HS techniques applied to the analysis of the plant volatile fraction, in particular:

- characterization and quantitation of biologically active markers in aromatic plants and spices,
- determination of free and glucosidically-bound volatiles in peppermint and in cloves,
- evaluation of the topographical dynamics of volatile organic compounds emission from lima bean in response to mechanical and herbivory damages.

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Shot-Gun Presentations

SG1 - GREEN EXTRACTION PROCEDURES OF LIPIDS FROM SEEDS OF DIFFERENT TUNISIAN DATE PALM VERITIES

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Abstract

This study was performed to compare conventional and green extraction of vegetable oil from date palm (*Phoenix dactylifera* L.) seeds of three cultivars (Deglet Nour, Allig, and Belah) in of term of alternative solvents and modern techniques. It was observed that Deglet Nour presents the highest lipids extraction yield (7.24-5.97%). Hence, Deglet Nour variety was selected to perform green and clean alternative extraction procedures (Microwave and Ultrasound) for lipids extraction. These eco-techniques were qualitatively and quantitatively compared to conventional extraction techniques (Soxhlet and maceration) using gas chromatography coupled with a flame ionization detector (GC/FID) and high performance thin-layer chromatography (HPTLC). These four extraction procedures were carried out with *n*-hexane and MeTHF, a bio-sourced solvent. Hansen solubility simulations according to the classical "like dissolves like" demonstated that MeTHF is a good alternative solvent for the selectivity of fat and oils. In addition, it was concluded that ultrasound and micro-wave are the most interesting extraction procedures giving the highest yield (6.18-5.57 %, 5.52-4.74%) within the shorter time (30 min). The recovered fatty acids were mainly oleic (44.02-46.9%), lauric (20.00-23.10%), myristic (8.88-11.26%), palmitic (9.00-10.73%) and linoleic (6.13-9.21%). The obtained green-extract would be used in some agro-food and cosmetic preparations as an alternative to the undesirable synthetic additives.

SG2 - NEW ULTRASOUND APPLICATIONS

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Abstract

In this contribution, equipments for innovative ultrasound applications are presented:

- SONICATOR TYPE F_RI

Frequency 20.000 Hz,

MAX Power 1.500 W

Adjustment from 0 - 100%

Frequency regulation based on transducers geometry, respecting the research protocol.

Temperature control, control of treatment or process time. Possibility to connect to a computer and read constantly the frequency.

- TRANSDUCER TYPE FT_RI

Length of emission 1250 cm

Diameter 65 mm

Radial vibration transducer

Frequency of 27.000 Hz

Power 2.500 Watts

Important features: high power in a small space and pressurized head with dry Elio (He) to prevent overheating and keep the temperature low.

- PLANT ATP1

Ultrasonic device for researches in laboratories, universities and industries. Ultrasound on all sides, above and below. In a continuous flow, temperature control. It allows you to work under pressure or depression.

Power generator 380-400 V three-phase + neutral + ground

Treatment tank with capacity of 20 L.

Air in: 4-5 bar

N. 24 ultrasound emitters, with split of 16 on the 8 sides (2 per side), 4 on the top, 4 on the bottom.

Total ultrasound Power: ca. 2800 W

Frequency of 23.000 Hz with scansion +- 100 Hz

SG3 - FRUIT PROCESSING BY-PRODUCTS AS POTENTIAL BIO-FACTORIES FOR GREEN SYNTHESIS OF METALLIC NANOPARTICLES

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Abstract

In the past few years, a growing number of studies on bio-inspired nanomaterials has been published. Among nanomaterials, metallic nanoparticles (MNPs) are the most promising ones due to their biological properties (e.g. antimicrobial, antifungal, anti inflammatory properties). In this scenario, the use of fruit processing by-products as potential bio-factories for green synthesis of MNPs is an innovative and challenging field of research with considerable prospects. The present study aimed to assess the feasibility of using an aqueous white grape pomace extract (WGPE) as well as two grape pomace flavonoids (FLs) (e.g. catechin, and epicatechin gallate) in the preparation of silver NPs through a simple and rapid microwave assisted method of green synthesis. FLs were used as pure compounds, while WPGE was obtained from freeze-dried grape pomace by microwave-assisted extraction. All these green reagents were used as both reducing and capping agents for the synthesis of AgNPs. Bio-reduction of Ag^+ ions in solution was monitored by visual observation and confirmed by using UV-Vis spectral analysis. A clear Surface Plasmon Resonance (SPR) band in the range of 400 – 500 nm was observed for all green reagents used. SPR bands were centred at 426 and 432 nm for FL- and WPGE-loaded silver nanoparticles, respectively. Scanning electron microscopy (SEM) investigation evidenced that obtained Ag powders were composed of nano-sized rounded particles with an average diameter less than 35 nm. Moreover, the electrochemical properties of synthetized AgNPs were assessed by cyclic voltammetry (CV), showing an electrochemical behavior similar to that reported in literature for AgNPs obtained by chemical synthesis. The collected results highlighted the potential of these natural reagents as innovative, inexpensive, green agents for the synthesis of bio-inspired nanoparticles, with optical and electrochemical characteristics similar to those of conventional ones. Results also suggested the possibility of using these NPs for the design of new and improved sensing devices.

Poster Presentations

P1 - ALTERATION OF THE YIELDS AND COMPOSITION OF THE POLYPHENOLICS EXTRACTED FROM PLANT BIOMASS AFTER IT TREATMENT IN THE MICROWAVE FIELD

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Abstract

Microwave (MW) treatments of lignocellulosic materials are widely recognized for improvement their goal-oriented characteristics such as hydrophobicity, thermostability, non-volatile carbon content increasing. These processes accompanied by decreasing of material density conditioned by thermolabile volatiles removing. These changes directly depends on the regimes of MW treatment, in particularly, power of MW irradiation, its duration and type of biomass [1].

The research object was evaluation of influence of MW treatment realized at torrefaction regimes of granulated plant biomass on the yield and composition of extracted polyphenolics.

Pine and spruce wood sawdust mixture, aspen wood and wheat straw, preliminary granulated without any additives were used as raw material for MW treatment. The original laboratory device (1.3 kWh), designed using numerical modeling for optimization of influence of resonator geometry on the inside distribution of electromagnetic field was isolated [2]. Temperature of biomass varied in the range of 150 -300ºC and was hold 10 min. In parallel accelerated solvent extraction with ethanol water (60:40) was applied for both initial and treated biomass.

Recovery of polyphenols from microwave treated biomass is three times higher for aspen wood, 2,4 times - for wheat straw and 1.7 times - for softwood in comparison with phenolic recovery from untreated biomass. However, chemical composition of extracts varied depending on severity of treatment: the yield of low molmass compounds (< 1000 Da) decrease, but compounds with higher molmass (> 1000 Da) may be additionally extracted. In some cases (extraction of proanthcyanidins) changes in extract composition were observed. The regimes of treatment have to be adjusted taking into account properties of polyphenols to be isolated. So growth of pinosylvins yield is stopped at 180ºC (0.4% of dry wood), and at the 230ºC – only trace amount of it can be found in the extracts.

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P2 - NEW HETEROGENEOUS CATALYSTS FOR THE PREPARATION OF BIOMASS-DERIVED: ALKYL LEVULINATES*

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Abstract

Lignocellulosic biomass, composed by hemicellulose, cellulose and lignin, is classified as one of the most important clean and renewable resource that is not in competition with food chain biomasses. There is a great interest in biomass as a source of energy or power, whereas this raw material has been much less exploited for chemicals production. The list of chemical products that can be derived directly from biomass as well as from their key derivatives (called platform chemicals) is surprisingly long, and includes a wide range of value added products such as polymers, additives, solvents, cosmetics, pharmaceutical principles. In 2004 the Department of Energy of USA included levulinic acid in the top 10 platform chemicals derived from biomass [1].Among the materials with high added value obtained from levulinic acid it should be mentioned the alkyl levulinates. In fact, over the past five years alkyl levulinates have attracted the attention of researchers due to their specific physical-chemical properties and their possible applications. At this regards, they are commonly employed as flavoring agents, biofuel additives and important precursor for producing γ-valerolactone [2]. γ-valerolactone is useful as a fuel additive, green solvent [3] and precursor for the production of other valuable chemicals. Interestingly, alkyl levulinates may also used directly as green bio-based solvents in a range of organic transformations or extractions [4]. In this communication we report the first results of a broad research project aimed at the development of fully green synthetic methodologies for the preparation of alkyl levulinates on a large scale. The preparation of alkyl levulinates starting from levulinic acid implies the use of an alcohol as a reagent, in the presence of an acid catalyst . Our approach is based on the preparation of novel solid acid catalysts featuring different properties in term of density of active sites, porosity and site accessibility. These catalytic systems are specifically tailored with the final goal of defining flow protocols able to manipulate levulinic acid in continuous mode.

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**We gratefully acknowledge the ''Fondazione Cassa di Risparmio di Terni e Narni (CARIT)'' for support.*

P3 - ENABLING TECHNOLOGIES FOR THE GREEN EXTRACTION OF LIGNIN USING BIOMASS-DERIVED γ-VALEROLATTONE.

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Abstract

Lignocellulosic biomass is the most abundant renewable source of biofuels and biochemicals [1]. Agricultural waste represent a valuable source of platform chemicals as they do not compete with food feedstock. γ-Valerolactone (GVL) is the most attractive and versatile lignocellulose chemical derivative as it is renewable, safe to store, biodegradable and can be used as a green solvent in fine chemicals synthesis [2] and biomass processing [3]. Several attempts have been studied in the last three decades to make processes more sustainable. From this point of view, new technologies such as microwaves (MW) and ultrasound (US) provide high-energy microenvironments that strongly promote biomass deconstruction at shorter reaction times or mild conditions [4]. Lignin extraction from lignocellulosic biomass (wood, annual plants) is the key step to large scale use in industrial applications. For example, it has attracted increasing interest over the last few years as a means to produce dispersants, adhesives and surfactants, as an antioxidant in plastics and rubbers, as well as the raw material for the synthesis of value-added products [5]. Concerning biomass pretreatment for microbiological processes, the extraction of lignin allows hydrolytic reagents an improved access to cellulose in the subsequent hydrolysis steps. In this work, γ-valerolactone (GVL) was synthesized starting from biomass derived levulinic acid through a new solvent-free protocol under microwave irradiation. GVL was then used in a cascade MW-assisted integrated protocol for lignin extraction (6) and as a green solvent for US biomass pre-treatment. Different US device were tested in order to compare the energy consumption, the yields of the solid residue and liquid fraction, and the generation of inhibitors. The parameters for the treatment with US were optimized in order to obtain an effective disruption of the lignocellulose matrix and increased accessibility for further processing steps.

Aknowledgements: This work was funded by European Community (H2020 project US4GREENCHEM Grant Agreement No 669055) and Università degli Studi di Torino (Fondi Ricerca Locale 2013).

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P4 - GREEN ENABLING TECHNOLOGIES FOR PROCESS INTENSIFICATION IN BIOMASS EXTRACTION AND CONVERSION

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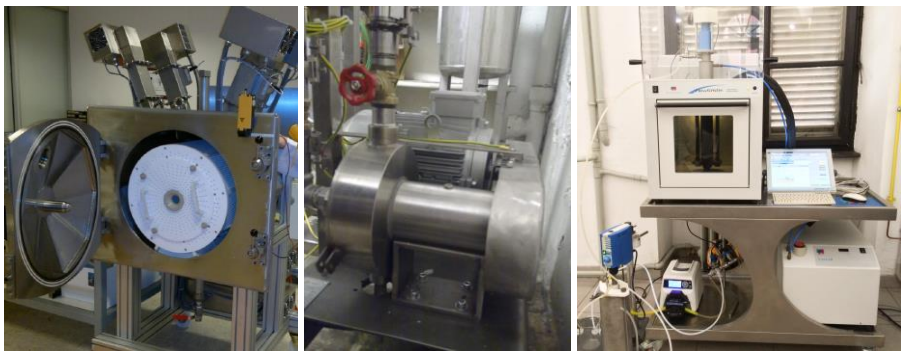
Abstract

In the last two decades green extraction technologies moved from lab-scale curiosity to R&D pilot reactors and in few cases even industrial plants. Besides ecological issues and product quality, green extraction principles [1] could generate business profit, safer protocols and energy saving. Remarkable advantages of non-conventional extraction techniques are also higher selectivity, shorter extraction time and often a higher yield. Among the main technologies exploitable in this field, we can mention microwaves, ultrasound, hydrodynamic cavitation, ball milling, supercritical CO_2 and subcritical water [2-4]. The development of new dedicated reactors allowed continuous or semi-continuous extraction processes, enhancing scalability and productivity. A smart combination of these techniques may offer unexpected synergistic effects. Striking examples are the cryomilling in planetary ball-mills or hammer-mills drums as first step of sequential processes for biomass extraction and conversion under microwaves and/or ultrasound [5]. Rotor-stator hydrodynamic cavitation reactors are the most attractive recent achievement to operate in flow mode (continuous or semi-continuous treatments). Different valuable examples of microwave-assisted extraction and hydrodistillation, with or without a steam flow will be presented. Different techniques and methods will be compared in the extraction of lycopene and carotenoids from residual tomato peels, alginates and oil extraction from algae and the recovery of essential oils, pectin and cellulosic material from citrus peels [6].

a **b** **c**



a: MAC75 Milestone (BG) b: Rotocav Epic (TO) c: FlowSynth Milestone (BG)

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P5 - BIO-BASED SOLVENTS FOR GREEN EXTRACTION OF LIPIDS FROM OLEAGINOUS YEAST BIOMASS FOR SUSTAINABLE AVIATION BIOFUEL

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Abstract

Lipid-based oleaginous microorganisms are potential candidates and resources for the sustainable production of biofuels. They can accumulate 20% to 70% of their dry cell mass as lipids (Triglycerides: TAG, Diglycerides: DAG, Monoglycerides: MAG and Phospholipid: PL) converting carbon sources contained in various substrates. Currently, technique for extraction of lipids from microorganisms use petroleum solvents, such as hexane, methanol or chloroform. From the point of view of environmental protection, these flammable and toxic petroleum solvents will have to be replaced in the future by bio-based solvents.

This work was designed to evaluate the performance of several alternative bio-based solvents for extracting lipids from yeasts. It combines an experimental and theoretical approaches. The experimental part is based on the extraction of oil from *Yarrowia lipolytica* IFP29. The oil from yeast obtained was analyzed by high performance thin-layer chromatography (HPTLC) to obtain lipid classes and gas chromatography coupled with a flame ionization detector (GC/FID) for fatty acid profiles. For the simulation part, we used simulation sofware: the conductor-like screening model for realistic solvation (COSMO-RS).

COSMO-RS evaluates the interactions between solvents and solute (FFAs, DAGs, TAGs and PLs) present in the yeast. It simulates the relative solubility of the solute in the selected solvent, here 2-methyltetrahydrofuran (MeTHF), cyclopentyl methyl ether (CPME), isopropanol (IPA), ethanol (EtOH), ethyl acetate (EtOAc), ethyl lactate, dimethyl carbonate (DMC), *p*-cymene, D-Limonene, α-pinene and hexane. The aim of this work was to correlate simulation with experimentation for extraction and solvation of lipids with bio-based solvents in order to make a preliminary evaluation for the replacement of hexane to extract lipids from yeast.

P6 - PRODUCTION OF MICROALGAL VOLATILE ORGANIC COMPOUNDS IN AGROINDUSTRIAL WASTEWATER

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Abstract

The objective of this study was to investigate the volatile organic compounds (VOCs) produced from heterotrophic cultivation of the cyanobacterium *Phormidium autumnale*. The volatiles were isolated by headspace solid-phase micro-extraction in different residence times (0, 24, 72, 96, 120 e 144 hours), separated by gas chromatography, and identified by mass spectrometry (SPME-GC/MS). A total of 73 compounds were separated in the start of the cultive. The major one was ρ-cresol, benzaldehyde, dimethyl sulfide, toluene, skatole and indole. As consequence of microalgal cultive 48 compounds disappeared, whereas 38 was formed. The descriptor odor of the compounds formed detected in experiments was mainly classified as fruity, spice, and floral compounds. The major was the compounds formed 2-nonanone (1.37 μg mg^{-1}), 2-heptanone (1.81 μg mg^{-1}), 2,4-dimethyl-3-pentanone (2.08 μg mg^{-1}), benzophenone (2.57 μg mg^{-1}), cyclohexanol (5.25 μg mg^{-1}), and benzothiazole (6.36 μg mg^{-1}). In conclusion, the results have shown that the heterotrophic cultivation of the *Phormidiun autumnale* can be a potential biotechnological to produce natural flavours. In view comercial significance, efforts should be made to elucidate the pathways of formation for these compounds.

P7 - FROM WASTE TO NATURAL PIGMENTS: PRODUCTION OF MICROALGAL CHLOROPHYLL IN AGROINDUSTRIAL WASTEWATER

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Abstract

The metabolic versatility of cyanobacteria allows high productivity of biomass from industrial waste, which may be potentially exploited as raw material in biorefinery systems. Through the production of multiple bioproducts, as natural pigments of commercial interest, a biorefinery can use all of the biomass components and intermediates, there by maximizing the value derived from the biomass feedstock. In this sense, the aim of this work was to evaluate the chlorophyll production from *Phormidium autumnale* using agroindustrial wastewater as culture medium. The microalgae cultivation was conducted in a bubble column bioreactor, operating in batch system. The experimental conditions were: initial inoculum concentration of 100 mg/L (dry weight), pH of 7.6, temperature of 26 ºC at constant aeration of 1 VVM (volume of air per volume of medium per minute), in absence of light. The chlorophyll were extracted by ultrasound-assisted extraction (UAE), with a 13 mm diameter probe, dried samples, were placed in a jacketed vessel through which water was circulated at 20°C to avoid existence of hot spots, amplitude applied for extraction was set to 50% and samples were processed at a constant frequency of 20 kHz. The pigments were determined by high performance liquid chromatography coupled to photodiode array and mass spectrometry detectors (HPLC-PDA-MS/MS) on a C30 column. The chlorophyll industrial production was estimated based in industries of different capacities, operating 24 h per day and 336 days per year considering the microalgae biomass productivity (26.25 mg/L/h). Thirteen chlorophyll were separated in biomass from bioprocess for conversion of agroindustrial wastewater. The major cholorophyll were pheophytin a' (5363.81 μg/g), chlorophyll a (156.24 μg/g) and hydroxychlorophyll a' (33.96 μg/g). The results indicated that it is possible to produce 7,112.4, 71,124.5 and 711,244.8 ton biomass/year in a small (100 m^3/d), medium (1,000 m^3/d) and large industry (10,000 m^3/d), resulting in a total chlorophyll production of up to 702,240.0 kg/year. Individually, this represents a production of the majoritary chlorophylls: pheophytin a (2,586.3 to 258,634.9kg/year), chlorophyll a (1,110.2 to 111,024.1 kg/year) and hydroxychlorophyll a' (240.8 to 24,086.8 kg/year). Based on these results, we

observed the potential of *Phormidium autumnale* to the production of microalgal chlorophyll from agroindustrial wastewater.

Keywords: Microalgae; Chlorophyll; Wastewater; HPLC-PDA-MS/MS

P8 - POLYMER FILMS AND COATINGS CONTAINING ANTIMICROBIAL OLIVE LEAF EXTRACT FOR FOOD PACKAGING

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Abstract

The main objective of the project "EXTRUMiBi" (a CORNET project) was to develop a packaging solution for foods, that:

- extends the food shelf life, by the release of biosourced antimicrobial substances
- utilizes 100% biosourced material, i.e natural antimicrobial compounds and biopolymers
- is preferably produced by extrusion

Among the tested antimicrobial agents, an olive leaf extract (OLE), kindly developed and supplied by N-Zyme BioTec GmbH, Darmstadt, Germany, was selected because of its antimicrobial activity against *E. coli* DSM1576 (Gram -) and *Staph. aureus* (Gram +).

Lab and pilot scale batches of PLA and bio-based LDPE films containing OLE as an antimicrobial agent were produced both by extrusion and by coating. To protect OLE from high extrusion temperature, first trials were done with cyclodextrins encapsulated OLE.

Various analytical investigations were conducted to track the inclusion of two active antimicrobial species, oleuropein derivatives found in OLE, into the films. Their release in food simulants was monitored as well. Furthermore, films were characterized regarding their mechanical properties (e.g. elastic modulus and tensile strength, oxygen permeability, water vapor transition rate,…). The antimicrobial activity versus *E.coli* DSM 1576 was assessed following ISO22196:2011.

Coated films were produced using a lacquer composed of polyvinylacetate and OLE. Initial *E. coli* populations were significantly reduced (- 4.7 log) when 2 wt.% (dry mass) was added into lacquer. Total inhibition was observed at samples with 3 wt. % of OLE in the lacquer. Scale-up trials confirmed the potent antimicrobial behavior.

Extruded films were produced included 5 wt. % OLE in the blend, free in the case of PLA, or included in beta-cyclodextrin in the case of LDPE. However both films showed a lower antimicrobial behavior (<-2 log against *Staph. aureus*).

P9 - SEQUENTIAL BIOMASS VALORISATION OF HALOPHYTES: HIGH PRESSURE EXTRACTION PRIOR TO BIOGAS PRODUCTION

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Abstract

Salinization of soil is caused by global warming and over-cultivation of crops. Saline crops or halophytes are naturally adapted to salinity and can grow under these conditions in contrast to the majority of our crops. The need of biomass for the production of food, chemicals and energy is apparent, considering the increase in costs and unknown reserves of fossil petroleum and the increase in world population. However, due to the food vs. fuel debate, only biomass which cannot be used for food can be considered for chemical and energy production. In order to satisfy all requirements, within this Cornet project (called SALICHEM), we envision an extraction of high-value natural products before the residual plant material is used for energy production. In terms of biological activity, we focus on antimicrobial, anti-oxidant and anti-aging potency to cover the cosmetics, nutraceuticals and food industry.

Screenings of six halophytes were conducted based on secondary metabolites profiling, bioactivity, lignocellulosic composition for fermentation, and anaerobic digestion for methane production. *Spartina maritima* Fernald, giving the best results in terms of biogas production, was selected to maximize the valorization potential. *S. maritima* (Poaceae) is an herbaceous perennial cordgrass that grows along Europe's western and southern coasts. Its moderately and non-polar extracts showed promising anti-collagenase and anti-elastase activity. According to literature, anti-aging compounds are found in polyphenols and terpenoids family. The next step of the project is to optimize the extraction of the chosen extracts. Knowing that the SALICHEM project gives priority to green extraction, supercritical CO_2 and subcritical water were selected for the optimization of extraction. The anti-aging activity will be assessed during the optimization process.

The lignocellulosic pretreatment of the biomass is currently under investigation. In terms of biogas production, *S. maritima* showed similar results compared to grass silage, a typical feedstock for agricultural biogas plants.

Acknowledgements: We are grateful to the AiF and the Federal Ministry for Economic affairs and Energy of Germany, and the Walloon region of Belgium for supporting this research through the Cornet Program. We wish to thank Serra Maris bvba (Belgium) who generously provided the plant material.

P10 - DEVELOPMENT OF NEW GREEN PROTOCOLS FOR THE EXTRACTION OF POLYHYDROXYALKANOATES

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Abstract

The availability of new methodologies for achieving a sustainable and economic recovery of polyhydroxyalkanoates (PHA) from microbial cultures is a key aspect for the development of these promising bioplastics. The various downstream strategies developed up to now are characterized by various disadvantages and a general lack of sustainability such as the extensive use of non-recyclable/toxic solvents and chemicals [1]. Here two new protocols for the extraction of PHA from *Cupravidus necator* are proposed [2]: 1) the solubilization of PHA through green organic solvents; 2) the dissolution of the non-PHA cellular matrix through green surfactants. *The first* is based on the use of dimethyl carbonate (DMC), a biodegradable solvent, not harmful for humans and the environment, applicable to concentrated microbial cultures or dry biomass: in both cases the recovery of the polymer is very high (>85%), as well as its purity (>95%). Moreover, DMC does not cause any degradation/decomposition of the polymer. *The second protocol* is based on the use of Switchable Anionic Surfactants (SAS) [3], a peculiar class of surfactants, soluble in H2O at alkaline pH (condition at which they are anionic), but insoluble, and thus easily recoverable, at lower pH. The addition and removal of CO2 to the system represents a simple and effective way to achieve the pH switch. The ammonium carboxylate of lauric acid is able to afford an optimal polymer recovery (>99%) and purity (around 90%); almost complete SAS recovery (~95%) from the aqueous medium is achieved by lowering the pH with the addition of CO2. Therefore, both the protocols here developed resulted effective and, at the same time, sustainable: the recovery and the purity of the obtained PHA were very high, the use of toxic chemical compounds has been avoided, and the recycling and recovery of the various solvents/ surfactants used in the various stages was optimal.

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P11 - BIOACTIVE COMPOUND EXTRACTION FROM PLANT MATERIAL: GENOTYPE INFLUENCE ON HERBAL PREPARATION COMPOSITION BY HPLC FINGERPRINT

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Abstract

Medicinal plants and their preparations have been widely used for hundreds of years all over the world, both as single herbs or as a combination of several herbs in composite formulae, traditionally extracted in boiling water. Different bioactive compounds (botanicals) are present in these species and their derived-products as key components: all these molecules are known to be quite variable in the plant material, according to intraspecific chemodiversity, different harvest stage or cultivation area, and post-harvest handling. Most studies have focused on the use of plant parts for the extraction of phytochemicals and for the preparation of phytotherapic products. The aim of this work was to study the bioactive compound composition of eight tree-species bud-preparations (herbal preparations derived from embryonic fresh plant tissues as buds and sprouts), commonly used in phytotherapy. Molecules were extracted through a process of cold maceration for 3 weeks, in a solution of ethanol (95%) and glycerol. Innovative protocols was used to identify and quantify the main bioactive compounds (polyphenols, organic acids and vitamins), and to obtain a specific profile in order to assess the contribution of each single bioactive class to the total phytocomplex. A chemometric approach was used to distinguish among different genotypes assuring the identity, safety and quality of the botanical raw materials. The applied protocol was simple, sensitive and reliable and it could be used for the routine quality control of natural products. The approach represents a good tool to characterize the herbal preparations according to the utilized genotype, avoiding substitutions, changes or adulterations with other species or synthetic drugs.

P12 - MICROWAVE ASSISTED LIPID EXTRACTION FROM MICRO-ALGAE

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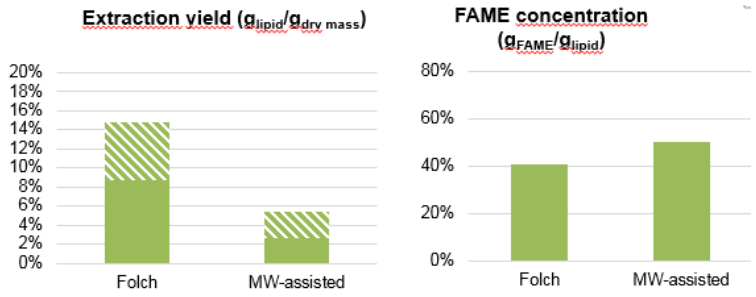
Abstract

Introduction. Increasing interest has been dedicated to the use of algal biomass for the production of energy through new technological and scientific platforms allowing the exploitation of the algal lipids as fuel, for food applications and in cosmetics. In this context, the development and optimization of sustainable processes in terms of environment, economy and energy for algae processing to obtain added value products is mandatory. Aim of this work is to evaluate the effectiveness of the process of microwave assisted extraction/*in-situ* trans-esterification on a microalgae population, able to minimize the use of solvents and maximize at the same time the extraction yield.

Materials & Methods. *Thalassiosyra weissflogii* microalgae population was selected as low demanding and with a very quick growth. Two extraction methods were tested, comparing standard Folch extraction method, considered as benchmark, with the microwave assisted technique, considering both yield of extraction and the Fatty Acid Methyl Esthers (FAME) content in the algae. Standard Folch method was performed, comprising a distillation with a mixture 1:2 of chloroform and methanol of the dried algal sample, followed by centrifugation, separation, washing with chloroform and subsequent evaporation. Results were compared with the Microwave-assisted method, which relies on the MW action to segregate the lipid phase in the algae cellular matrix. Sample was loaded in a cylindrical pressurized reactor and subject to a MW treatment for several minutes (P=1500W, T = 60°C, P = 300 bar) in presence of a mixture of hexane / methanol. Methanol was exploited also to promote the transesterification reaction during the extraction phase. After the treatment, elimination of the aqueous phase and organic waste was carried out. Parameters such as process duration, solvents quantity and solvents ratio were varied in order to optimize the process.

Results. Best extraction/transesterification results are shown in the graphs above, compared with the results from standard Folch approach. In particular, a ratio 1:8 of algae/solvents and a reaction time of 8 minutes gave an extraction yield of around 5%, having a FAME content of 50.7%, compared to an extraction yield of 15% for the Folch standard method, with the 40% of FAME in the extract.



The identified process represents a good compromise in terms of yield of extraction / solvents use / energy required. In particular allows a 50% saving of solvents and 20% saving of electric energy with respect to the standard Folch method.

Conclusion. Experimental campaign at lab scale was carried out to evaluate the effectiveness on the MW assisted extraction/in-situ transesterification technique on the algae samples. The process should be brought to an industrial scale and optimized for large biomass amount handling.

P13 - EVALUATION OF SOUR CHERRY POMACE AS A SOURCE OF BIOACTIVE COMPOUNDS

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Abstract

The production, processing and preservation of fruits generate a huge amount of waste, which currently are a burden on producers as the management of these wastes is becoming extremely difficult due to legislative restrictions on landfilling. However, fruit waste and by-products are an abundant source of valuable compounds, such as proteins, polysaccharides, fibers and above all antioxidant molecules, namely polyphenols, which may represent a considerable economic benefit for farmers and food chain stakeholders. The valorization of these residues represents a significant step towards the synthesis of valuable chemicals and materials to be further used in key sectors of the world economy. Sour cherry is a rich source of polyphenols, many of them located in the peels. Its processing by-products, mainly composed by peels and kernels, represent 15-28% of transformed raw material, depending on the process conditions. Starting from these considerations, the present study aimed to investigate the potentiality of sour cherry pomace as a value-added ingredient for the food and nutraceutical sectors. Dried pomaces from two organic sour cherry cultivars (cvs), Montmorency (M) and Bianchi d'Offagna (BO), an Italian native cv from Marche region, were evaluated for the nutritional and phytochemical content and antiradical potential. The content of phenolic compounds (TPC), flavonoids (FLC) and anthocyanins (TANC) was determined spectrophotometrically. The antiradical capacity of the analyzed samples was assayed by two in vitro tests (e.g. DPPH and ABTS). Results pointed out a strong influence of the cultivar on the parameters analysed, being BO one the richest in phytochemicals (TPC: 24 mg gallic acid equivalents g^{-1} DM; FLC: 11.31 mg catechin equivalents g^{-1} DM; TANC: 2 mg cyanidin-3-O-glucoside equivalents g^{-1} DM). Besides, the same samples showed the lowest level of ascorbic acid, measured by cyclic voltammetry (33 μg g^{-1} DM). Obtained results highlight the potentiality of sour cherry pomace as a high added value ingredient in the formulation of dietary supplements or new functional foods.

P14 - MODEL BASED SYSTEMATIC PROCESS DESIGN OF EXTRACTION AND PURIFICATION OF 10-DEACETYLBACCATIN III FROM TAXUS BACCATA L.

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Abstract

In food, fragrance and pharmaceutical industry, the market is growing for many years [1]. In order to meet this high demand in the future, improvements in extraction and purification steps of valuable compounds have to be done. 10-deacetlybaccatin III derived from European Yew (*Taxus baccata* L.), is used for the production of the anti-cancer drug Paclitaxel by semi-synthesis, which is only one example.

In this work, a methodical approach for extraction and purification of 10-deacetylbaccatin III as a typical example system is shown. A combination of rigorous modeling and the determination of physico-chemical data was found to be a promising approach. This procedure includes the use of the quantum-chemical model COSMO-RS [2] to determine the relevant physico-chemical data of pure substances [3], such as solubility. The physico-chemical data were not only used to design the extraction process but also to choose a suitable purification strategy. A valid rigorous model of the extraction process, based on the distributed plug flow assumption with a pore diffusion approach [4] was used for optimizing the extraction of 10-deacetylbaccatin III.

The model and experimental based process design will be shown and discussed. The new designed process is compared to an already patented one.

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P15 - COMPARISON OF VOLATILE FRACTIONS OBTAINED FROM *CISTUS CRETICUS* AND LABDANUM RESIN USING SUPERCRITICAL FLUID EXTRACTION AND HYDRODISTILLATION TECHNIQUES

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Abstract

Cistus creticus (Pink Rock-Rose) is a species of shrubby plant in the Cistaceae family. The genus *Cistus* L. comprises of 20 species distributed mainly in the Mediterranean area[1]. It is widely used as a decorative plant but it is also known for its ability to produce labdanum, a resin secreted from the glandular trichomes of its aerial parts. This resin contains a number of potent compounds with antioxidant, antibacterial, antifungal and anticancer properties. Moreover, in folk Medicine, herbal tea infusions and extracts have been used for the treatment of digestive problems and colds[2]. For this reason, the resin has been commercially available in Spain, Portugal, Morocco, Greece, Cyprus and a number of Arabic countries[3].

Both *C. creticus* and labdanum have been used by the perfume industry for the exploitation of odoriferous compounds. The recovery of these volatile constituents has been achieved by many techniques in the past, such as steam distillation, hydrodistillation (HD) and solvent extraction. However, because of the elongated extraction times, the high energy costs and the thermal degradation of sensitive compounds, alternative new technologies should be investigated. Among these, supercritical fluid extraction (SFE) seems to be a promising alternative for this purpose due to its advantages [4].

The aim of the study is the comparison of the essential oils and non-polar extracts obtained from the aerial parts of *C. creticus* and labdanum by hydrodistillation and SFE. The analysis (with GC-MS) revealed that the oils from SFE are superior, since less volatile compounds were detected in the HD preparations, due to thermal degradation. SFE essential oils are richer in volatile compounds that contribute to the smell of the oils and these aromas resemble closer to the natural smell of the plant.

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P16 - IMPROVEMENT OF ANTIOXIDANT ACTIVITY OF ESSENTIAL OIL OF BASIL, LEMON AND LEMONGRASS EXTRACTED BY HYDRODISTILLATION UNDER OXYGENATED ATMOSPHERE

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Abstract

Essential oils (EOs) are considered economically valuable products, being recognized by their biological and odoriferous properties. The use of these oils has been increased in food and flavoring industries, mainly due to the current demand from natural products. Chemical composition plays an important role into the properties of EOs, being generally oxygenated compounds the most actives. The influence of extraction under oxygenated atmosphere on EO quality from tree plants (basil, lemongrass and lemon) was evaluated. EOs were extracted by hydrodistillation during 3 h, using 50 g of plant and 500 ml distillated water, with and without oxygen flow (0.7 L min^{-1}). Quantification and identification of compounds were obtained by GC-FID and GC/MS, respectively, and antioxidant activity was evaluated by ORAC method. Extraction with oxygenated atmosphere reduced the yield of EOs of basil from 2.75% to 2.06% and lemon from 5.32% to 4.19%. The yield for lemongrass was 1.49% for both extractions. Oxygen addition in extraction influenced the chemical composition of EOs presenting in general higher amount of oxygenated compounds. An increase in linalool, camphor and eugenol concentration was observed for EOs extracted from basil under oxygenated atmosphere. For lemongrass and lemon EOs an increase of neral and geranial was observed, changing their sensorial behavior, because these compounds are important for citric note of these oils. Oxygenated atmosphere provided EOs with high antioxidant activity and for basil the ORAC values were increased from 2744 to 3357 µmol TE/g oil. For lemongrass the values were increased from 1277 to 1979 µmol TE/g oil and for lemon EOs from 120 to 414 µmol TE/g oil. This improvement in antioxidant activity is in agreement with the composition results, once oxygenated compounds were obtained in higher amount with extraction under oxygenated atmosphere. Therefore, it was possible to see that oxygenated atmosphere changed EOs composition, enabling a product with more oxygenated terpenes. In this way, odoriferous and most valuable compounds with higher antioxidant capacity could be obtained by the simple change of the atmosphere of extraction.

P17 - EFFECT OF ULTRASOUND PRE-TREATMENT WITH DEEP EUTECTIC SOLVENTS ON THE QUALITY OF ESSENTIAL OIL FROM *OCIMUM BASILICUM, CYMBOPOGON CITRATUS* AND *PERSIAN LIME* OBTAINED BY HYDRODISTILLATION

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Abstract

The search of natural compounds with antioxidant activity to scavenging free radicals and delay or inhibit the oxidative damage is an area of great interest. It is known that the quality of essential oils (EOs) depends on the extraction method selected and several alternatives to replace or improve conventional hydrodistillation could be found in literature. In this work, the effect of ultrasound pre-treatment with and without the use of deep eutectic solvents (DES) for further hydrodistillation extraction was evaluated. In this way, 50 mg of fresh aerial parts of basil (*Ocimum basilicum*), lemongrass (*Cymbopogon citratus*) and the peel of lemon (Persian lime) were previously ground in a knife mill and heated with 500 ml of distillated water or DES solution for extraction by hydrodistillation. DES was prepared with 250 ml of the mixture with citric acid: choline choride (1:1, v/v) in concentration of 0.5 mol L^{-1}. US treatment (25 kHz, 20 min by 80%) was performed with 250 ml of DES and before extraction 250 ml of deionized water was added. Extraction by conventional method take 3 hour for all samples, and only 1 h when a pre-treatment using ultrasound was used. EOs were analyzed by GC-FID and GC-MS and the antioxidant activity was measure by ORAC method. Different profile were observed according the sample; for basil, an increase on the antioxidant activity (from 2744 to 2920 μmol TE/g oil) was observed. This fact could be explained due the increase in the concentration of oxygenated compounds by US pre-treatment with DES, as alpha-terpineol (from 2.53 to 8.96%). For lemongrass, an increase on the antioxidant activity of 25% (from 1278 to 1601 μmol TE/g oil) was found. This could be explained by a slightly increase of oxygenated compounds (from 86.1 to 88.1%). For EOs from lemon, the antioxidant activity was increased using DES during the pre-treatment with ultrasound, and it could be related to the increase of terpinolene (from 0.72 to 2.41%), 4-terpineol (from 0.28 to 1.11%) and alpha-terpineol (from 0.48

to 4.34%). On the other hand, using only the ultrasound as pre-treatment the majority compounds with antioxidant activity did not presented important changes. Therefore, the use of DES could be an alternative to improve the extraction of EOs in order to obtain compounds more valuable or provide EOs with high antioxidant capacity.

P18 - ULTRASOUND ASSISTED EXTRACTION: TOWARDS A COMPREHENSION OF ULTRASOUND EFFECT ON THE VEGETABLE MATRIX. APPLICATION TO POLYPHENOL EXTRACTION FROM OLIVE LEAVES

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Abstract

The effectiveness of ultrasound in processing has been demonstrated by a great number of authors. More specifically, ultrasound is used for intensified natural product extraction (e.g. bioactive components such as essential oil, antioxidants, oil and dyes). Applied during extraction, ultrasound induce an increased extraction rate of monitored compounds compared to conventional extraction processes such as maceration.

Reported mechanisms for this mass transfer enhancement are generally attributed to the effect of cavitation bubbles generated in the extraction media. Toma *et al.* (2001) demonstrated that power ultrasound (frequency comprised between 20 kHz and 1 MHz) specifically impacted cellular structures such as excretion hairs of pot marigold. Our study aims at a going further in the understanding of mechanical effects induced by ultrasound on the vegetable matrix. Different effects were identified by reviewing literature on ultrasound assisted extraction of several raw materials (e.g. spinach, carvi seeds and apple pomace).

Ultimately, mechanical impacts of ultrasound will be specifically investigated by a multiscale approach on olive leaves. Effect of ultrasound has been assessed by submitting a single leaf maintained under an ultrasonic field (US probe, 20 kHz) at different treatment durations (5, 15, 30 and 60 min). Extraction was performed in an ethanol/water solvent (80/20, v/v), which is conventionally used to extract phenolic compounds. The media temperature was maintained at 25°C.

After sonication, the solvent and extracted compounds was recovered to determine the polyphenol content and profile. Treated leaves were studied by histology and leaf surface observations.

Reference

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P19 - GREEN EXTRACTIONS FROM POMEGRANATE JUICE BY-PRODUCTS AND THEIR POTENTIAL USE AS NATURAL FOOD/COSMETIC PRESERVATIVES AND/OR BIOACTIVE INGREDIENTS

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Abstract

The high demand for pomegranate products has caused a significant increase in pomegranate juice production and a consequent substantial production of waste to be disposed of. The aim of this study is to investigate the potential of retrieving polyphenolic antioxidants directly from wet pomegranate juice marcs, the fresh by-products obtained after pomegranate juice processing. These by-products consist mainly of internal membranes (endocarp) and of arils residues (including seeds) that remain after juice pressing, since in the classic production of pomegranate juice, exocarp (rind) and mesocarp (white spongy tissue) are preliminarily separated to avoid excessive turbidity and astringency of the juice itself. Membranes and arils residues are characterized by the presence of hydrolysable tannins, phenolic acids, ellagic acid, flavonoids that can be retrieved for nutraceutical and cosmeceutical applications.

In particular, green extraction technologies such as ultrasound assisted extraction (UAE) and microwave assisted extraction (MAE) were used both in parallel and in series. Water, as an environmental friendly extraction solvent, or solventless techniques have been employed. The results were compared with conventional extractions using a water bath and water as solvent. HPLC and costant-wavelength synchronous spectrofluorometry have been used to characterize the polyphenolic content of the extracts.

Total concentration of polyphenols and radical scavenging ability of the extracts have been determined in vitro by Folin-Ciocalteu and DPPH methods respectively, while the capacity of the extracts in inhibiting platelets aggregation has been tested ex-vivo on human platelets.

At the end, the most promising extract has been tested by Diffuse Reflectance UV–Visible (DRUV–Vis) spectroscopy to assess its efficacy in providing protection against oxidative browning both in foodstuffs and in cosmetic formulations.

P20 - A FULL SCALE ULTRASOUND SYSTEM FOR VIRGIN OLIVE OIL ELABORATION PROCESS

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Abstract

The aim of virgin olive oil extraction process is mainly to obtain the best quality oil from the fruits, applying only mechanical actions and guaranteeing the highest overall efficiency. Currently, the mechanical methods used to extract virgin oils from olives are basically of two types: the discontinuous systems (obsolete) and the continuous one. Anyway the system defined as "continuous" is composed by several steps not all completely continuous, due to the presence of the malaxer, a devices that works in batch. The aim of the paper was to design, realize and test the first full scale sono-exchanger for the virgin olive oil industry, placeable immediately after the crusher and before the malaxer. From a constructive point of view, the innovative device is composed by a triple concentric pipe heat exchanger combined with two ultrasound probes. This mechanical solution allows synergically to better destroy the cell walls freeing the olive droplets and the minor compounds and to accelerate the heat exchange between the olive paste and the process water. This strategy could represent the first step towards the transformation of the malaxing step from a batch operation into a real continuous process, improving the working capacity of the industrial plants. Considering the heterogeneity of the olive paste that is composed by different tissues, the design of the sono-exchanger required a fluid dynamic analysis. The thermal and the mechanical effects of the sono-exchanger were monitored measuring the temperature of the product at the inlet and the outlet of the device, and the concentration of chlorophylls in the product respectively. The effects of the innovative

process were evaluated in terms of extra virgin olive oil yields and quality, evaluating the main legal parameters, the polyphenol and tocopherol content.

P21 - GREEN SOLVENTS FOR EXTRACTION OF FLAVONOIDS FROM *NITRARIA RETUSA*: EXPERIMENTAL AND THEORETICAL STUDY USING HANSEN SOLUBILITY PARAMETERS

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Abstract

The present study was designed to evaluate the performance of seven green solvents or mixtures *ie.* bio-ethanol (EtOH); isopropanol (IPA), EtOH/H_2O (80/20; v/v), EtOH/H_2O (90/10; v/v); IPA/H_2O (80/20), IPA/EtOH (50/50; v/v) and IPA/EtOH (90/10; v/v) for the substitution of methanol, conventionally used in the extraction of phenolic compounds. This flammable and toxic organic solvent cause adverse health and environmental effects.

Firstly, solvent selection was made through the theoretical physicochemical solvent properties and solubility results obtained using Hansen Solubility Parameters (HSPs) for comprehension of the dissolving mechanism.

Secondly, the extracts were analysed to compare the solvents performance in terms of phenolic composition (total polyphenol content, total flavonoids content and condensed tannins content) and antioxidant activities (Total antioxidant activity and DPPH assay). The Hansen analysis indicates that the mixture IPA/EtOH (50/50; v/v) was the most suitable solvent for extraction of flavonoids from *Nitraria retusa*. However, the experimental study using a conventional solid–liquid extraction by maceration showed that the best green solvent was the mixture EtOH/H_2O (80/20; v/v) (Total antioxidant activity = 22.1 mg EAG/ g MS and DPPH test = 28.4µg/mL as compared to methanol: 28.2 mg EAG/ g MS and 26 µg/mL respectively).

Keywords: *Nitraria retusa*, green solvents, flavonoids, HSPs.

P22 - ESSENTIAL OIL AS SEED TREATMENTS AGAINST FUSARIUM WILTS OF LETTUCE AND BASIL

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Abstract

Essential oils from savory (*Satureja montana* L.) and thyme (*Thymus vulgaris* L.) were evaluated on basil cv. Italiano classic belonging to Genovese type seeds and lettuce cv. Crispilla seeds, as treatment against *Fusarium oxysporum* f. sp. *basilici* and *Fusarium oxysporum* f. sp. *lactucae* respectively. The efficacy of the treatments was assessed *in vivo* in glasshouse in comparison with thiram and prochloraz used as reference chemical treatments. The trials also assessed the fumigant effect of essential oils on the seed germination as well as on Fusarium wilt development. Essential oils seed treatment showed in most cases an efficacy limited but statistically comparable to that obtained with chemical treatments. The seedlings transmission of *F. oxysporum* f.sp. *lactucae* was significantly reduced to 83.4% by savory EO at 1%, that was as effective as prochloraz (91.7% of efficacy), while both the tested essential oils significantly reduced *F. oxysorum* f.sp. *basilici* symptoms with a reduction in the infection rate statistically similar to the chemical references. Concentration and interaction among principal compounds may determine the antifungal activity of an essential oil. The essential oils seed treatments were non-phytotoxic and did not negatively affect the germination rate of the treated seeds.

P23 - ADSORPTION KINETIC AND ISOTHERMS OF (+)-CATECHIN ON HIGHLY CROSS-LINKED 12% AGAROSE GEL

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Abstract

In the last decade, adsorption chromatography on highly cross-linked 12% agarose gel appears as an attractive, innocuous, environmentally friendly and scalable method for polyphenols purification (1). However, although there are some studies on polyphenols purification in column packed with highly cross-linked 12% agarose gel (1), there are no studies regarding adsorption process (adsorption isotherms, thermodynamics and kinetics) of polyphenols on this gel. The objective of this work is to present a first approach to the characterization of the adsorption process of polyphenols on 12% agarose gel. (+)-catechin was used as a case of study to determine both the adsorption kinetics [30 mg (dry basis) of 12% agarose gel were combined with 30 mL of (+)-catechin solution (600 mg/L) and continuously stirred (300 rpm) at 20°C, taking 1 mL of solution at different time intervals] and adsorption isotherms at 20, 30 and 40°C [10 mg (dry basis) of 12% agarose gel were combined with 10 mL of (+)-catechin solutions at different concentrations (100-1000 mg/L) and continuously stirred (300 rpm) for 12 h]. At the condition tested, the adsorption equilibrium is reached rapidly (about 15 minutes). In addition, adsorption kinetics data was properly fit (R^2=0.9951) to pseudo first-order kinetics model [$\ln(q_e-q_t)=\ln(q_e)-k*t$], obtaining the parameters k=0.8 (1/min) and q_e= 335 [mg of (+)-catechin solution/g agarose gel (dry basis)]. The adsorption capacity of 12% agarose gel increases with increasing temperature, reaching a maximum at 40°C with a value of 409.0 [mg of (+)-catechin solution/g agarose gel (dry basis)]. According to results, Freundlich equation ($q_e=K_f*C_e^{(1/n)}$) was more adequate than Langmuir equation for describing the polyphenol adsorption system, with R^2 values of 0.9873, 09917 and 0.9904 for isotherms at 20, 30 and 40°C, respectively. The parameters of Freundlich equation, K_f and n, range from 26.41 to 30.47 (mL/mg) and 2.32 to 2.57, respectively.

Acknowledgments: The authors would like to acknowledge the financial support from FONDECYT/Postdoctoral Project 3150622 from CONICYT-Chile.

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P24 - ENZYME-ASSISTANT EXTRACTION OF PHLOROTANNINS FROM BROWN SEAWEED

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Abstract

Seaweeds are a good source of chemicals and building block materials that can be tailored through proper biorefining processes. Amongst the compounds that have a commercial interest are the phlorotannins. Phlorotannins are a type of polyphenol that are found exclusively in brown seaweeds. They exhibit a variety of different biological properties, including antioxidant, anti-inflammatory, antiallergic and neuroprotective. The objective of this work was to improve the extraction of phlorotannins from brown seaweed *Macrocystis pyrifera* using enzymatic hydrolysis as pre-treatment. The conditions of pre-treatment were optimized evaluating simultaneously several variables: time (12, 24 and 36 h), temperature (25, 37 and 50°C), pH (4.5, 55 and 7.0) and algae to enzymatic extract ratio (1/10, 1/20 and 1/30) using Taguchi experimental design. A set extracellular carbohydrate active enzymes (alginate lyase, Fucoidanase and 1,3-β-D-glucanase) produced by marine *Alternaria sp*. (accession N°: KU163454) was used as enzymatic extract. Finally, for each liquid fraction obtained in each dot (extracts) the total concentration of protein was determined using the Folin-Ciocalteu method and the antioxidant activity of the extract by the method of radical DPPH. The optimal conditions of pre-treatment were 25°C, pH 7.0 by for 36 hours and algae to enzymatic extract ratio of 1/20 which allowed the obtention of an extraction yield of phlorotannins of 2.14±0.25 wt.%. Additionally, the statistical analysis showed that incubation time had a significant effect ($p<0.05$) on phlorotannins extraction, 46% and that the optimal pre-treatment conditions predicted were 24 h incubation at 50°C, pH 7.0 and algae to enzymatic extract ratio ratio of 1/10 obtaining under these conditions a maximum extraction yield of 3.31 wt.% of phlorotannins. Future work includes the use of the extract of phlorotannins in nutraceutical platforms.

Grant support: CONICYT (Project AKA-ERNC 0009), CeBiB (Project FB-0001) and The Academy of Finland (Grant N°: 125113 and 138448). We are thankful to Dr. Buschmann (University of Los Lagos) for providing us the algae (*M. pyrifera*).

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