Valorisation des coproduits agroalimentaires grâce à la bioraffinerie et l’éco-extraction du végétal

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Fields of Application
Cosmetics, food, pharmaceutical, perfumes, biofuel industry......

Equipment
Batch and continuous ultrasonic devices for lab and pilot scale
Lab and pilot microwave ovens. Microwave autoclave
Analysis Techniques : GC/FID, HPLC/DAD, HP-TLC

Missions
Green extraction of natural products on lab and pilot scale to approach an optimal consumption of raw materials, solvents and energy:
(1) improvement and optimization of existing processes,
(2) using non-dedicated equipment,
(3) innovation in processes and procedures but also in discovering alternative solvents.
Part. 1: Concepts and Principles of Eco-Extraction of Natural Products.

Part. 2: Enrichment of Edible Oil with Sea Buckthorn By-Products Using Ultrasound Assisted Extraction

Part. 3: Valorization of Ginger By-Products: A Bio-Refinery Concept

Part. 4: Valorization of Food By-Products

Part. 5: Production of Aromatic Extracts from Fruits By-Products: Natarome Project
Part. 1: Concepts and Principles of Eco-Extraction of Natural Products.
PLANT EXTRACTION

PERCOLATION
MACERATION
DECOCTION
INFUSION
HYDRODISTILLATION

Plant material → Contact → Filtration → Centrifugation → Plant Extract

HERBS, SPICES
FRUITS, FLOWERS
VEGETABLES, ..... COSMETICS
FOOD
FRAGRANCES
PHARMACEUTICAL,....
Extraction of Natural Products: Industrial Problems

- Extraction time
- Problems of degradation
- Energy cost
- « Batch » Extraction
- Use of solvents from fossil resources
- Need for new products
- Reduction of waste: solid and liquid

Request from Industry: Room temperature, rapid extraction, without solvent or water, eliminate wastes, continuous process, and competitive in price and quality.
Green Extraction

Six Principles of Green Extraction

1. Intensive extraction
   - Overexploitation of plant resources
   - Towards renewable resources,
   - Varietal selection.

2. Old methods, expensive process...
   - Reduce operation units
   - Safe, robust and controlled processes

3. Contaminated extracts, not comply with the regulations
   - Obtain a non-denatured and biodegradable extract with “green” values instead of contaminants

4. Large quantity of residues generated by the extraction
   - Valorisation of those co-products:
     - Identification and characterization
     - Harvest conditions
     - Valorisation opportunities

5. Important energy consumption linked to the extraction
   - Optimisation and/ or assistance of existing processes,
   - Reduce energy consumption,
   - Using innovative technologies.

6. Massive use of petro-chemical solvents
   - Set up of REACH

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Eco-Extraction

Principe 1
The plant

Principe 2
Solvent

Principe 3
Energy

Principe 4
Co-products

Principe 5
Process

Principe 6
Extraction
PARTIE 2: ENRICHMENT OF EDIBLE OIL WITH SEA BUCKTHORN BY-PRODUCTS USING ULTRASOUND ASSISTED EXTRACTION


Research Article

Enrichment of edible-oil with sea buckthorn by-products using ultrasound-assisted extraction

Farid Chemat¹, Sandrine Périso-Issartier¹, Lynda Loucif¹, Mohamed Elmaataoui¹ and Timothy J. Mason²
Processing of sea buckthorn berries

Whole fruits → Hydraulic Press → Presse cake with residual carotenoids

Enrichment in residual carotenoids of sunflower oil: to produce a high-value oil
Ultrasound assisted extraction (UAE)

- **Design of experiment – Central Composite Face-Centered design**
  - Study of ultrasonic intensity and temperature
    - To improve the direct enrichment of edible oil with SBT carotenoids
    - To determine the optimal conditions of UAE.

**OPTIMUM**

- **Power**: 0.67 W.g\(^{-1}\), **Temperature**: 40 °C

**Response**:
- Carotenoids content

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- Ultrasound-assisted extraction
- Conventional extraction
- Thermostated reactor
- Oil
- SBT by-product
- Magnetic stirred
**Conventional process intensification using Ultrasound**

Extraction kinetics of SBT by-products with and without US

![Graph showing extraction kinetics](image)

<table>
<thead>
<tr>
<th>Extraction Time (min)</th>
<th>CSE</th>
<th>UAE</th>
</tr>
</thead>
<tbody>
<tr>
<td>β-carotene (mg/L extract)</td>
<td>14,58</td>
<td>18,06</td>
</tr>
<tr>
<td>Caroténoïdes totaux (mg/L extract)</td>
<td>52,65</td>
<td>63,84</td>
</tr>
<tr>
<td>Temps d’extraction (min)</td>
<td>120</td>
<td>30</td>
</tr>
</tbody>
</table>

Extraction of β-carotene was enhanced by the procedure as shown in this table.
PART 3: VALORIZATION OF GINGER BY-PRODUCTS: A BIO-REFINERY CONCEPT
Context

- Ginger production in 2015: 2,140,451 t (FAOSTAT 2015)
- Valorization of press cakes considered as waste in high valued compounds

**Diagram**

- **Fresh ginger rhizomes** → **Juice processing** → **Ginger press cake** → **Ginger juice**
- **Ginger press cake**
  - **Essential oil**
    - zingiberene
    - α-curcumene
  - **Phenolics**
    - 6-gingerol
    - 6-shogaol
  - **Fibers**
Concept developed in the study

- Ask from industry: rapid, cold and organic solvent-free extraction to obtain high quality product, competitiveness

$\xrightarrow{\text{Process}}$ Press cake $\xrightarrow{\text{MHG}}$ Solid residue $\xrightarrow{\text{UAE}}$ Fibers

$\xrightarrow{\text{Juice}}$ Essential oil $\xrightarrow{\text{“In situ” water}}$ UAE

- A “dry” bio-refinery concept

MHG: Microwave Hydrodiffusion and Gravity
UAE: Ultrasound Assisted Extraction

Gingérol, 6-shogaol
1st step: Microwave Hydrodiffusion and Gravity (MHG)

OPTIMIZATION OF MICROWAVE (MW) POWER
Microwave Hydrodiffusion and Gravity (MHG)

Patented Chemat et al., 2008
1st step: MHG assessment

Press cake
1.5 kg
4.8 g phenolics

MHG
20 min
1.6 W/g

Essential oil
3 mL

“In situ” water
750 g (50% yield)
0 g phenolics (0% yield)

Solid residue
720 g (48% yield)
4.8 g phenolics (100% yield)
2nd step: Ultrasound Assisted Extraction (UAE)

- Rhizomes → Process → Press cake → MHG → Solid residue → UAE → Fibers
- Juice → Essential oil → Phenolics
- "In situ" water
Ultrasound Assisted Extraction (UAE)

- Effect of ultrasonic intensity on **extraction yield** assessed (from 4.4 W/cm² to 16.7 W/cm²)
- Reference: conventional maceration (CM)

From 3 to 500 L
2nd step: UAE assessment

"In situ" water

Solid residue
720 g
4.8 g phenolics

UAE
90 min
UI 16.7 W/cm²

Evaporation
Aqueous extract
DW = 68 g
1.2 g phenolics (26 % yield)

Drying
Residue
DW = 307 g
3.6 g phenolics (74 % yield)
Process assessment of “dry” and conventional bio-refineries

→ Evaluation according to the six principles of eco-extraction

<table>
<thead>
<tr>
<th>Principle</th>
<th>Evaluation</th>
</tr>
</thead>
<tbody>
<tr>
<td>1. Raw material</td>
<td>% of valorized raw material from food processing industry</td>
</tr>
<tr>
<td>2. Solvent</td>
<td>mass ethanol / total mass of solvent used in the bio-refinery (%)</td>
</tr>
<tr>
<td>3. Energy</td>
<td>mass waste / total mass of solvent + raw material used in the process (%)</td>
</tr>
<tr>
<td>4. Process</td>
<td>extraction duration for the bio-refinery (min)</td>
</tr>
<tr>
<td>5. Product recovery</td>
<td>mass of final product recovered / mass of available product in the plant material</td>
</tr>
</tbody>
</table>

**Conventional bio-refinery:** hydrodistillation + ethanolic extraction
“Dry” bio-refinery: MHG + UAE

→ Reduction of footprint with the “dry” bio-refinery
PART 4: VALORIZATION OF FOOD BY-PRODUCTS
Valorisation of food by products (lettuce) using microwave energy

Laboratory to pilot scale: Microwave extraction for polyphenols lettuce

Sandrine Périso, Jean T. Pierson, Karine Ruiz, Giancarlo Cravotto, Farid Chemat

1 Université d’Avignon et des Pays de Vaucluse, INRA, UMR406, GREEN Extraction Team, F-84000 Avignon, France
2 Dipartimento di Scienze e Tecnologie del Farmaco, Università di Torino, Via P. Giuria 5, 10125 Torino, Italy
An innovative grape juice enriched in polyphenols by microwave-assisted extraction
Flavoring vegetable oils by microwave

Flavor + olive oil ➔ MW ➔ Flavored oil
PART 5: PRODUCTION OF AROMATIC EXTRACTS FROM FRUITS BY-PRODUCTS:
NATAROME PROJECT (2015-2019)
Natarome project

- Develop new intermediate food products "clean label" that integrate technology functions previously made by the additives (flavors, textures and color)

- 5 Industrial partners:
  - SENAGRAL
  - AGRO'NOVAE
  - Les Comtes de Provence
  - ATELIER DU FRUIT
  - JEAN NIEL
  - PROTEUS

- 2 Academic partners
  - UNIVERSITE D'AVIGNON
  - INRA

- Duration: 42 months
- Budget: 2,6 M€
- Consequences: 12 M€ turnover by 2019 and over 63 million after 5 years of operation
- 35 direct jobs in France
Natarome project

Eco-Extraction

Enzyme

Jam

Fruit drink

Flavored yoghurt

Ice
Thank you for your attention