Extraction of lipids and bioactive molecules from coffee silverskin (WP1)
Summary

- Silverskin composition
- Aims of WP1
- Extraction techniques
- Preliminary results
- Future perspectives
Coffee Silverskin (CS) composition

- 73 articles on CS since 1997
- In the last decade CS became a hot topic
- First paper about CS composition published in 2004

Source: ISI web of knowledge
Coffee Silverskin (CS) composition

CS composition from mix of Arabica and Robusta coffee

- **70-80% of dietary fiber** (15% soluble dietary fiber and 85% insoluble dietary fiber) such as hemicellulose, cellulose, lignin, oligosaccharide, polysaccharide, pectines, gum and waxes.
- **16.2-19% protein**
- **2-7% fat** (60.6% saturated fatty acid and 39.4% unsaturated fatty acids)
- **0.81-1.4% caffeine**
- **3.6% total phenolic compounds** (CGAs)

The constituents of CS can be considered bioactive compounds conferring to this by-product antioxidant and prebiotic properties

**Currently use of CS:** as direct fuel (e.g. firelighters), for composting and soil fertilization

**Future use of CS:**

- Source of cellulose for paper production
- Source of bioactive compounds (polyphenol, caffeine, fiber) to employ as costituens of pharmaceutical/cosmetic produts-functional foods

Y. Narita et al. Food Res. Int. 2014, 61, 16-22
Borrelli et al., 2004; Costa et al., 2017
Coffee Silverskin (CS) composition

CS composition of *Arabica/Robusta* coffee from six different geographical origin

<table>
<thead>
<tr>
<th>Geographical origin</th>
<th>Nutritional composition (%)</th>
<th>Dietary fiber</th>
<th>Available carbohydrates</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Moisture</td>
<td>Ash</td>
<td>Fat</td>
</tr>
<tr>
<td>Cameroon</td>
<td>9.91 ± 0.53&lt;sup&gt;a&lt;/sup&gt;</td>
<td>8.31 ± 0.08&lt;sup&gt;d&lt;/sup&gt;</td>
<td>1.81 ± 0.03&lt;sup&gt;d&lt;/sup&gt;</td>
</tr>
<tr>
<td>India</td>
<td>10.3 ± 0.20&lt;sup&gt;a&lt;/sup&gt;</td>
<td>7.34 ± 0.03&lt;sup&gt;e&lt;/sup&gt;</td>
<td>1.19 ± 0.05&lt;sup&gt;e&lt;/sup&gt;</td>
</tr>
<tr>
<td>Indonesia</td>
<td>9.28 ± 0.75&lt;sup&gt;a&lt;/sup&gt;</td>
<td>8.71 ± 0.04&lt;sup&gt;e&lt;/sup&gt;</td>
<td>2.46 ± 0.07&lt;sup&gt;e&lt;/sup&gt;</td>
</tr>
<tr>
<td>Brazil</td>
<td>9.53 ± 1.2&lt;sup&gt;a&lt;/sup&gt;</td>
<td>10.4 ± 0.02&lt;sup&gt;a&lt;/sup&gt;</td>
<td>3.15 ± 0.07&lt;sup&gt;b&lt;/sup&gt;</td>
</tr>
<tr>
<td>Vietnam</td>
<td>9.55 ± 0.64&lt;sup&gt;a&lt;/sup&gt;</td>
<td>9.29 ± 0.01&lt;sup&gt;b&lt;/sup&gt;</td>
<td>2.27 ± 0.13&lt;sup&gt;bc&lt;/sup&gt;</td>
</tr>
<tr>
<td>Uganda</td>
<td>9.35 ± 0.64&lt;sup&gt;a&lt;/sup&gt;</td>
<td>10.5 ± 0.10&lt;sup&gt;a&lt;/sup&gt;</td>
<td>1.86 ± 0.03&lt;sup&gt;cd&lt;/sup&gt;</td>
</tr>
</tbody>
</table>

Data are presented as mean ± standard deviation. Different letters within each column represent significant differences at p < 0.05.

The fatty acid composition, followed by the antioxidant profile, had a major role in the discrimination of CS of the same coffee species, but from different geographical origins.
Aims of WP1: work flow

- **Step 1**: extraction of fatty acids (FA)
- **Step 2**: extraction of caffeine
- **Step 3**: extraction of polyphenols

Three-step process:
- Partially or total delipidisation of CS to make paper (WP4) to be derivatized (WP2)

Additional aims:
- Selective extractions of fatty acids, caffeine and chlorogenic acids from biomass
- One-pot sequential extractions
- Valorization of mixed fractions
- Characterization of components by GC-FID, HPLC-DAD/MS, NMR

DEPARTMENT OF ENVIRONMENTAL SCIENCE AND POLICY
Extraction techniques

**Conventional**
(i.e. soxhlet, percolation, maceration)

**Not conventional**
(i.e. MicroWave, ultrasound, Pressurize Liquid Extraction, enzyme-based extraction)

- Not selective
- Expensive
- Not eco-friendly

**Supercritical fluid extraction (SFE)**

SFE employs carbon dioxide (CO₂) at its supercritical conditions (T 31°C, 73 atm) as extractor fluid ensuring a safe, not expensive and eco-friendly extractions.

- Selective (co-solvent)
- No degradation of thermolabile substances

*Key parameters:* **temperature, pressure, particle size of substrate** that can influence the external, internal and diffusional mass transfer

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Couto R.M et al. *J. Supercri. Fluids* 2009, 51, 159-166
Step 1: extraction of fatty acids (FA)-preliminary results

**Soxhlet extraction:**
10 grams, 60-80°C, n-hexane, 3-8 h

**GC analysis:** extracts were transesterified in n-hep using 2M KOH/MeOH, r.t.
Then they were analysed by GC-FID (column: SP-2560 non-bonded poly(biscyanopropyl) siloxane, 100 mt)

### Chemical profile of FA

<table>
<thead>
<tr>
<th>Matrix</th>
<th>FA composition (%mol)*</th>
<th>Yield (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>C16:0</td>
<td>C18:0</td>
</tr>
<tr>
<td>SK0</td>
<td>30.2</td>
<td>6.6</td>
</tr>
<tr>
<td>SK1</td>
<td>26.9</td>
<td>6.4</td>
</tr>
<tr>
<td>SK2</td>
<td>29.0</td>
<td>6.8</td>
</tr>
</tbody>
</table>

*Data provided by ISTM-CNR, Milan

- All extracts show a similar FA profile
- Behenic acid (C22:0) is not present in spent coffee ground
Step 1: extraction of fatty acids (FA)

No literature data reported the application of SFE on CS, only on spent coffee ground.

A preliminary analysis was carried out at Supercritical fluid technologies laboratories (USA) on 50 grams of SK0.

The same experimental conditions were successively applied to SK1 and SK2.

Form left to right:
SFT-10, (SF pump), SFT-110 XW,(SF extractor),
Supercritical Fluid Technologies, inc, Newark, USA
Secondary pump 515, Waters, USA

The instrument is present at Department of Environmental Science and Policy of the University of Milan.
Conclusions and perspectives

- Modulation of some parameters (i.e. temperature and pressure) can lead to a selective extraction of fatty acids.
- Addition of co-solvent will be optimized.
- Necessity to work on unique biomass in order to have comparable results.

Optimization of extraction conditions \textit{via} Design of Experiments (DoE)

To construct a mathematical model for postulating the extraction profile of bioactive molecules useful for industrial scale-up (\textit{i.e} Del Valle, Sovová, Martinez)