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Characterization of complex phenolic compounds in rapeseed and sunflower biomass generated during biorefinery

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Introduction
In addition to lipids, proteins and polysaccharides, rapeseed and sunflower seeds are known to contain significant amounts of phenolic compounds. They are located both in the kernels and in their fibrous hulls. After oil production, valorization of those phenolic compounds can be considered due to their putative health benefits. Previous studies reported that the concentration of some complex polyphenols such as condensed tannins in rapeseed hulls tends to decrease with advanced plant maturity. They may be oxidized or form strong interactions with other polymers in the plant (proteins, polysaccharides). Our work aims at evaluating the applicability of acidolysis methods (i.e. direct phloroglucinolysis and butanol-HCl acidolysis) for determining complex phenolic compounds in rapeseed and sunflower biomass (oil cake and hull).

Main phenolic compounds in the rapeseed

**Kernel**: sinapine, sinapic acid and sinapoyl glucose

**Hull**: condensed tannins & flavonols

Main phenolic compounds in the sunflower

**Kernel**: chlorogenic acids

**Hull**: few information (Possibly "oxidized polyphenols")

Materials and methods

<table>
<thead>
<tr>
<th>Rapeseed</th>
<th>% oil</th>
</tr>
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<tbody>
<tr>
<td>Seed</td>
<td>47.1</td>
</tr>
<tr>
<td>Oil cake</td>
<td>1.5-2.3</td>
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<tr>
<td>Hull</td>
<td>23.4</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Sunflower</th>
<th>% oil</th>
</tr>
</thead>
<tbody>
<tr>
<td>Seed</td>
<td>47.7</td>
</tr>
<tr>
<td>Oil cake</td>
<td>2.2</td>
</tr>
<tr>
<td>Hull</td>
<td>6.3</td>
</tr>
</tbody>
</table>

Seed, oil cake, hull of rapeseed/sunflower Delipidation

**Phloroglucinolysis reaction**

**Analysis by Ion Trap LC-MS**

**Butanol-HCl reaction**

**Analysis by colorimetry**

Complex phenolic oxidation products

**Oligomers**

**Procyanidins**

**Polymers**

Results

- For rapeseed hull, the reaction products of butanol-HCl assay present spectral profile close to cyanidin (confirmed by LC-MS)
- Presence of condensed tannins
- The phloroglucinolysis assay confirm the presence of procyanidin in the rapeseed hull.
- Less or no condensed tannins were detected in rapeseed oil cakes and in sunflower samples.

Conclusions

- After delipidation, phloroglucinolysis or butanol-HCl reaction can be applied directly on rapeseed or sunflower samples for the determination of complex phenolic compounds (e.g. condensed tannins).
- Contrary to rapeseed hulls, few condensed tannins were detected in rapeseed oil cakes and in sunflower samples.
- Further work will be done to improve the estimation of oxidized polyphenolic compounds.

Acknowledgement

This work was performed, in partnership with the SAS PIVERT (PHENOLEO Project), within the frame of the French Institute for the Energy Transition (Institut pour la Transition Énergétique - ITE) PIVERT (www.institut-pivert.com) selected as an Investment for the Future ("Investissements d'Avenir"). This work was supported, as part of the Investments for the Future, by the French Government under the reference ANR-09-IAH.