Analysis of complex phenolic compounds in rapeseed by optimised phloroglucinolysis reaction

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P2M2 Platform

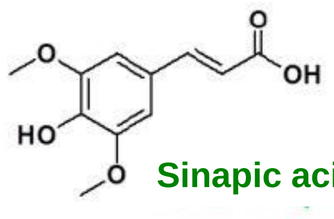
Introduction

Rapeseed
(*Brassica napus* L.)
(Image source : Terres Inovia)

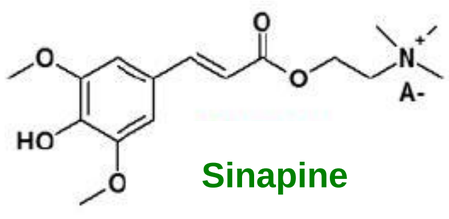
Rapeseed kernel and hull contain important value-added products such as phenolic compounds. They can find applications directly or after modifications in many fields such as food, health and cosmetic due to their antioxidant properties. Extraction and analysis of simple polyphenols in rapeseed such as phenolic acids and flavonols have already been widely studied. However, complex phenolic compounds such as condensed tannins in particular those having high molecular weight (polymerized or oxidized) still remain largely unexplored because they are difficult to extract in general solvents. Our work proposes a phloroglucinolysis method optimised by Response Surface Methodology (RSM) for the analysis of complex phenolic compounds in rapeseed. Only reagents with relatively low environmental impact are required which could make this method feasible to be integrated into the valorization process of rapeseed biomass.

Main phenolic compounds in the rapeseed

Kernel : Sinapic acid and its derivatives (sinapine, sinapoyl glucose)



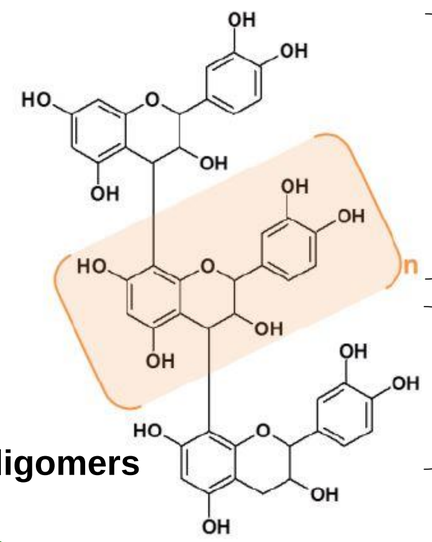
Sinapic acid

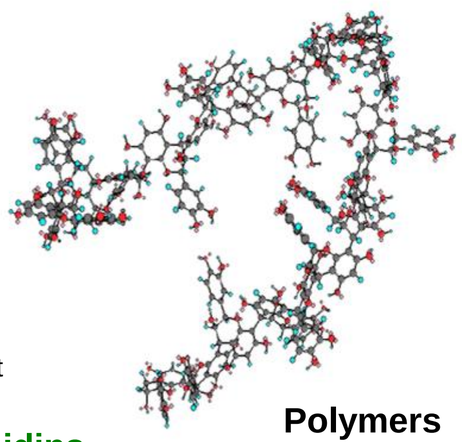


Sinapine

Hull : Complex tannins (polymerized proanthocyanidins) & flavonols

(Auger et al. 2010)





Procyanidins

Polymers

Materials and methods

Rapeseed hull → Delipidation → Phloroglucinolysis reaction → Analysis by Ion Trap LC-MS →

$$mDP^* = \frac{[EC\text{-}PLG]+[CAT]+[EC]}{[CAT]+[EC]}$$

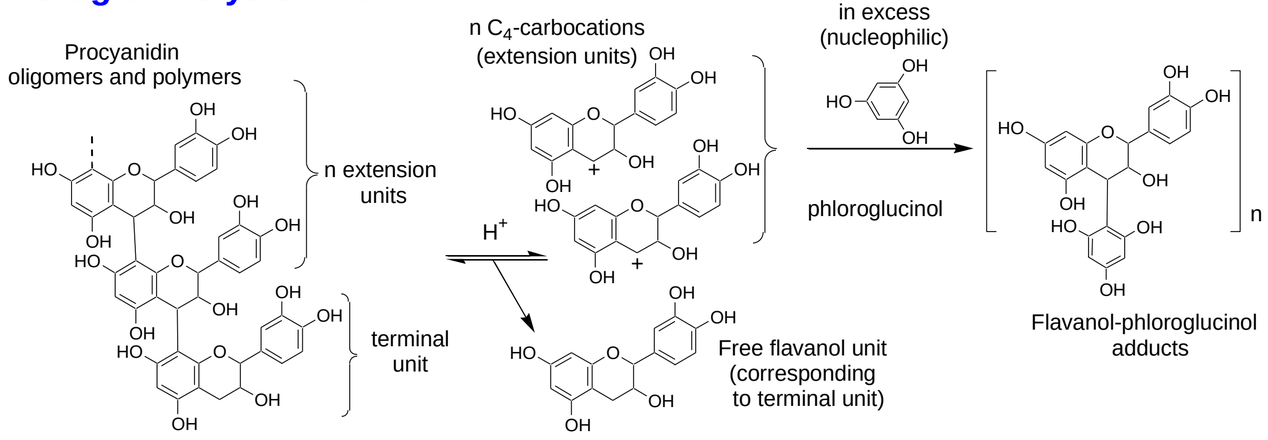
mDP: mean degree of polymerization

EC-PLG: adduct (extention unit + phloro)

CAT: catechin after reaction (terminal units)

EC: epicatechin after reaction (terminal units)

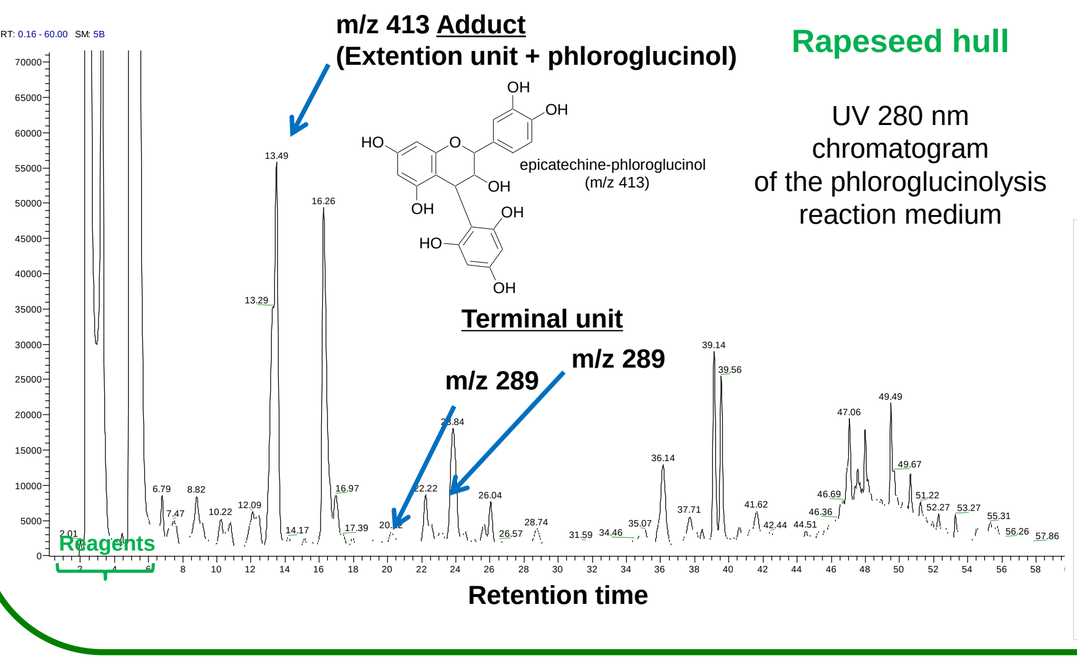
Phloroglucinolysis reaction



Experimental values and coded levels of the variables used for the CCD

Coded levels	Uncoded levels		
	Incubation temperature (°C)	Hydrochloric acid concentration (N)	Incubation time (min)
-1.68179	29.7	0.13	19.5
-1	40	0.4	40
0	55	0.8	70
+1	70	1.2	100
+1.68179	80.2	1.47	120.4

Results

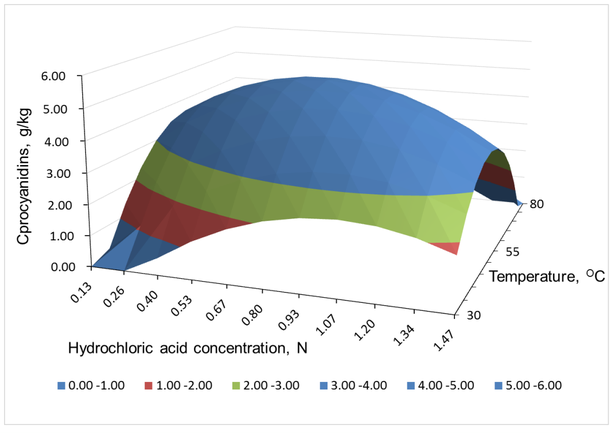


Rapeseed hull

UV 280 nm chromatogram of the phloroglucinolysis reaction medium

❖ A significant ($p < 0.0001$) second order polynomial model was determined to study the main and quadratic effects of incubation temperature (°C) (X_1), acid concentration (N) (X_2) and incubation time (min) (X_3) on the estimation of procyanidins in rapeseed hull (g/kg) (Y) and the first order interaction between the factors :

$$Y = 5.23 - 1.11X_1^2 - 0.64X_2^2 - 0.52X_1X_2 - 0.65X_1X_3$$



❖ The maximum estimation of procyanidins in rapeseed hull (5.40 g/kg) was predicted at moderate temperature (60 °C) with moderate acidity (0.8 N) during 30 min.

❖ Some oxidation markers (*m/z* 699) were found in the optimized phloroglucinolysis reaction products revealing the presence of oxidation bonds inside of the procyanidin structures.

Conclusions

✓ *The analysis of procyanidins in rapeseed hull by phloroglucinolysis reaction could be optimised by RSM using an adequate derived model.*

✓ *Further work will be done to improve the estimation of oxidized procyanidins and other complex phenolic compounds in rapeseed .*

Acknowledgement

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