



HAL
open science

Innovative Coupling of Supercritical Carbon Dioxide and Water for the Recovery of Lipids and Polyphenols from Spent Coffee Grounds

Alexandre Vandepoosele, Damien Bernard, Micheline Draye, Christine Piot, Gregory Chatel

► **To cite this version:**

Alexandre Vandepoosele, Damien Bernard, Micheline Draye, Christine Piot, Gregory Chatel. Innovative Coupling of Supercritical Carbon Dioxide and Water for the Recovery of Lipids and Polyphenols from Spent Coffee Grounds. French-Chinese Conference on Green Chemistry, Oct 2021, Lyon, France. , 2021. hal-03425615

HAL Id: hal-03425615

<https://hal.science/hal-03425615v1>

Submitted on 10 Nov 2021

HAL is a multi-disciplinary open access archive for the deposit and dissemination of scientific research documents, whether they are published or not. The documents may come from teaching and research institutions in France or abroad, or from public or private research centers.

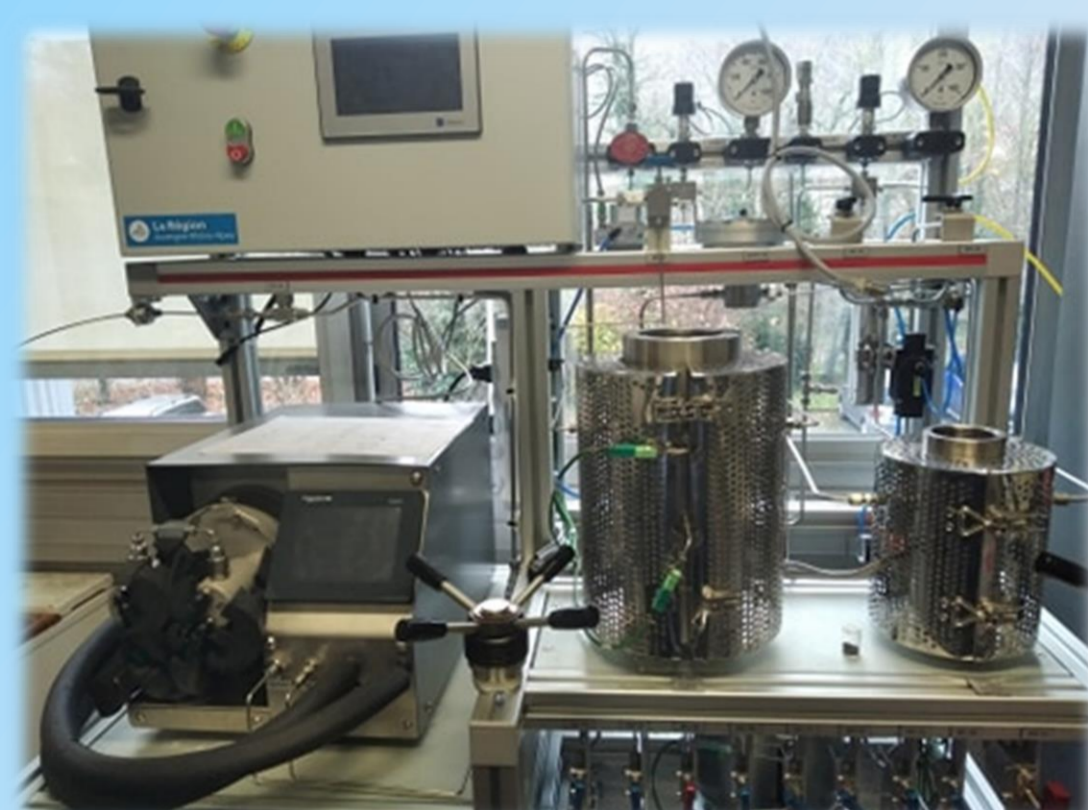
L'archive ouverte pluridisciplinaire **HAL**, est destinée au dépôt et à la diffusion de documents scientifiques de niveau recherche, publiés ou non, émanant des établissements d'enseignement et de recherche français ou étrangers, des laboratoires publics ou privés.

Innovative Coupling of Supercritical Carbon Dioxide and Water for the Recovery of Lipids and Polyphenols from Spent Coffee Grounds

Alexandre VANDEPONSEELE, Damien Bernard, Micheline DRAYE, Christine PIOT and Gregory CHATEL*
 Univ. Savoie Mont Blanc, CNRS, EDYTEM, F-73000 Chambéry, France.
 alexandre.vandeponseele@univ-smb.fr

Context

- **Renewable resource:** > 6 Mt Spent Coffee Grounds (SCG) year worldwide
- **Green solvent:** SuperCritical CO₂ (SC-CO₂) is CO₂ above 31 °C, 74 bars
- **Solution:** To remove drying pre-treatment of SCG → reduce cost of process



✓ **Water = co-solvent**
 → favors polyphenols extraction

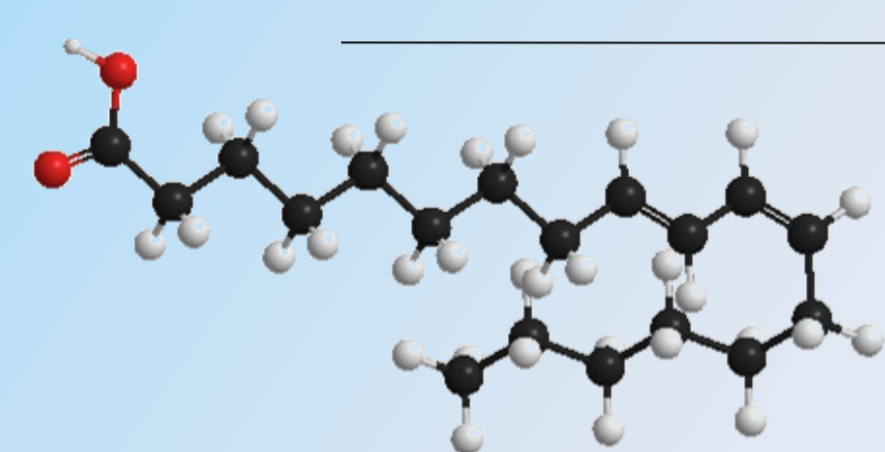
✗ **Water = barrier to CO₂ diffusion**
 → limitates lipids extraction

Objectives

- **Identify** role of pressure, temperature and moisture in SC-CO₂/H₂O/SCG system
- **Optimize** the simultaneous extraction for lipids and polyphenols recovery
- **Method** → Design of Experiments (DoE) - Central Composite Design Rotatable (CCDR)

Independent variables	Unit	Level of the factors				
		- α	-1	0	+1	+ α
Pressure	bars	115.9	150	200	250	284.1
Temperature	°C	33.2	40	50	60	66.8
Moisture content	wt%	6.4	20	40	60	73.6

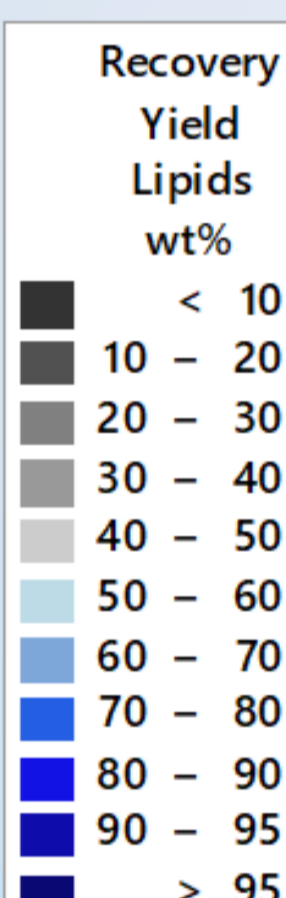
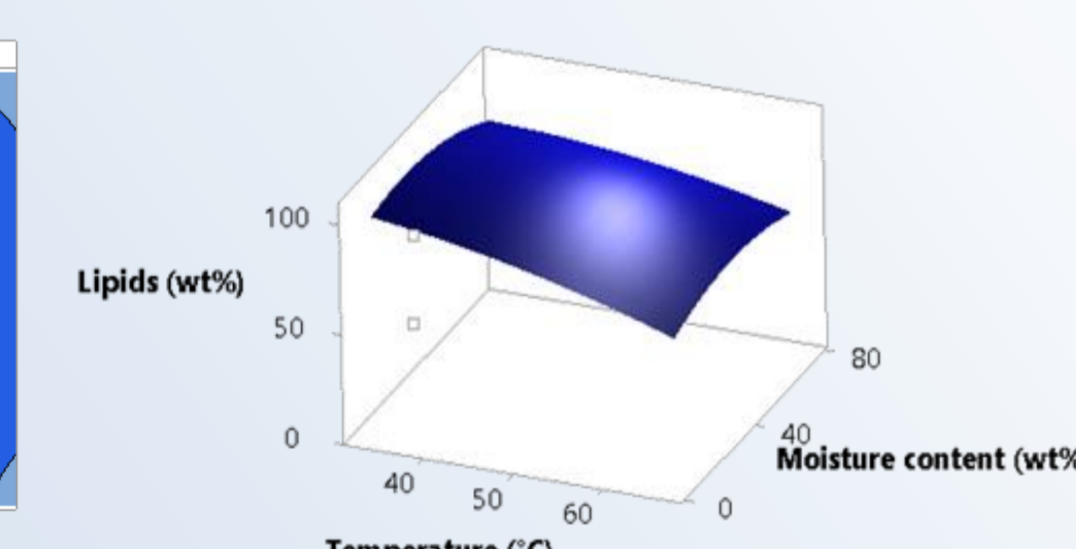
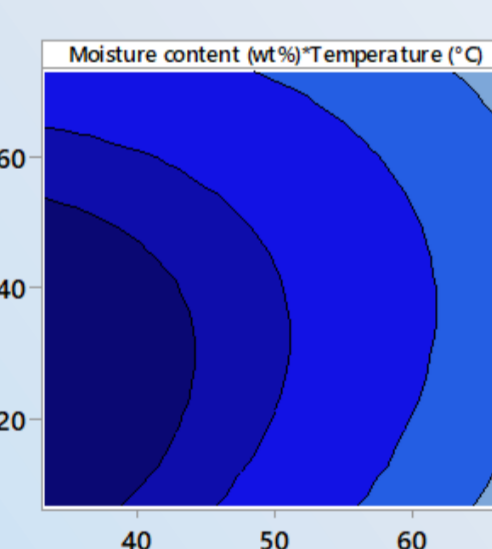
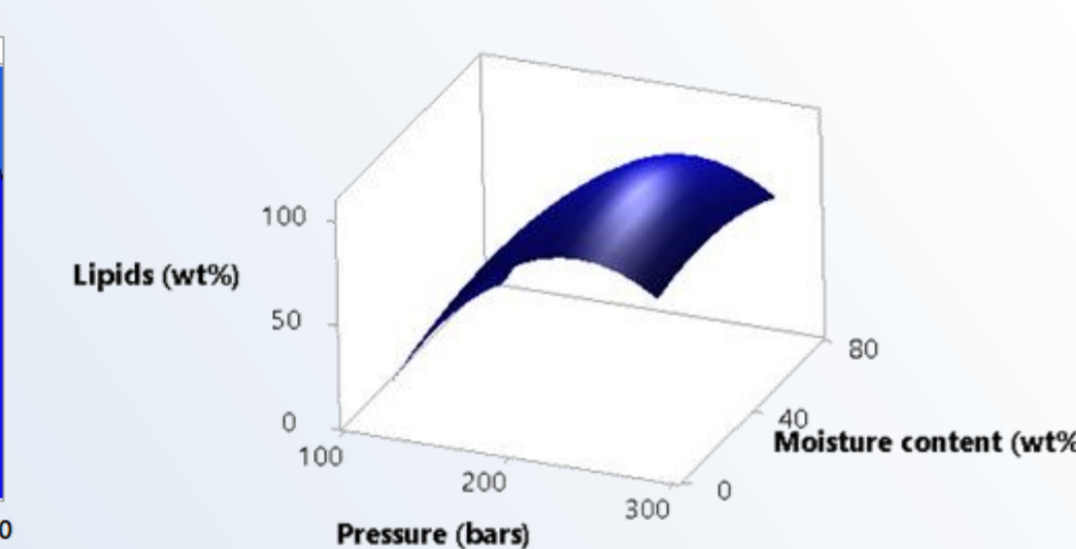
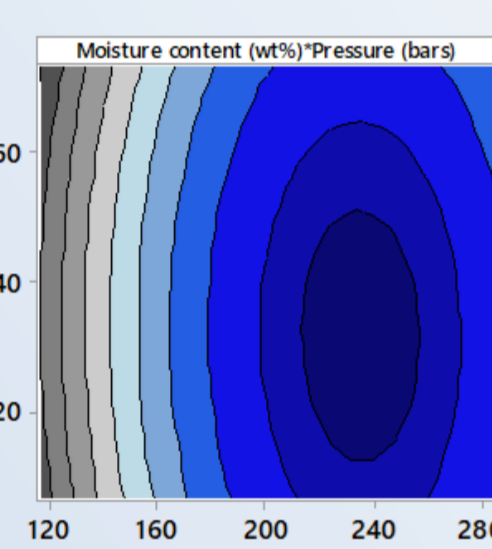
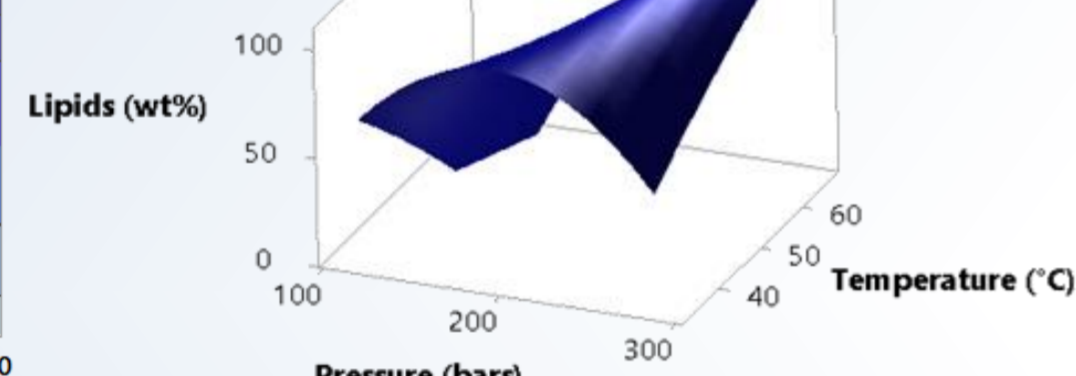
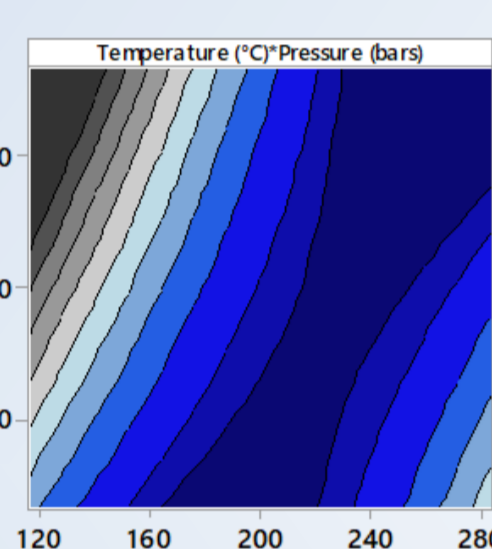
Design of Experiments



Lipids

Recovery Yield of Lipids (RYL)

$$RYL = \frac{\text{Yield of lipids extracted by SC-CO}_2}{\text{Yield of lipids extracted by n-hexane Soxhlet}}$$



RYL	Response	RYP
Polynomial equation		
P >>> T > M	Influent parameters	M >>> P > T
0.9225	R ²	0.7125
Cross optimization lipids and polyphenols extraction		
265 bars	55 °C	55 wt% moisture content
92.68 (11.40g _{oil} .100g ⁻¹ _{SCG})	Measured response	5.36 (0.66g _{polyphenols} .100g ⁻¹ _{SCG})
0.0137 (p < 0.05)	p-value	0.1362 (p > 0.05)

Pressure influence
 → Increase of CO₂ density

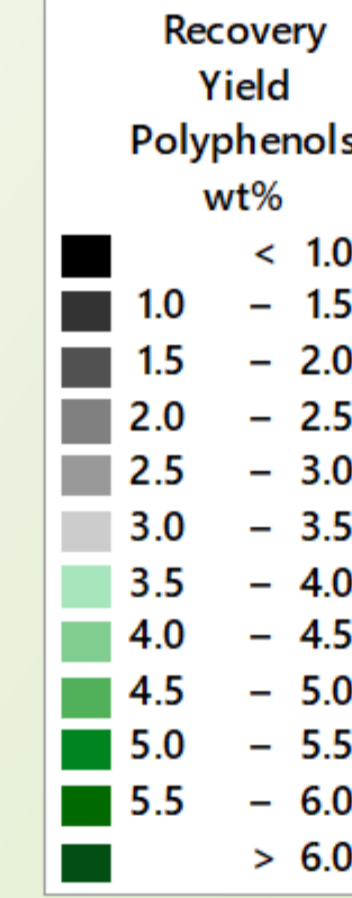
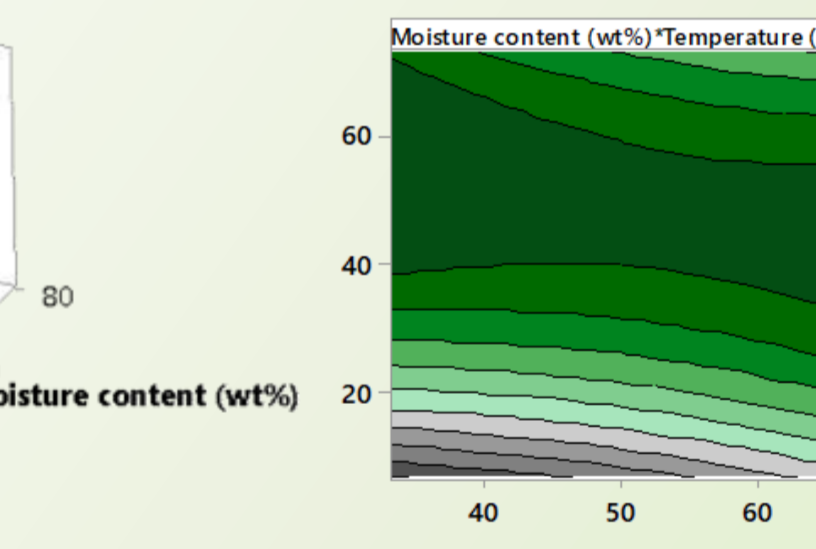
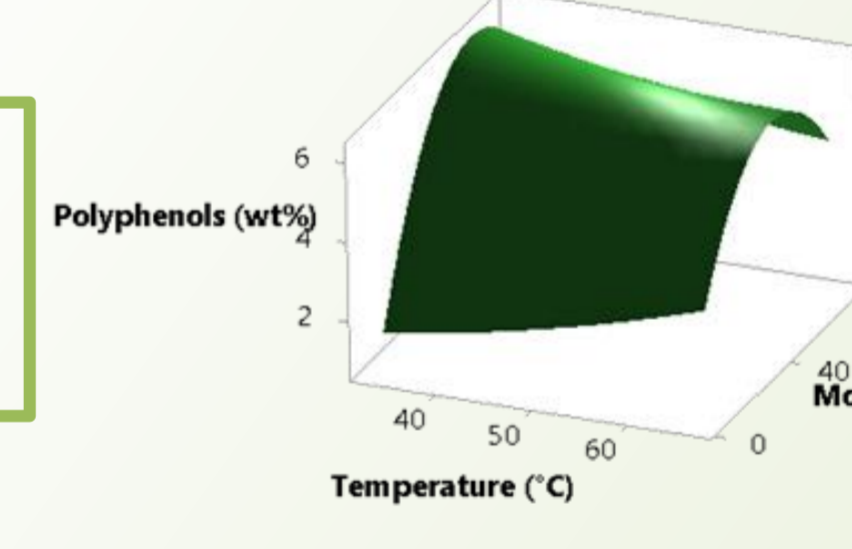
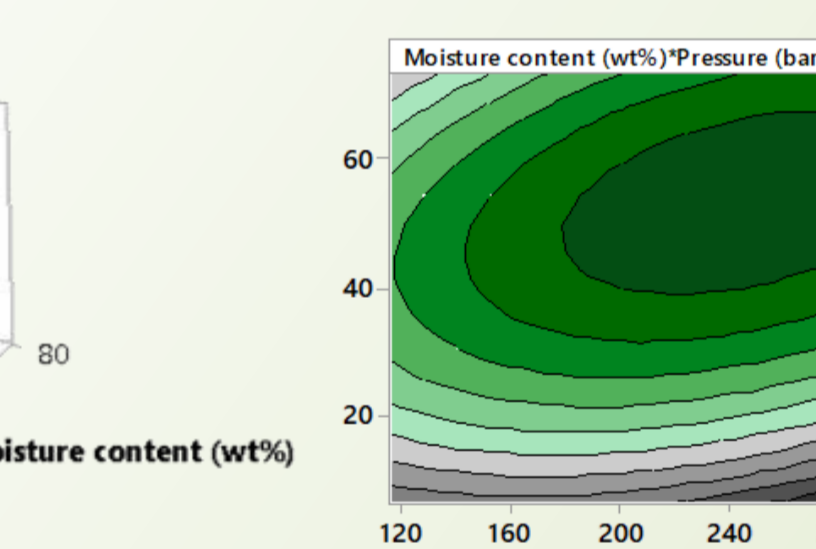
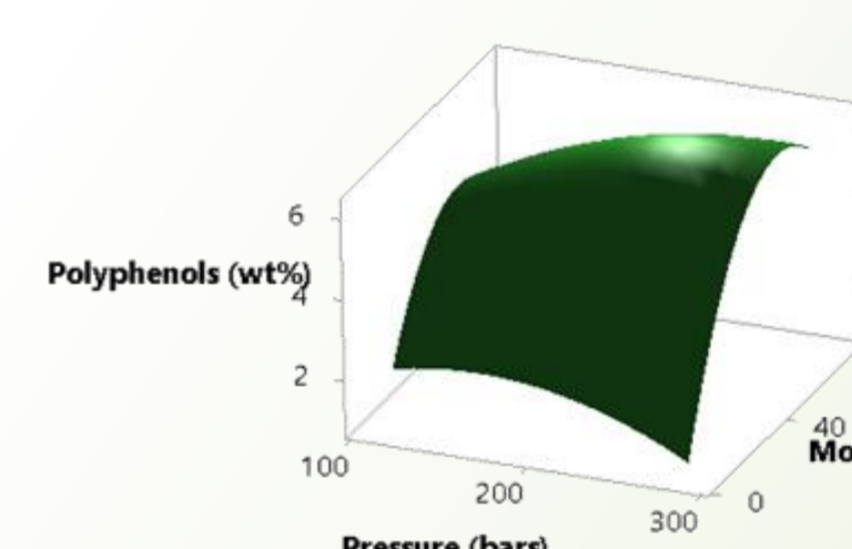
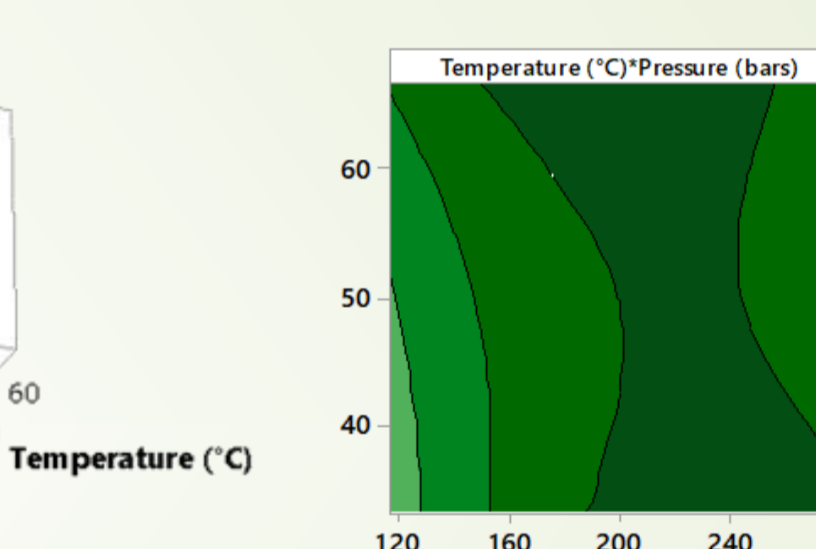
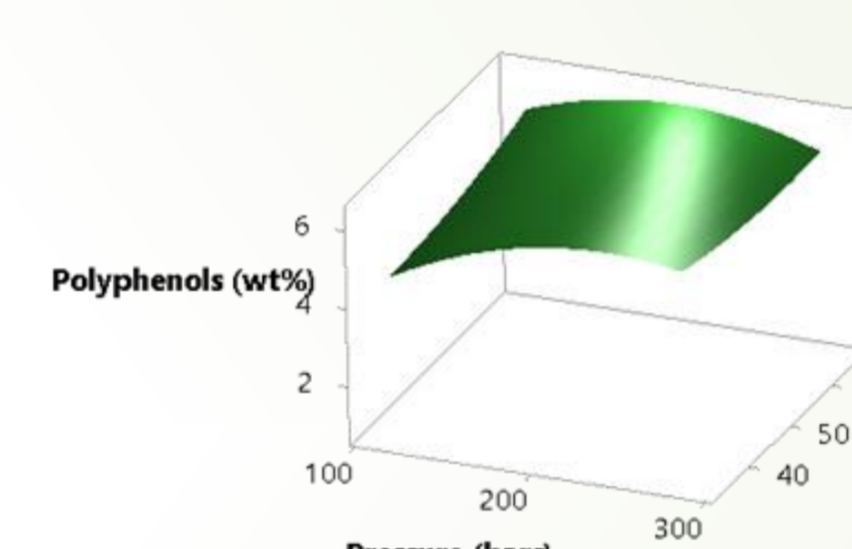
Moisture influence
 → Increase polar interactions

A. Vandeponseele et al., 2020, *Green Chemistry*, 22, 8544–8571.

Polyphenols

Recovery Yield of Polyphenols (RYP)

$$RYP = \frac{\text{Yield of polyphenols extracted by SC-CO}_2}{\text{Yield of polyphenols extracted by hydroalcohol}}$$



Characterization of oil

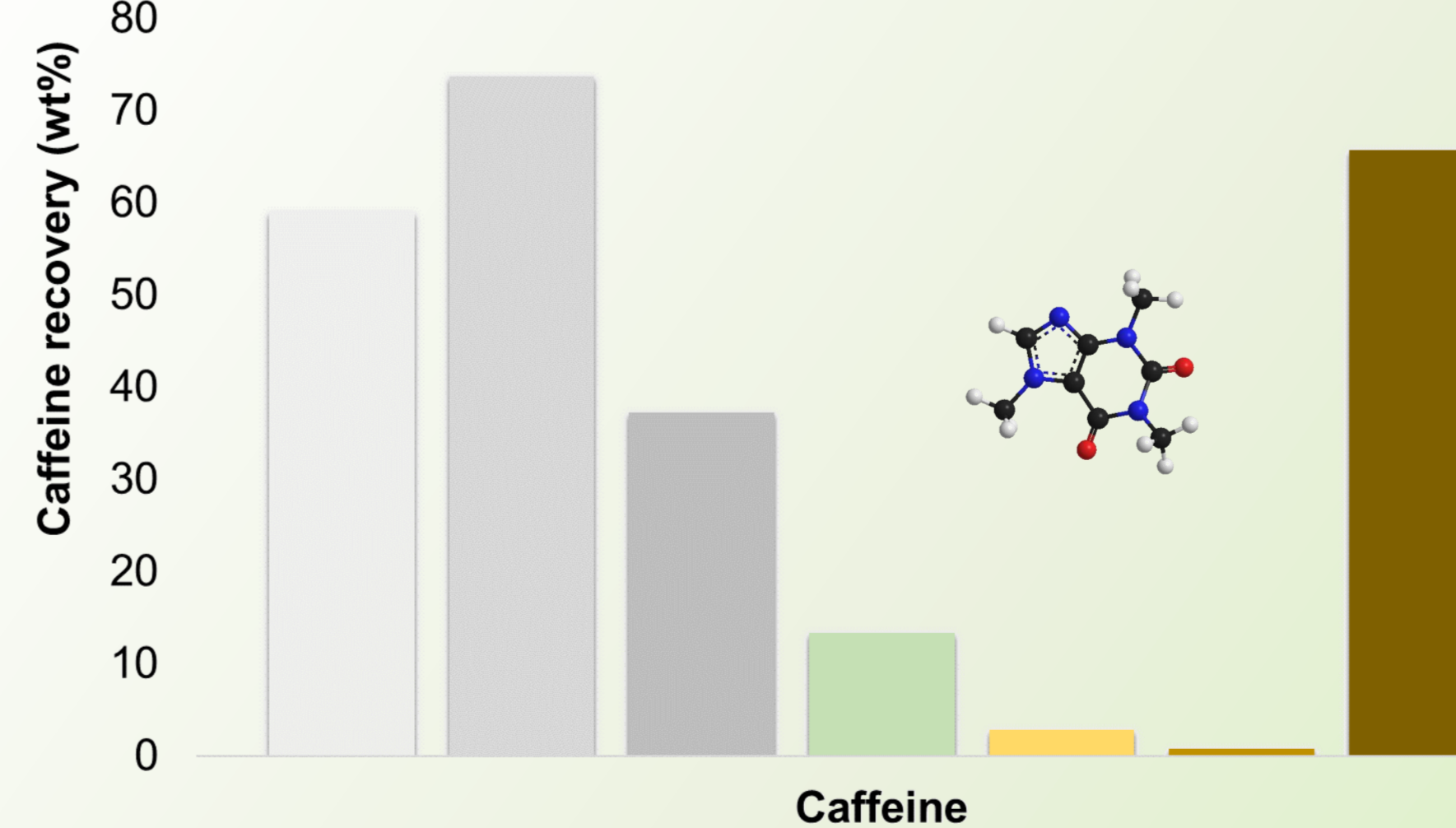
Experiments	Fatty Acids Profile (wt%)					Fatty Acids Level (g _{fatty acids} .100g ⁻¹ _{oil})	AntiOxidant Capacity (μmol _{trolox} .100g ⁻¹ _{oil})
	C16:0	C18:2	C18:1	C18:0	C20:0		
Soxhlet n-hexane oil	30.25	48.87	10.58	7.41	2.89	42.92	2319.1
Cross optimization oil	39.24	42.30	9.30	6.61	2.55	53.37	700.2
Kaffe Bueno oil	34	44	9	7.3	2.6	/	/

SCG oil obtained by SC-CO₂ VS n-Hexane
 → Fatty Acids Profile ≈, Fatty Acids Level ↗ and Antioxidant Capacity ↘

SCG oil VS commercial Kaffe Bueno oil for cosmetics
 → competitive quality compared to the commercial one

Mechanism of extraction for caffeine

Supercritical CO₂ extraction at 265 bars, 55 °C of caffeine present in different matrices



- No matrix, as pure caffeine
- With cellulose by addition of pure caffeine in dry conditions
- With cellulose by addition of pure caffeine in wet conditions
- No matrix, recrystallized chlorogenic acid-caffeine mixture
- From dry extract of hydroalcoholic extraction
- From dry spent coffee grounds
- From wet spent coffee grounds

J. Tello et al., 2011, *The Journal of Supercritical Fluids*, 59, 53–60

E. Sondheimer et al., 1961, *Archives of Biochemistry and Biophysics*, 93, 63–71

Extract composition
 → purity > 99 wt% in caffeine

Caffeine behavior in coffee
 → complexes with chlorogenic acid
 → interacts with its natural matrix

Mechanism of caffeine extraction
 → i) H₂O breaks caffeine interactions
 → ii) SC-CO₂/H₂O liquid/liquid extraction

Perspectives

- **Modelize** the SC-CO₂/H₂O/SCG interactions with DFT simulation

- **Explore** novel combination of SC-CO₂ with green solvents

- **Study** the SC-CO₂/H₂O system applied to other biomasses