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

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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



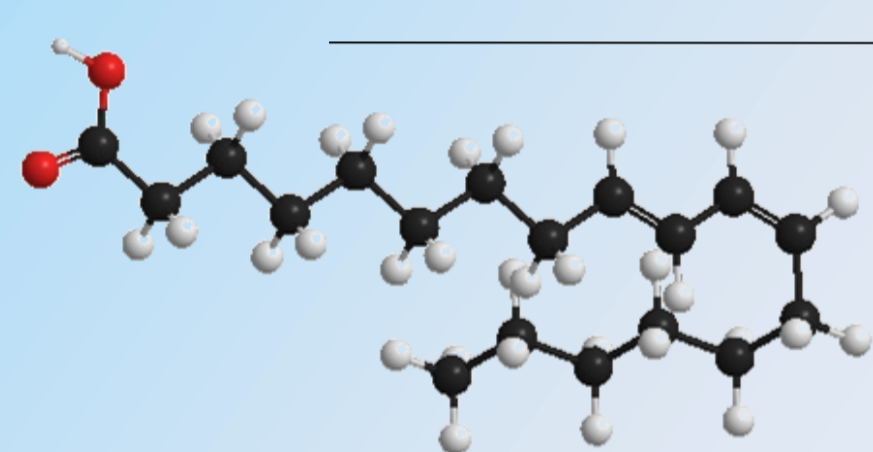
✓ **Water = co-solvent**
 → favors polyphenols extraction

✗ **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)

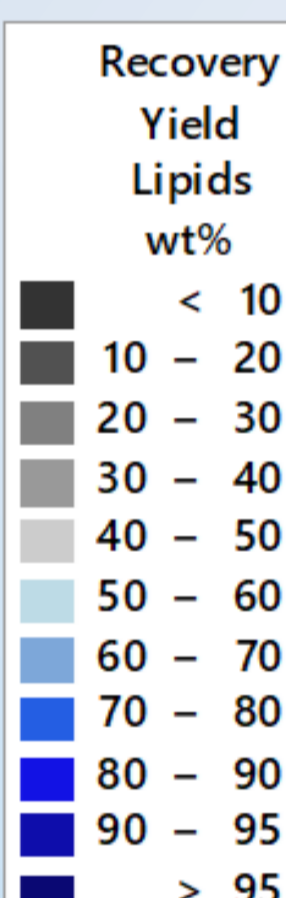
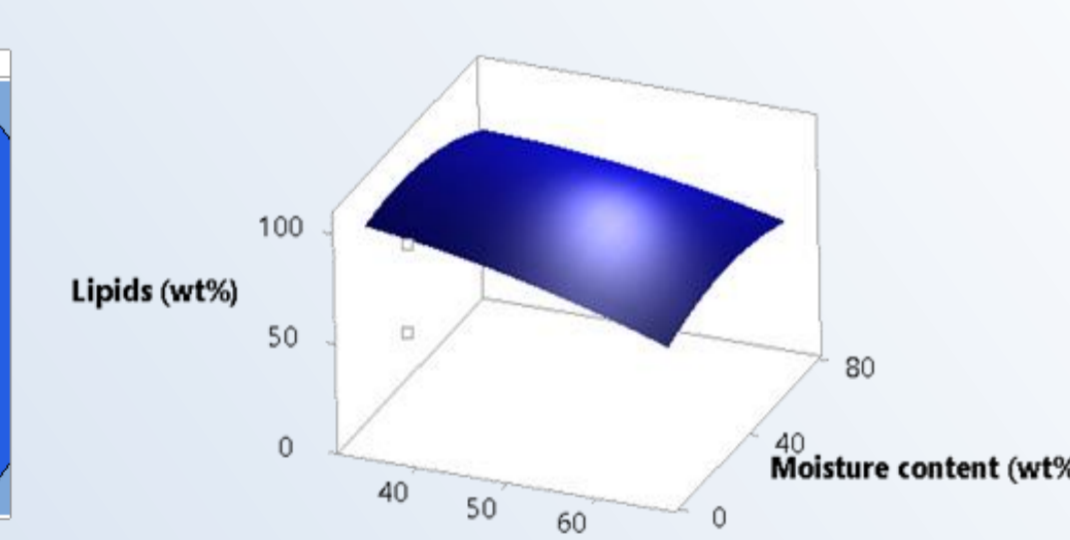
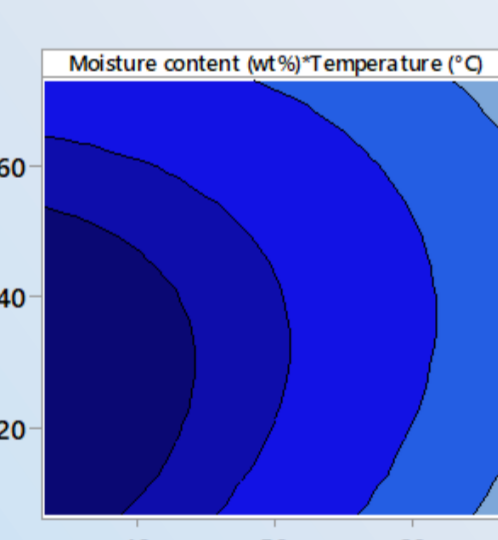
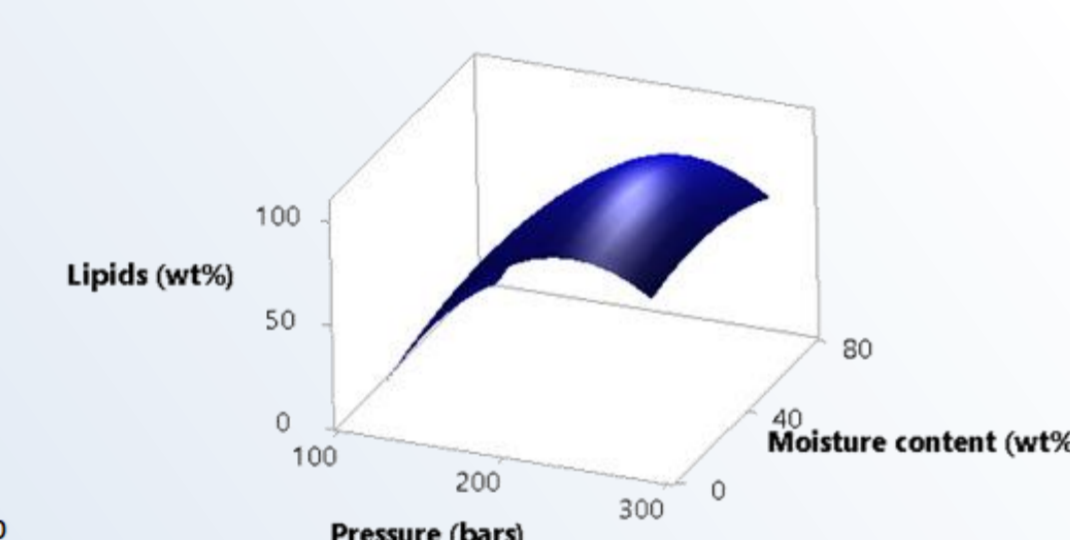
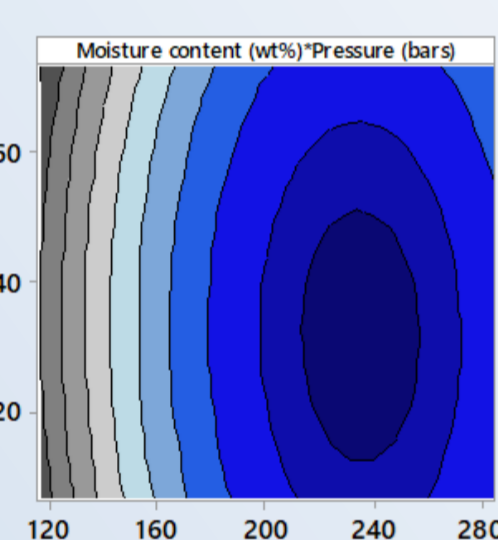
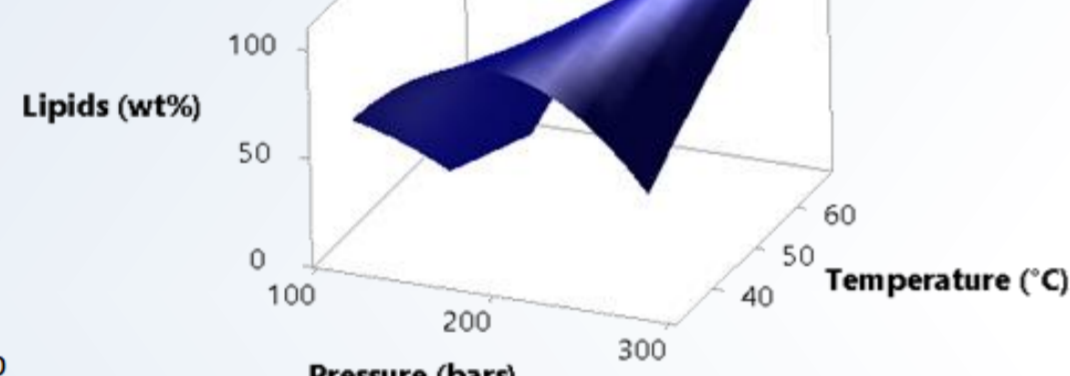
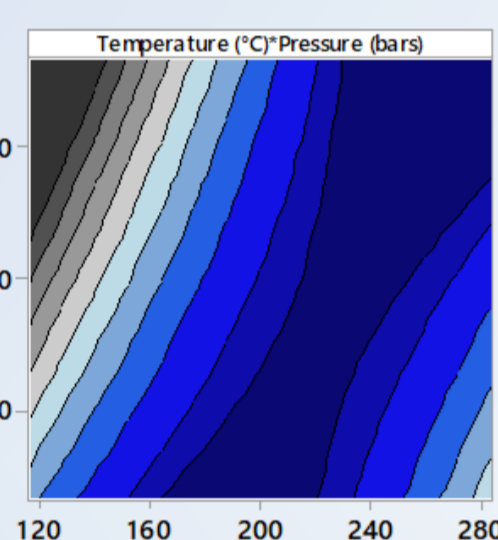
Independent variables	Unit	Level of the factors				
		- α	-1	0	+1	+ α
Pressure	bars	115.9	150	200	250	284.1
Temperature	°C	33.2	40	50	60	66.8
Moisture content	wt%	6.4	20	40	60	73.6



Lipids

Recovery Yield of Lipids (RYL)

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



Design of Experiments

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

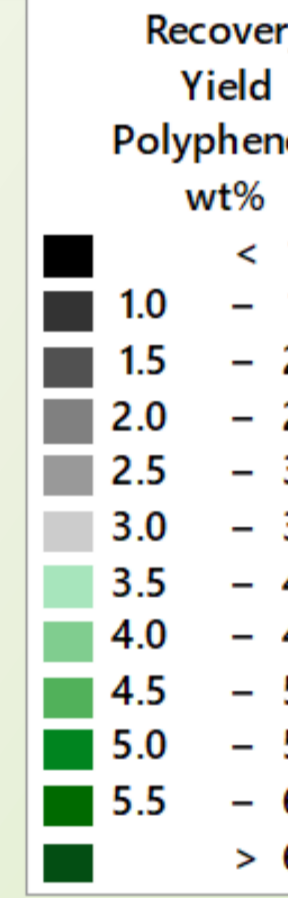
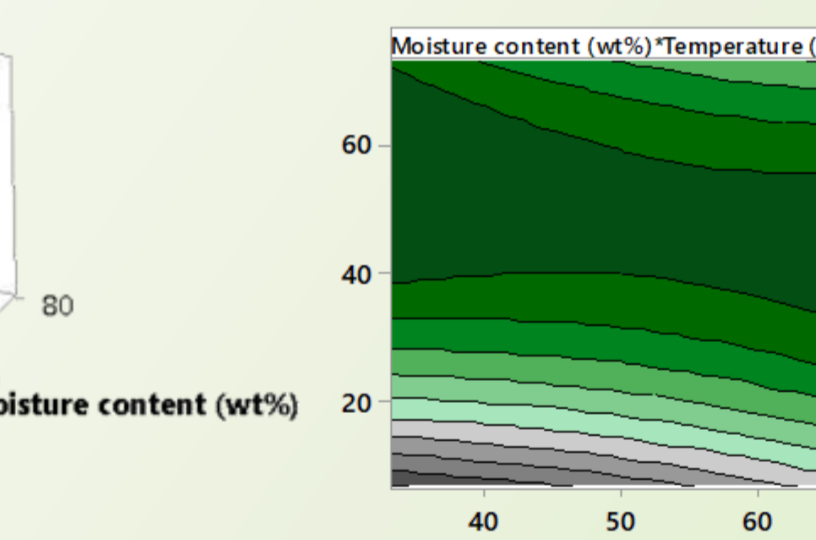
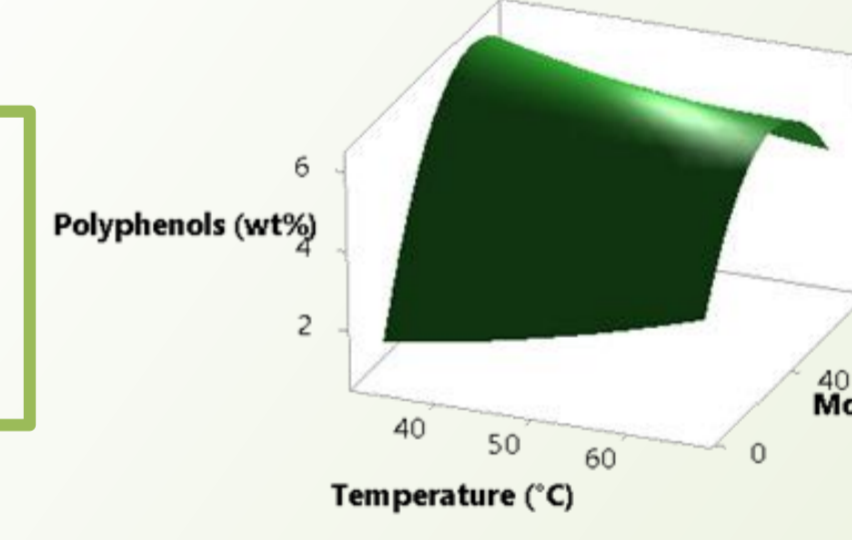
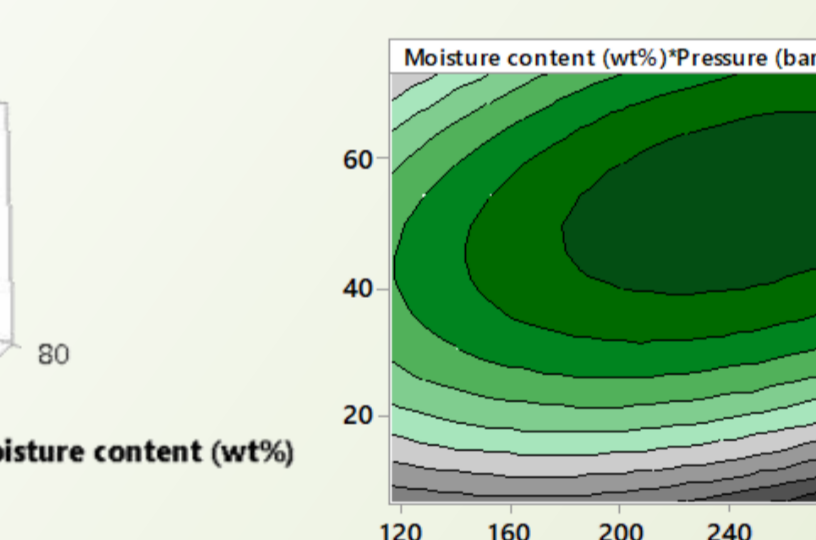
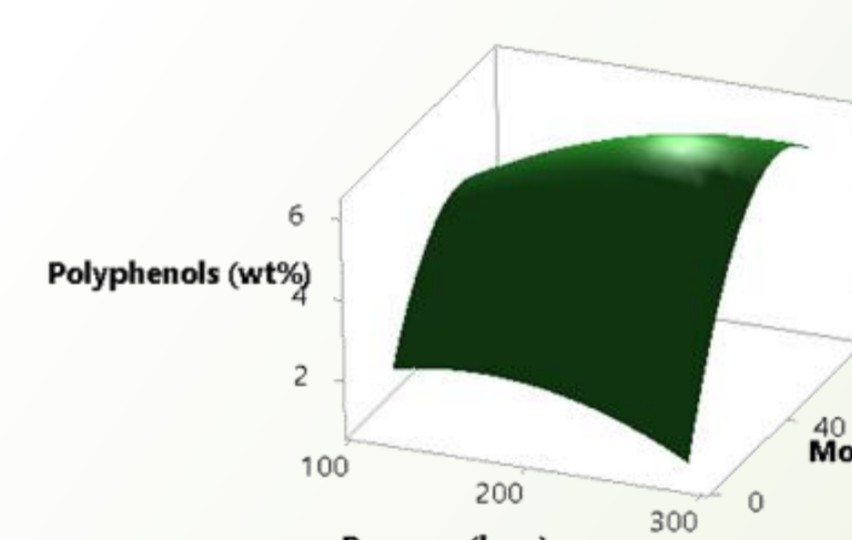
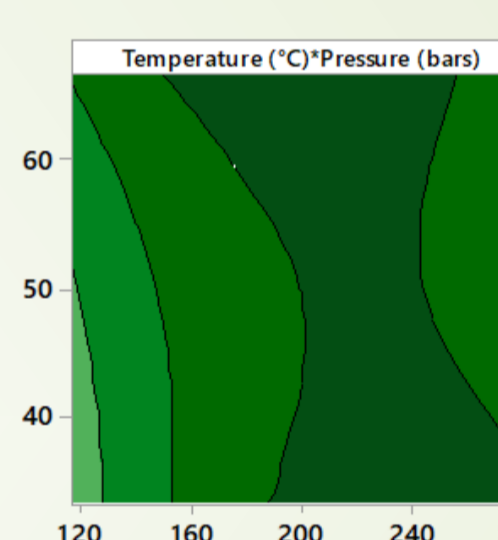
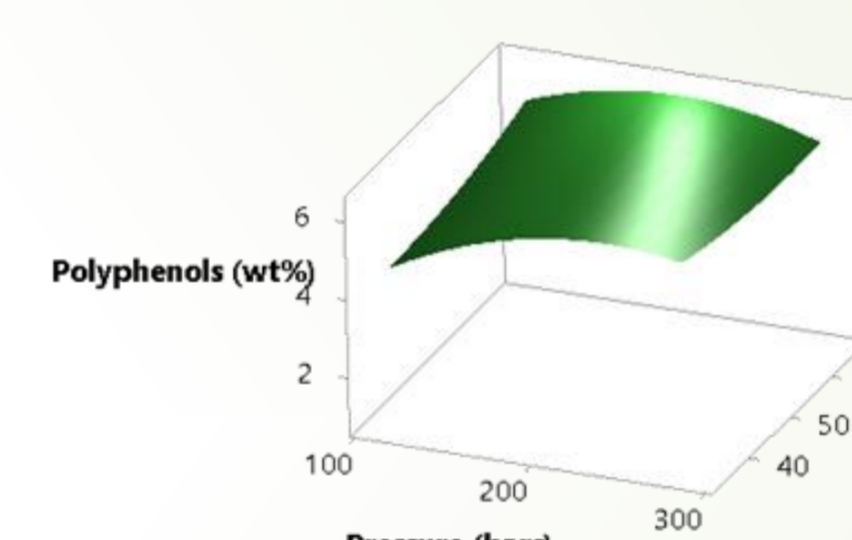
Moisture influence
 → Increase polar interactions

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

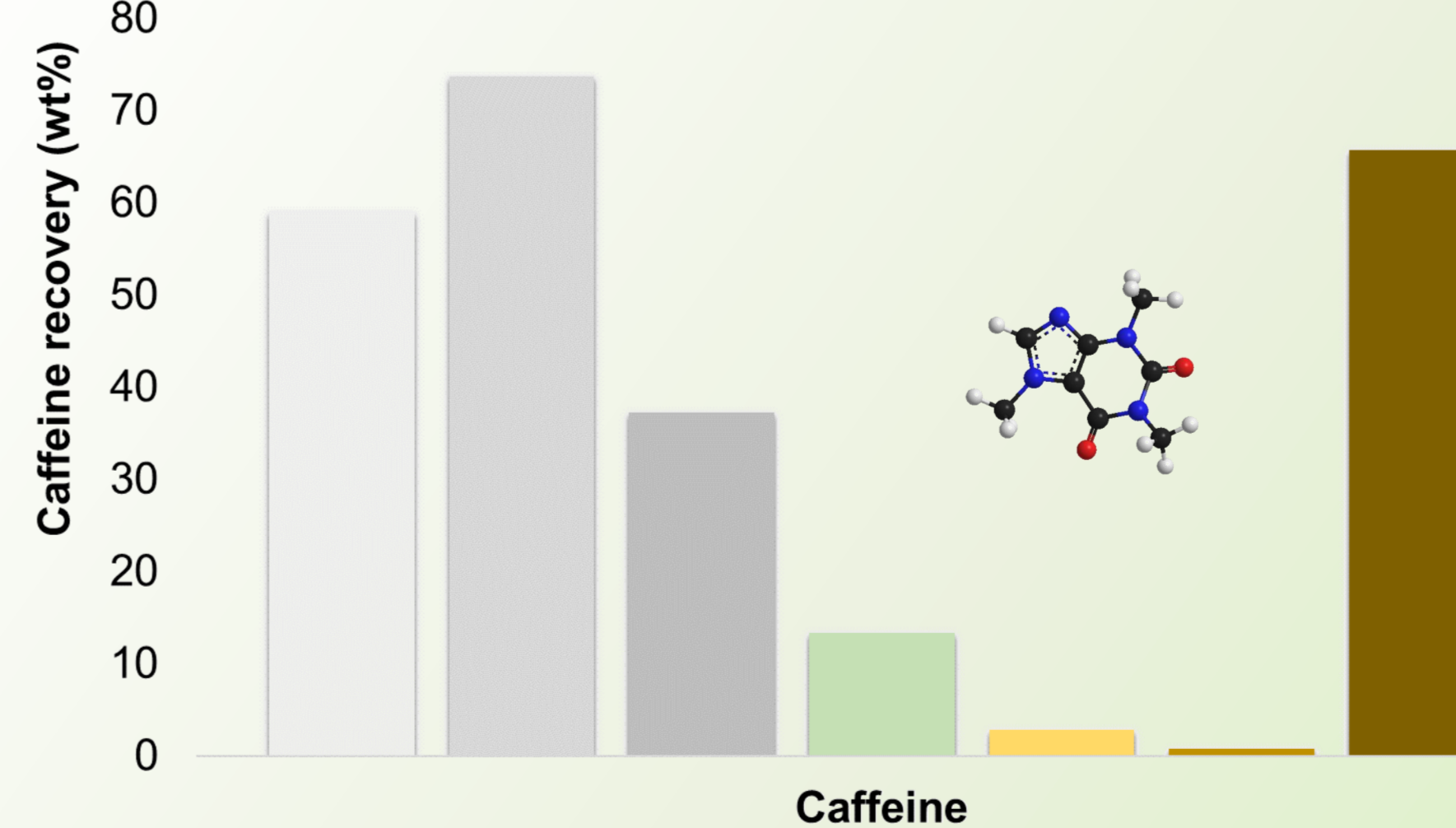
Experiments	Fatty Acids Profile (wt%)					Fatty Acids Level (g _{fatty acids} .100g ⁻¹ _{oil})	AntiOxidant Capacity (μmol _{trolox} .100g ⁻¹ _{oil})
	C16:0	C18:2	C18:1	C18:0	C20:0		
Soxhlet n-hexane oil	30.25	48.87	10.58	7.41	2.89	42.92	2319.1
Cross optimization oil	39.24	42.30	9.30	6.61	2.55	53.37	700.2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