

Coffee Pulp Valorization Strategy: flavonoid- and lipid-enriched food-grade ingredients obtained from an advanced extraction technology

Shuai Hu^{1,2}, María Martín-Trueba^{1,2}, Silvia Cañas^{1,2}, Miguel Rebollo-Hernanz^{1,2}, Yolanda Aguilera^{1,2}, Vanesa Benítez^{1,2}, María Ángeles Martín-Cabrejas^{1,2}, Alicia Gil-Ramírez^{1,2}

¹ Department of Agricultural Chemistry and Food Science, Faculty of Science, C/ Francisco Tomás y Valiente, 7. Universidad Autónoma de Madrid, 28049, Madrid, Spain.

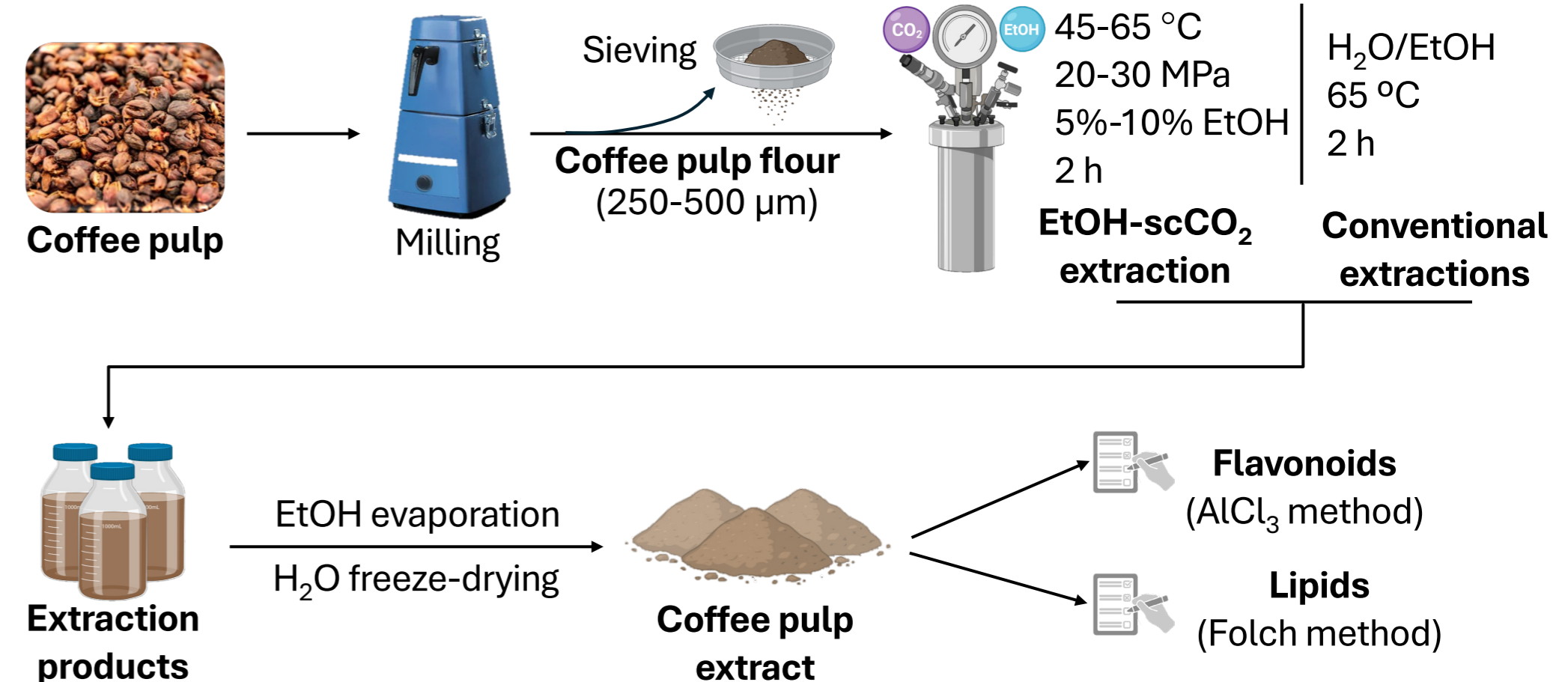
² Department of Production and Characterization of Novel Foods, Institute of Food Science Research, CIAL (UAM-CSIC), 28049 Madrid, Spain

Introduction & Aim

The valorization of coffee pulp (CP) is gaining interest because of its content in high-value bioactive compounds. **Flavonoids and lipids** have been traditionally recovered from coffee by-products using organic solvents such as methanol, hexane, or acetone [1]. However, the toxicity of these solvents limits their food applications. Accordingly, the development of food-grade extraction processes is promoted, supporting the idea of sustainability [2].

This work investigated the feasibility of **ethanol-modified supercritical carbon dioxide (EtOH-scCO₂) extraction** as a safe and green technique for recovering flavonoids and lipids from CP. The extraction conditions were screened by response surface methodology to **maximize selectivity** (per unit of extract) and **efficiency** (per unit of CP) of lipid and flavonoid recovery.

Method



Results & Discussion

Table 1. Experimental data of the response variables

Sample	Factors			Response variables expressed per unit of extract		Response variables expressed per unit of CP	
	T (°C)	P (MPa)	EtOH (w/w, %)	Flavonoids (µg CE/mg)	Lipids (mg/g)	Flavonoids (µg CE/mg)	Lipids (mg/g)
N1	45	20	5	33.3 ± 1.3 ^g	466.9 ± 17.0 ^d	0.9 ± 0.0 ^{ab}	12.9 ± 0.5 ^{ab}
N2	45	30	10	29.1 ± 1.0 ^{def}	345.2 ± 7.7 ^c	1.4 ± 0.1 ^{de}	17.9 ± 0.4 ^{gh}
N3	45	30	5	33.7 ± 2.9^g	483.7 ± 23.6 ^d	0.9 ± 0.0 ^{ab}	13.2 ± 0.7 ^b
N4	45	20	10	27.7 ± 0.6 ^{cde}	328.2 ± 3.7 ^c	1.4 ± 0.1 ^d	17.7 ± 0.2 ^{gh}
N5	45	20	5	31.7 ± 1.7 ^{fg}	476.5 ± 31.1 ^d	0.9 ± 0.0 ^{ab}	13.4 ± 0.9 ^b
N6	55	20	10	31.8 ± 1.7 ^{fg}	308.2 ± 7.5 ^c	1.7 ± 0.0 ^e	17.2 ± 0.5 ^{fgh}
N7	55	20	5	26.9 ± 2.0 ^{bcd}	493.5 ± 1.6 ^d	0.7 ± 0.0 ^a	14.2 ± 0.1 ^{bc}
N8	55	30	10	27.6 ± 1.0 ^{cde}	318.6 ± 6.4 ^c	1.6 ± 0.0 ^{de}	18.3 ± 0.4 ^{gh}
N9	55	30	5	30.4 ± 1.2 ^{efg}	505.3 ± 10.2^d	0.9 ± 0.0 ^{ab}	15.5 ± 0.3 ^{cde}
N10	65	30	10	23.7 ± 1.5 ^b	309.4 ± 8.2 ^c	1.4 ± 0.1 ^{cd}	18.7 ± 0.5 ^{hi}
N11	65	30	5	29.6 ± 0.7 ^{def}	468.3 ± 16.9 ^d	0.9 ± 0.0 ^{ab}	15.0 ± 0.6 ^{cd}
N12	65	20	10	25.4 ± 1.5 ^{bc}	319.4 ± 8.3 ^c	1.5 ± 0.1 ^{de}	20.0 ± 0.5 ^{ij}
N13	65	20	5	30.9 ± 1.5 ^{efg}	492.6 ± 1.2 ^d	0.9 ± 0.1 ^{ab}	15.3 ± 0.1 ^{cd}
N14	45	30	10	31.7 ± 2.5 ^{fg}	316.6 ± 11.5 ^c	1.6 ± 0.1 ^{de}	17.0 ± 0.6 ^{efg}
N15	65	30	5	33.6 ± 0.8 ^g	479.0 ± 6.7 ^d	1.1 ± 0.1 ^{bc}	15.9 ± 0.2 ^{def}
N16	65	20	10	26.9 ± 1.6 ^{bcd}	347.1 ± 17.4 ^c	1.6 ± 0.1 ^{de}	21.4 ± 1.1 ^j
Water				17.0 ± 0.8 ^a	30.0 ± 2.4 ^a	6.6 ± 0.2^g	11.6 ± 0.7 ^a
Ethanol				16.6 ± 1.1 ^a	136.7 ± 8.2 ^b	3.6 ± 0.1 ^f	29.8 ± 0.2^k

^{a-k} Different superscript letters indicate statistically significant differences at $p < 0.05$ within the same response.

- Compared to conventional water and ethanol extractions, **EtOH-scCO₂ yielded lower flavonoid recovery efficiency**; while the **opposite was observed for lipids** compared to water extraction.
- A superior extraction selectivity was achieved using EtOH-scCO₂ compared to conventional extractions, up to 33.7 mg CE/g extract for flavonoids at **45 °C, 30 MPa, and 5% ethanol**; and 505.3 mg/g extract for lipids at **55 °C, 30 MPa and 5% ethanol**.

- EtOH significantly ($p < 0.05$) **increased the extraction efficiency** (regression coefficients=0.31 and 0.20 for flavonoids and lipids, respectively) but **decreased the extraction selectivity** (regression coefficients=-1.75 and -8.17 for flavonoids and lipids, respectively). Similar effects were observed for temperature, although they lacked statistical significance ($p > 0.05$).
- Interactions** between factors were observed, but no significant ($p > 0.05$) effects were noted.

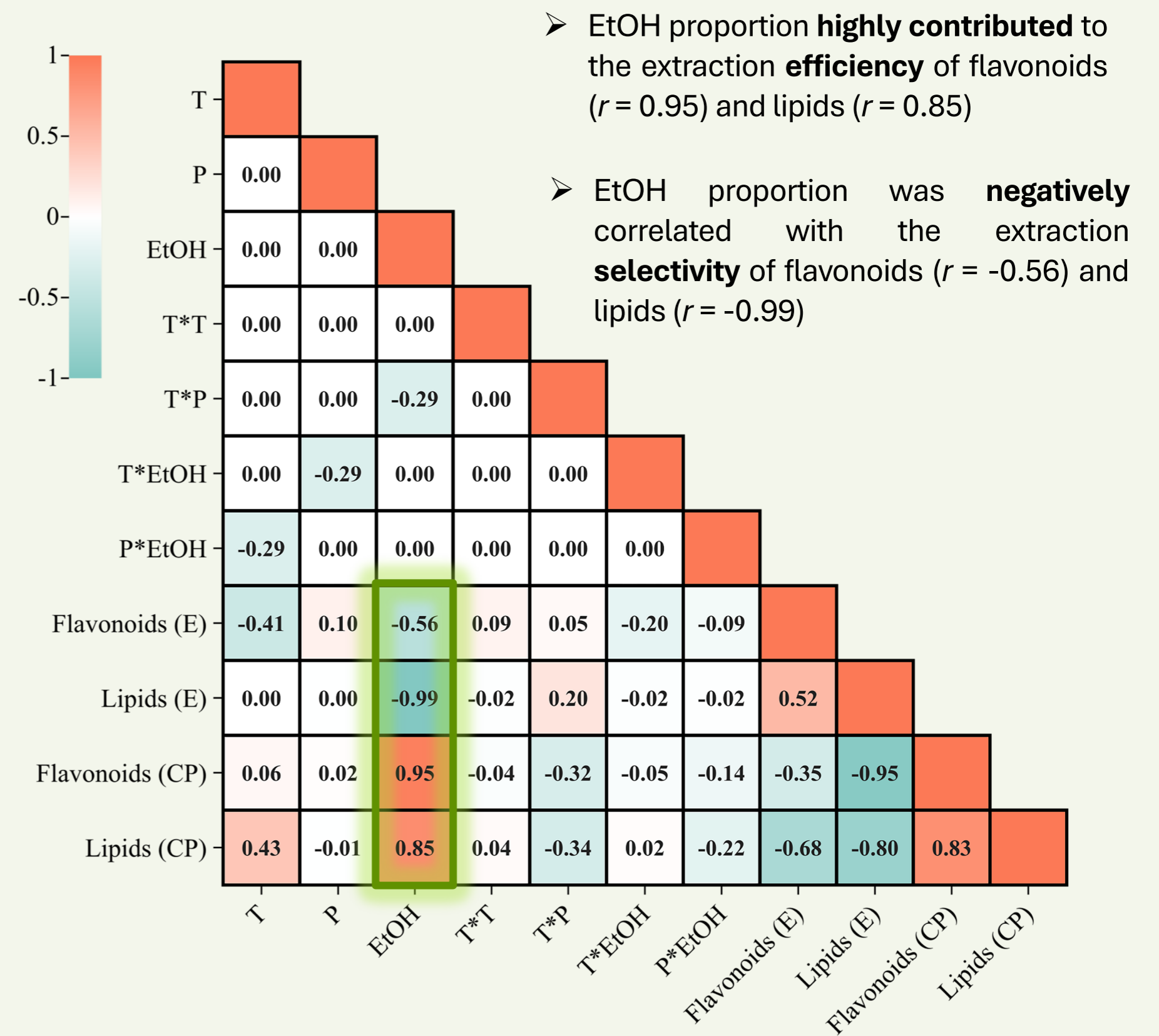


Figure 1. Heatmap of correlation among factors and response variables including their corresponding Pearson correlation coefficients

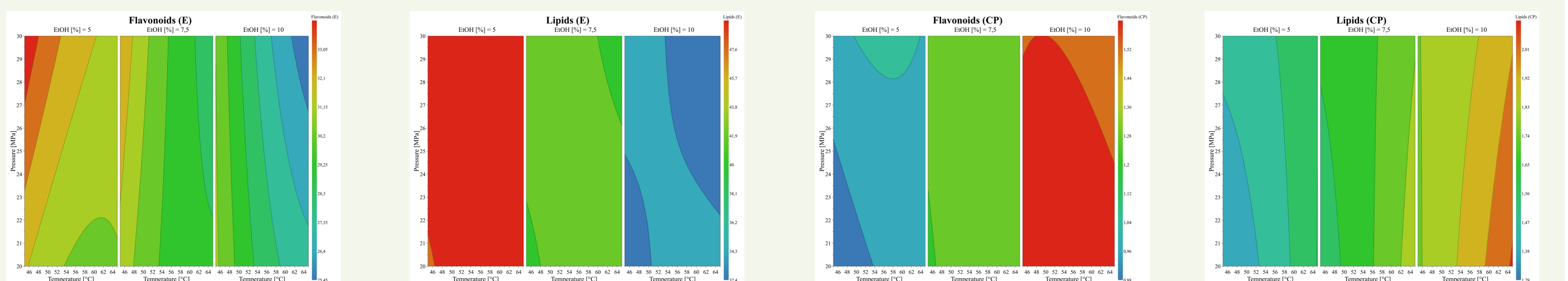


Figure 2. Response contour plots showing the impact of temperature, pressure and co-solvent on the response variables.

Conclusion

The increased amount of **ethanol** as a co-solvent **enhanced the extraction efficiency** but reduced selectivity for **flavonoids and lipids**, which might be attributed to the modification in polarity of the system induced by the mentioned co-solvent.

The **selectivity** results indicate that **EtOH-scCO₂** appears to be a **promising technique** for obtaining flavonoid- and lipid-enriched food-grade ingredients from coffee pulp, compared to the conventional extractions.

Further research is needed to clarify the potential applications of flavonoid- and lipid-enriched CP extracts to valorize coffee industry by-products as bioactive food-grade ingredients.

References

- Lee, Y. G.; Cho, E. J.; Maskey, S.; Nguyen, D. T.; & Bae, H. J. (2023). Value-added products from coffee waste: a review. *Molecules*, 28(8), 3562.
- Vandepoesele, A., Draye, M., Piot, C., & Chatel, G. (2020). Subcritical water and supercritical carbon dioxide: Efficient and selective eco-compatible solvents for coffee and coffee by-products valorization. *Green Chemistry*, 22(24), 8544-8571.

Acknowledgements

This work is part of the project TED2021-129262A-I00EFERENCA, funded by MCIN/AEI/10.13039/501100011033 and by the European Union "NextGenerationEU"/PRTR. The authors are grateful to the funding granted by the China Scholarship Council (CSC202208360052) and the Excellence Line for University Teaching Staff within the Agreement between the Community of Madrid and the UAM (2019-2024).