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Stilbenoids eco extraction from vineyard coproducts: vitisin B extraction by ASE compared to sc CO₂

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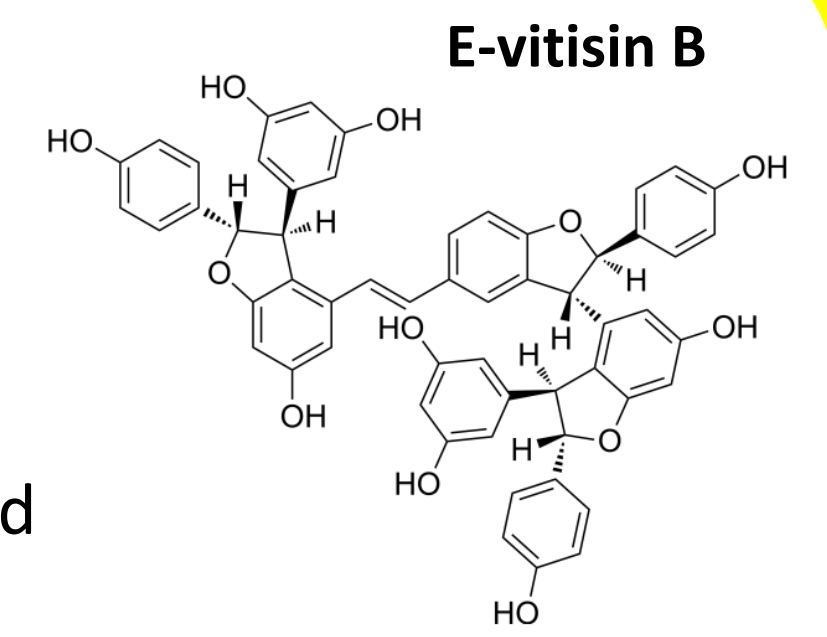
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Context:

The wine industry generates significant waste, including grape marc, vine shoots, lees, and vine roots during vineyard renewal. However, these by-products are rich in valuable compounds like polyphenols, with resveratrol being the most recognized. Beyond resveratrol, other stilbenoids are present in vineyard co-products, such as E-vitisin B, a tetramer of E-resveratrol formed through phenolic oxidative coupling. This compound is particularly abundant in vine roots and exhibits strong bioactivity, including antifungal and anti-mycotoxin properties. The aim of this study is to evaluate different extraction techniques — supercritical CO₂ and Accelerated Solvent Extraction (ASE) — for extracting E-vitisin B from *Vitis vinifera* L. roots. Optimization of the extraction process using these methods was achieved through Box-Behnken Design (BBD) and full factorial design.



Gabaston, J., Leborgne, C., Waffo-Teguo, P., Valls, J., Palos Pinto, A., Richard, T., Cluzet, S., Mérillon, J.-M., 2019. Wood and roots of major grapevine cultivars and rootstocks: A comparative analysis of stilbenes by UHPLC-DAD-MS/MS and NMR. *Phytochemical Analysis* 30, 320–331. <https://doi.org/10.1002/pca.2815>
 Tran, T.M., Atanasova, V., Tardif, C., Richard-Forget, F., 2023. Stilbenoids as Promising Natural Product-Based Solutions in a Race against Mycotoxigenic Fungi: A Comprehensive Review. *J. Agric. Food Chem.* 71, 5075–5092. <https://doi.org/10.1021/acs.jafc.3c00407>

Materials and methods:

Raw material



Roots of *Vitis vinifera* L. were locally harvested in 2018. After drying at 25°C and milling, the roots were stored at room temperature protected from direct light.

ASE (Accelerated Solvent Extraction)



ASE Dionex 350, 5 g of roots treated, pressure 100 bar, 1 cycle, 40-80°C, 5-15 min time of contact, EtOH:water (%v/v) 90:10 -70:30

SFE (Supercritical Fluid Extraction)



50g of roots treated, pressure 100-250 bar, temperature 50-70°C, ethanol cosolvent 10-30% (m/m). Extraction with 550mL of cosolvent. Q_{CO₂} 7 mL/min (Patm, Tamb).

Characterization

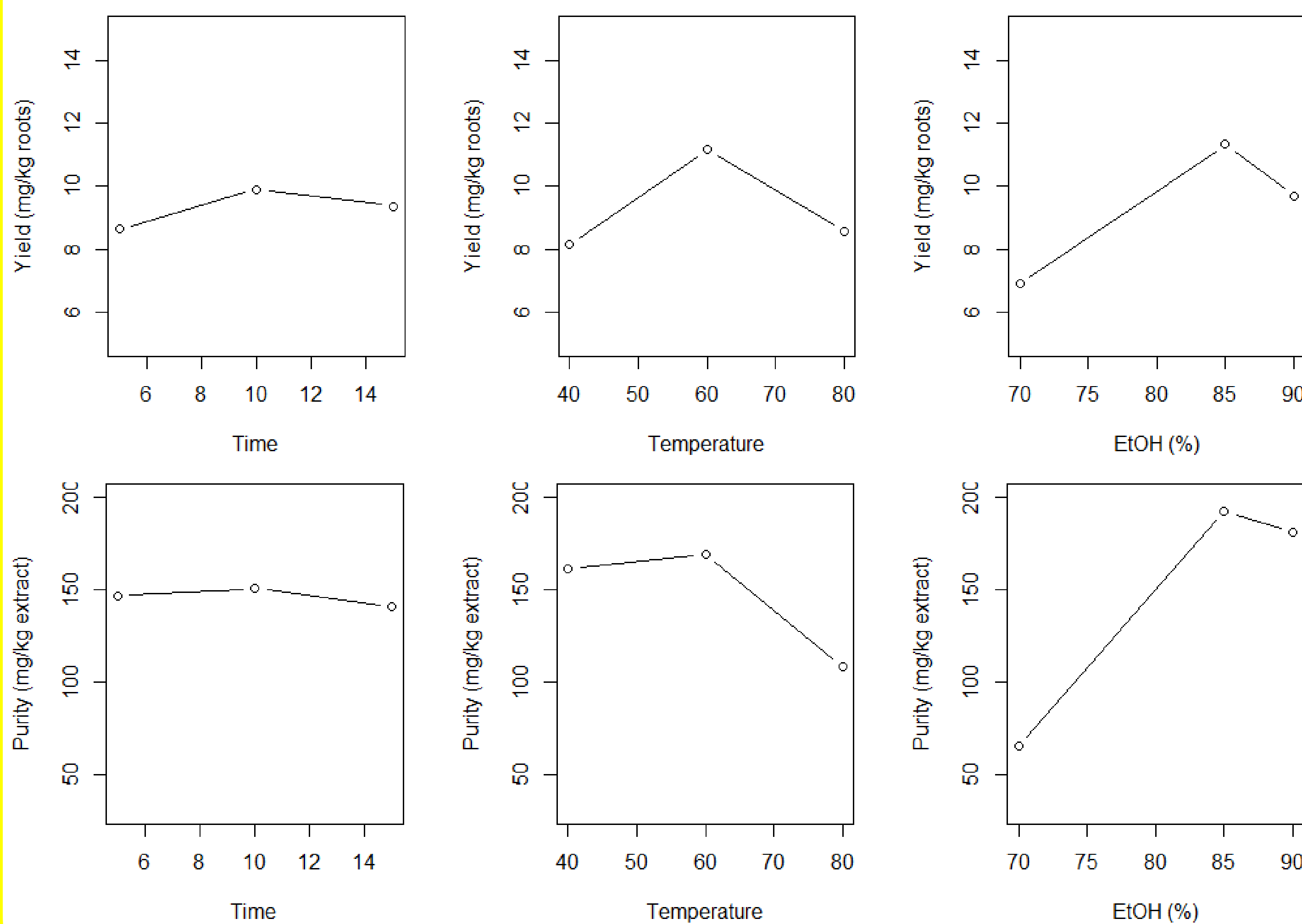


HPLC analysis (C18 column, DAD at = 254, 280, 306 and 320 nm, elution with a gradient of acidified water (0.25%TFA) and ACN).

ASE optimization

3³full factorial design
 response = vitisin B yield (g/kg roots)

Factor	Unit	-1 level	0 level	+1 level
Temperature	°C	40	60	80
Time of contact	Min	5	10	15
% of ethanol in solvent	% (v/v)	70	85	90



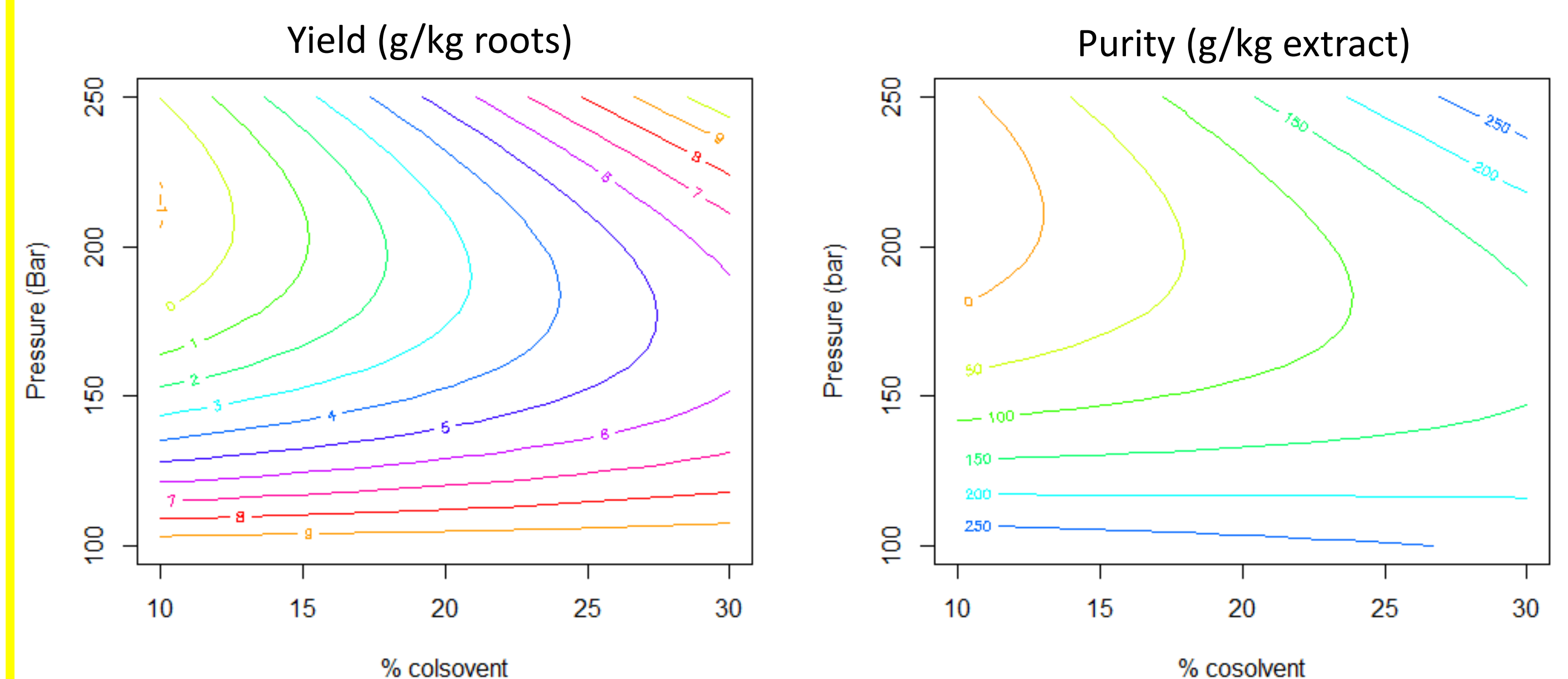
Yield poorly affect by conditions ($R^2_{adj}=0.27$) but purity is dependant on temperature et solvent composition ($R^2_{adj}=0.55$). Higher yield (22.3 g/kg roots) obtain with 85% EtOH at 60°C for 10 min. Corresponding purity is 348 g/kg of extract

SFE optimization:

3 factors Box Benkhen design
 response = vitisin B yield (g/kg roots) and purity (g/kg extract)

Factor	Unit	-1 level	0 level	+1 level
Temperature	°C	50	60	70
Pressure	bar	100	175	250
% of cosolvent	% (m/m)	10	20	30

Significant parameters for both responses : Pressure (simple and quadratic term), % of cosolvent (simple term) and their interaction. $R^2_{adj}=0.81$.



Best conditions obtained by a desirability approach : 250 bar, 30% ethanol in the extracting flux.

Predicted responses in such conditions : 10.8 g vitisin B/kg of roots (yield) and 298 g vitisin B/kg extract (purity). Experimental results in these conditions : 10.7 ± 0.7 g/kg roots yield and 300 ± 10 g/kg extract purity

Conclusions

The extraction of E-vitisin B using ASE and SFE was successfully optimized. ASE resulted in a higher yield (22.3 g/kg of roots) compared to SFE (10.7 g/kg of roots). However, both processes produced extracts with similar purity around 30 g of E-vitisin B per kg of extract. Both methods demonstrated effectiveness in the extraction of E-vitisin B from vine roots.

