Hydrothermal Extraction of Tannins from Pine Bark: Optimization, Characterization and Comparison with Commercial Antioxidants

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Summary

Figure 1 – Grounded pine bark

Hydrothermal treatment of pine bark was optimized using a Box-Behnken design. Quadratic models for all responses showed good fit (R^2 > 0.95; p < 0.0001). The optimal conditions were **130 °C, 45 min, solid/liquid ratio 1/20 g/mL**, yielding:

- Extraction yield: **13.54 ± 1.30 g/100 g dry bark**
- Total phenolics: **30.18 ± 2.11 mg GAE/g dry bark**
- FRAP: **13.32 ± 2.80 mmol AAE/100 g dry bark**
- Condensed tannins: **6.79 ± 1.57 mg CE/g dry bark**
- Stiasny number: **24.03 ± 4.27 %**

Extracts showed lower antioxidant capacity than BHA and α-tocopherol in FRAP, ABTS, and DPPH assays.
Conclusion: Hydrothermal treatment is a sustainable method to obtain phenolic-rich extracts with measurable antioxidant capacity and promising adhesive properties, supporting their potential application in bioadhesive formulations.

Introduction

Pine bark, a byproduct of the forestry industry, is rich in phenolic compounds with antioxidant capacity. Among them, condensed tannins are particularly relevant due to their antioxidant potential and their role in bioadhesive development. Sustainable valorization strategies encourage the use of green technologies such as hydrothermal treatment, which avoids organic solvents.

Objective: To optimize the hydrothermal extraction of phenolic compounds from pine bark, with emphasis on condensed tannins and adhesive properties, and to compare the antioxidant capacity of the extracts with commercial antioxidants.

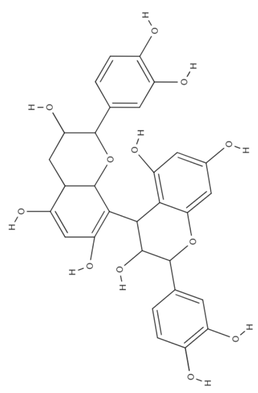

Figure 2 – Condensed tannin

Materials and Methods

A Box-Behnken experimental design was used to examine the effects of three factors (temperature, time, and solid/liquid ratio), with three replicates at the central point.

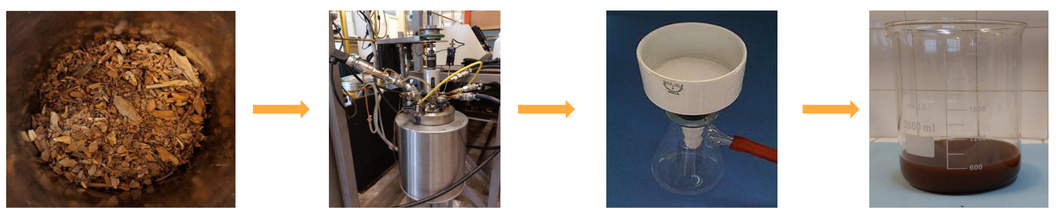

Figure 3 – Outline of the experimental work

The goodness-of-fit of the different models, was evaluated using analysis of variance (ANOVA) and regression coefficients (R^2). A multivariable optimization method was applied, focusing on maximizing extraction yield, condensed tannin content, and Stiasny number rather than optimizing all five responses simultaneously.

To compare with commercial antioxidants, antioxidant capacity was measured using FRAP, ABTS and DPPH assays.

Results

The experimental data were successfully fitted to quadratic models, which were used to generate the response surfaces (Figures 4–8). Based on these models and applying a multi-objective optimization approach, the optimal extraction conditions were identified as 130 °C, 1/20 g/mL, and 45 minutes. Under these conditions, three independent experiments were carried out for models' validation, and the results are summarized in Table 1.

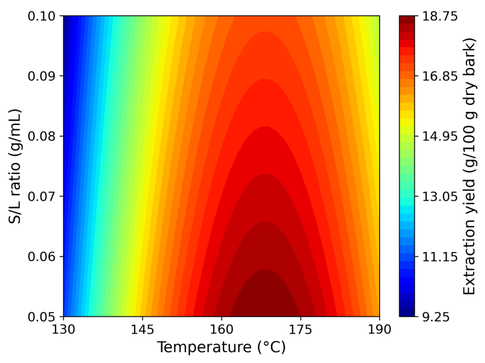

Figure 4 – Response surface of extraction yield as a function of temperature and solid/liquid ratio (at 15 min).

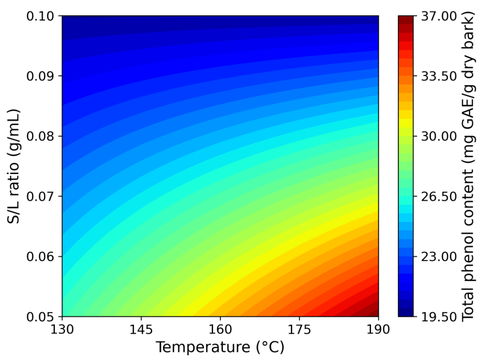

Figure 5 – Response surface of total phenol content as a function of temperature and solid/liquid ratio (independent of time).

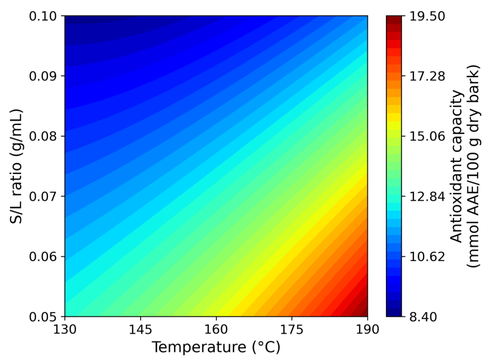

Figure 6 – Response surface of FRAP antioxidant capacity as a function of temperature and solid/liquid ratio (independent of time).

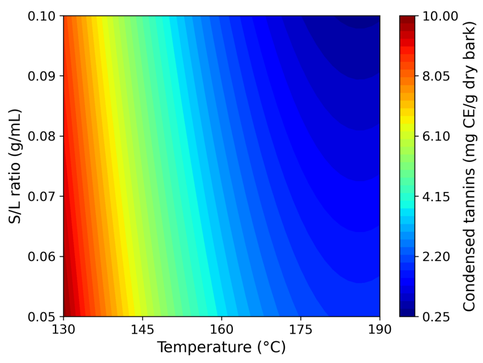

Figure 7 – Response surface of condensable tannin content as a function of temperature and solid/liquid ratio (at 15 min).

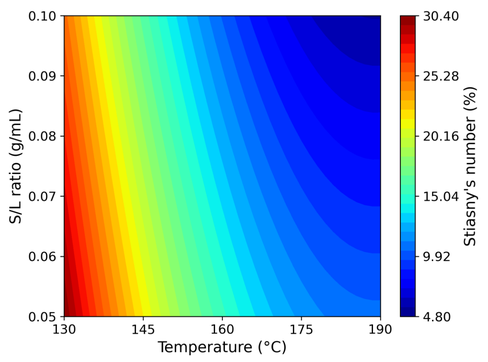

Figure 8 – Response surface of Stiasny's number as a function of temperature and solid/liquid ratio (at 15 min).

Table 1 – Model's validation on the optimal conditions

Response	Experimental data	Predicted value
Extraction yield (g/100 g dry bark)	13.54 ± 1.30	14.71 ± 2.22
Total phenol content (mg GAE/ g dry bark)	30.18 ± 0.59	26.87 ± 3.51
Antioxidant capacity (mmol AAE/ 100 g dry bark)	13.32 ± 2.80	12.77 ± 1.87
Condensed tannins (mg CE/ g dry bark)	6.79 ± 1.57	8.31 ± 1.93
Stiasny's number (%)	24.03 ± 4.27	26.23 ± 5.56

Table 2 shows the results of the comparison with commercial antioxidants

Table 2 – Comparison of antioxidant capacity

Antioxidant	FRAP (mmol AAE/g TSD)	ABTS (mmol TE/g TSD)	DPPH (mmol TE/g TSD)
Extract	1.02 ± 0.03^a	0.88 ± 0.07^a	0.54 ± 0.15^a
α-tocoferol	2.79 ± 0.41^b	1.92 ± 0.08^b	1.83 ± 0.33^b
BHA	7.68 ± 0.63^c	5.69 ± 0.61^c	3.35 ± 0.07^c

Values correspond to the mean with their 95% confidence intervals. Values with different letters are significantly different (Tukey's test, p < 0.05).

Conclusions

- Pine bark extracts obtained by **hydrothermal treatment** exhibited measurable **antioxidant capacity** and **potential adhesive properties**.
- Adhesive potential was supported by **condensed tannin content** and **Stiasny number**.
- Antioxidant capacity was **lower than BHA and α-tocopherol**, but the extracts are derived from a **sustainable and eco-friendly process**.
- These results highlight the extracts as a **promising alternative for industrial applications**, particularly in **bioadhesive development**.

Acknowledgments

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