Optimization of Subcritical Water Extraction of Bioactive Compounds from Vineyard Pruning Residues using Response Surface Methodology

Olena Dorosh[1], Manuela M. Moreira[1,*], Elsa F. Vieira[1], Andreia F. Peixoto[2], Cristina Freire[2], and Cristina Delerue-Matos[1]

[1]REQUIMTE/LAQV, Instituto Superior de Engenharia do Instituto Politécnico do Porto, Rua Dr. António Bernardino de Almeida, 431, 4249-015 Porto, Portugal

[2]REQUIMTE/LAQV, Departamento. de Química e Bioquímica, Faculdade de Ciências, Universidade do Porto, Rua do Campo Alegre s/n, 4169-007 Porto, Portugal

*manuela.moreira@graq.isep.ipp.pt

✓ Evaluation of the optimal Subcritical Water Extraction (SWE) conditions of antioxidants and polyphenols from vine-canes using Response Surface Methodology (RSM). For this purpose, a central composite design (CCD) was conducted to analyse the temperature (X1, 150–280 °C) and time (X2, 20–50 min) effects in Total Phenolic Content (TPC) and antioxidant activity (ABTS and FRAP).

Introduction

The cultivation of *Vitis* (*Vitaceae*) grape varieties is one of the most important economic activity in Portugal [1]. According to the International Organization of Vine and Wine, in 2017 the Portuguese vitiviniculture area cultivated was 193,672 hectares [2]. Therefore, one of the biggest challenges for wine-producing is to create alternatives for processing the vast amount of grape wastes generated during the harvest season. Vine-canes constitute one of the most abundant vineyard wastes, being estimated that for each hectare of vineyard 1,75 tons of vine-canes wastes are produced [3]. Traditionally, these vineyard pruning are used as a heating source or left on the ground, however using this raw material as a source of phenolic compounds could increase its economic value [1,3].

Materials and Methods

SAMPLE PRE-TREATMENT:

Vine-canes from Touriga Nacional variety (TN) used in this work were supplied by Sogrape and collected in Quinta dos Carvalhais. Vine-canes were oven-dried at 50°C for 24 h, and then milled to a particle size smaller than 1 mm.

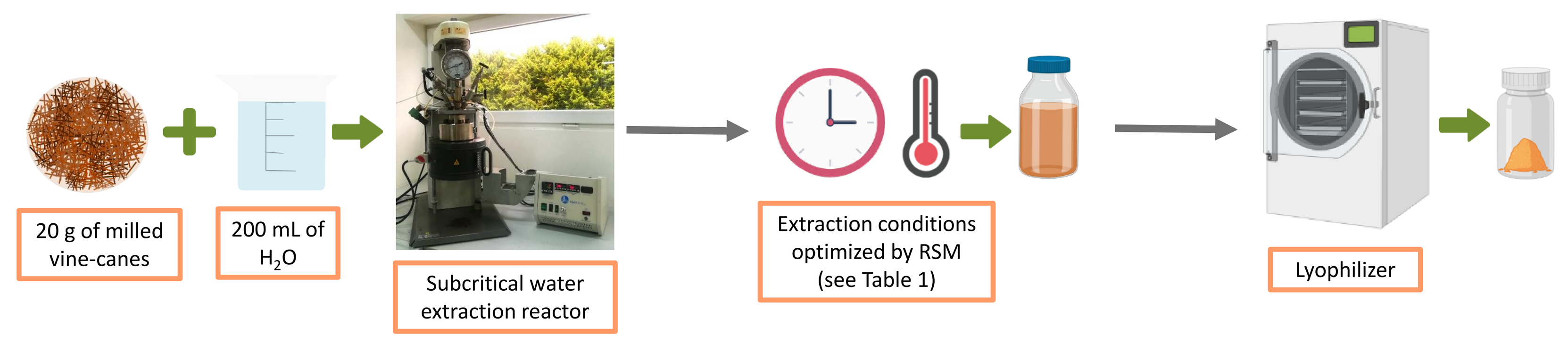
EXPERIMENTAL DESIGN AND STATISTICAL ANALYSIS:

The experimental design and statistical analysis were performed using the software Design Expert trial version 7 (Stat-Ease Inc., Minneapolis, MN, USA). The response surface methodology (RSM) and a Central Composite Design (CCD) were chosen to evaluate the effect of temperature (X1) and time (X2) of SWE extraction on the phenolic and antioxidant activity of TN vine-cane extracts. CCD consisted of a complete 22-factorial design as cubic points, with four axial points at a distance of α=1.414 from the design centre and five centre points. A total of 13 experiments were randomly performed (Table 1).

TOTAL PHENOLIC CONTENT AND ANTIOXIDANT ACTIVITY ASSAYS:

Total phenolic content (TPC) was determined as described in detail by Moreira et al. [1]. Antioxidant activity was evaluated by the ferric reducing antioxidant power (FRAP) and 2,2′-azino-bis(3-ethylbenzothiazoline-6-sulfonic acid) (ABTS) assays [1]. The results were expressed by gram of dry extract (dw).

Experimental Procedure



Extracts analysis

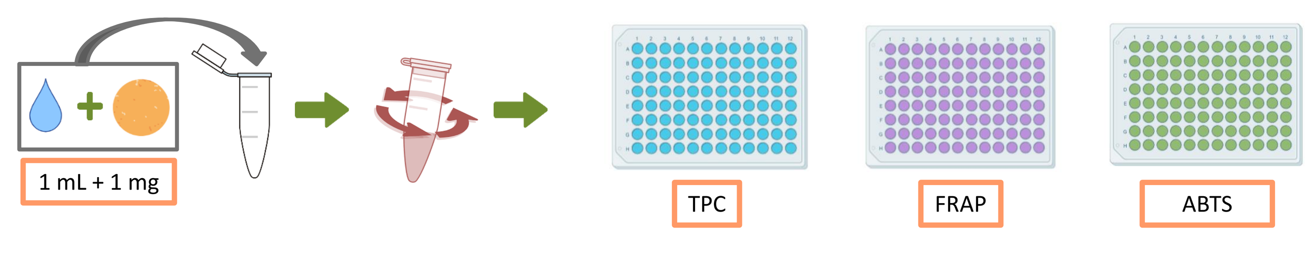


Table 1. Subcritical water extraction conditions tested

Experiment	Temperature / °C	Time / min
1	215	35
2	123	35
3	150	50
4	215	56
5	307	35
6	280	20
7	280	50
8	150	20
9	215	14
10	215	35
11	215	35
12	215	35
13	215	35

Results and Discussion

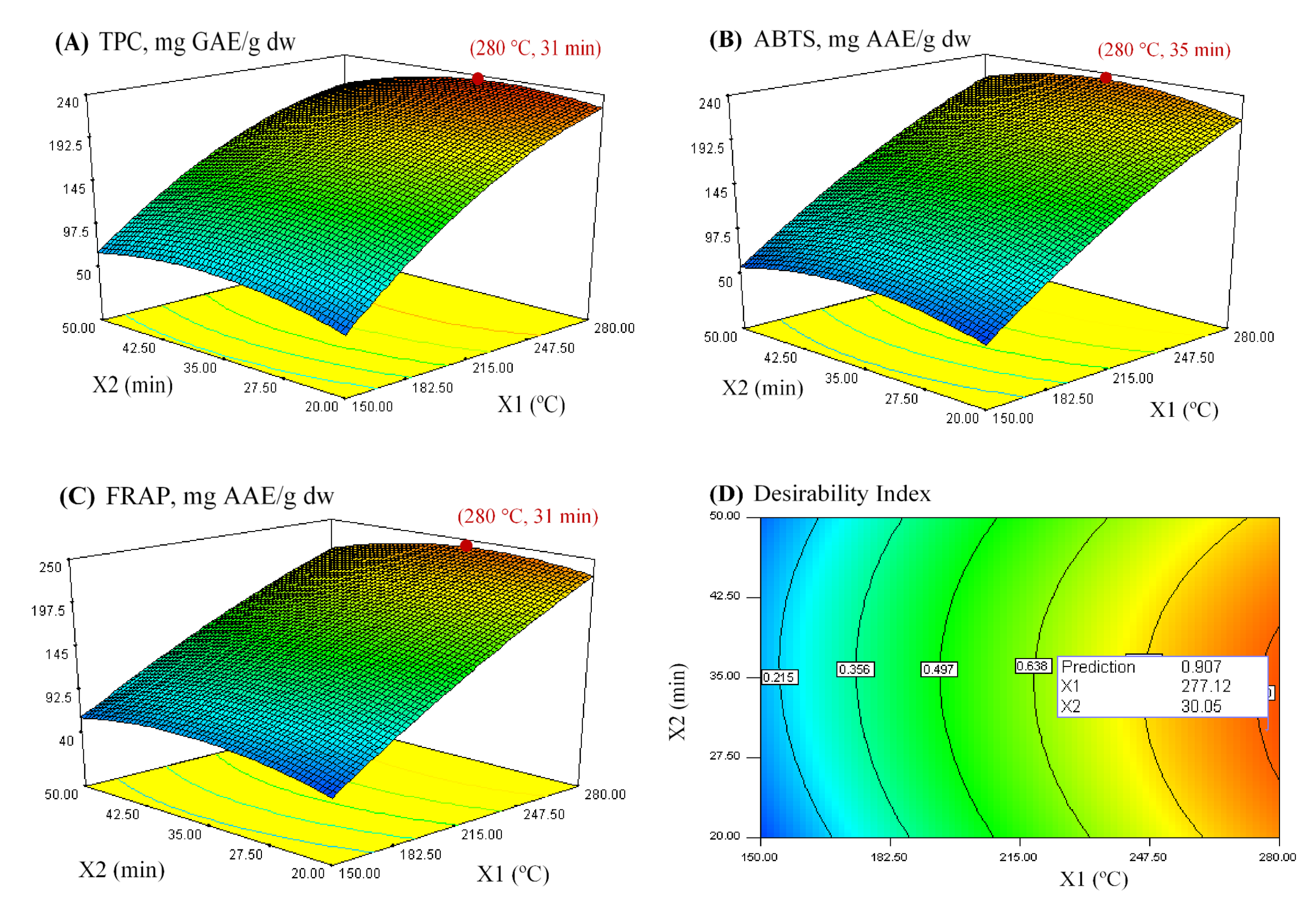


Figure 1. Response surface 3D and contour plots for the interaction effects of independent variables of temperature (X1, °C) and time (X2, min) on dependent variables of TPC (A), ABTS (B) and FRAP (C) and on the desirability index for combined responses (D) of vine-cane extract obtained through SWE. The optimum point (●) was identified on the response surface.

✓ Amongst the examined parameters, to maximize the phenolic content and antioxidant activity of the vine-cane extracts, the extraction temperature demonstrated to exerts the highest influence ($p < 0.05$).

✓ Obtained results showed that the optimal conditions were 33 min extraction time and 280 °C extraction temperature.

✓ The CCD model was adequate to describe the experimental data and for the prediction of the three studied parameters.

Comparison of extraction techniques

Method	CE [1]	MAE [1]	UAE lab scale [3]	UAE pilot scale [3]	SWE
Extraction conditions	ethanol:water 50:50 (v/v) 55 °C 120 minutes	ethanol:water 60:40 (v/v) 100 °C 20 minutes	ethanol:water 50:50 (v/v) 62 to 76 °C 60 minutes	ethanol:water 50:50 (v/v) 64 to 70 °C 60 minutes	water 280 °C 33 minutes
TPC (mg GAE/g dw)	14.1 ± 0.4	21.2 ± 1.0	20.1 ± 0.6	15.4 ± 1.4	33.9[a]

[a]Value converted considering the amount of dried extract obtained after lyophilizing the subcritical water extract.

Advantages

- SWE allows to extract approximately 2-fold more phenolic compounds than the CE technique.
- The method is an effective alternative to polyphenols recovery from vine-canes and a promising approach for a large-scale extraction.

Conclusions and Future Work

✓ The presented results exhibited that SWE technology is a time-saving, high yield, and bioactive environmentally friendly technique for obtaining phenolic compounds.

✓ The optimum SWE conditions are currently being employed to different vine-cane varieties collected at different years to select the extracts with the highest antioxidant activity, which can be further applied to food or cosmetic industries in substitution of synthetic antioxidants.

References

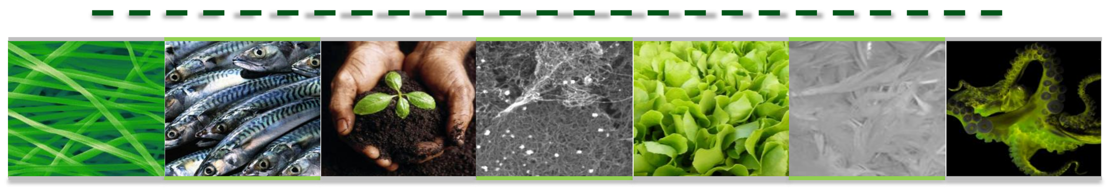
[1] Moreira MM, Barroso MF, Porto JV, Ramalhosa MJ, Švarc-Gajić J, Estevinho L, Morais S, Delerue-Matos C. Sci Total Environ, 2018, 634, 831-842.

[2] International Organization of Vine and Wine—OIV. http://www.oiv.int/en/international-organisation-vine-and-wine.

[3] Dorosh O, Moreira MM, Rodrigues F, Peixoto AF, Freire C, Morais S, Delerue-Matos C. Mol., 2020, 25, 1739.

Acknowledgements: This research was funded by FCT/MCTES through national funds (UIDB/50006/2020). This work was also financed by the FEDER Funds through the Operational Competitiveness Factors Program—COMPETE and by National Funds through FCT within the scope of the project "PTDC/BII-BIO/30884/2017—POCI-01-0145-FEDER-030884". O.D. is thankful for the research grant from project PTDC/BII-BIO/30884/2017—POCI-01-0145-FEDER-030884. M.M.M. (project CEECIND/02702/2017), E.F.V. (project CEECIND/03988/2018) and A.F.P. (DL57/2016–Norma transitória) are grateful for the financial support financed by national funds through FCT—Fundação para a Ciência e a Tecnologia, I.P. and to REQUIMTE/LAQV. The supply of the vineyard pruning is acknowledged to Sogrape, S.A.

10º Encontro da Divisão de Química Analítica
26 – 28 October 2020, Lisboa, Portugal



REQUIMTE/LAQV – INSTITUTO SUPERIOR DE ENGENHARIA DO PORTO
Rua Dr. António Bernardino de Almeida, 431
4200-072 Porto, Portugal

www.requimte.pt/laqv | www.graq.isep.ipp.pt