Optimization of Pulsed Electric Fields-Assisted Extraction of phenolic compounds from white and red grape pomace using Response Surface Methodology

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Grapes of the cultivars Vitis vinifera are the basis of most globally produced wines, with an annual production that, in 2018, has overcome 77 million tons (OIV, 2019). However, during the winemaking process several grape processing by-products are generated, including grape stalks, pomace (skins and seeds), and wine lees/sediments (Sirohi *et al*., 2020). Among them, grape pomace are the major by-products generated in the winemaking process, with a production of 20 kg for each hectoliter of wine, representing about 20-25% of the processed raw material (Chowdhary *et al*., 2021).

Factors such as consumer awareness, changing lifestyles, and ascending search for natural ingredients over synthetic ones, are likely to positively influence the demand for natural ingredients, driving the global natural extracts market. Therefore, the search for natural, and low-cost alternative sources of natural ingredients, represents the top priority among manufacturers.

To this purpose, the valorization of grape pomace might improve the sustainability of wine production and create economic and social benefits, especially through the recovery of bioactive compounds with high commercial significance, for their potential applications.

However, the key factor that is behind the recovery of bioactives from these plant cells is represented by an efficient permeabilization of the cell membrane, which constitutes a physical barrier that hinders the recovery of the target intracellular compounds from plant residues via conventional solvent extraction techniques. This is motivating scientists to explore the use of cell disruption pre-treatment of plant residues that induces weakening or rupture of cell envelops, thus enhancing the extractability of target intracellular compounds from plant matrices, such as winery by-products, with reduced solvent, time, and energy consumption (Barba *et al*., 2015b).

To this purpose, pulsed electric fields (PEF) is gaining great interest as gentle and scalable cell disruption technique of plant biomass, showing great potential to intensify the selective recovery of target intracellular compounds from various plant matrices derived from fruit and vegetable, while lowering the energy and solvent consumption, and reducing the treatment time (Frontuto *et al*., 2019; Pataro *et al*., 2020). However, so far, only few works demonstrated the feasibility of PEF technology to intensify the recovery yield of phenolic compounds from winery by-products (Corrales *et al*., 2008; Barba *et al*., 2015a; Boussetta *et al*., 2009; Brianceau *et al*., 2015).

Moreover, in order to maximize the benefits and advantages of PEF-assisted extraction over the conventional SLE, an optimization step of the whole PEF-assisted extraction process, should be carried out. Nevertheless, to the best of our knowledge, only in few works response surface methodology (RSM) was used as a tool for the optimization of the main variables involved only in the conventional SLE process, in order to maximize the extractability of bioactive compounds from grape by-products (Melo *et al.,* 2015; Caldas *et al.,* 2018; Kwiatkowski *et al.,* 2020).

However, as per literature survey, no study was addressed to the optimization of the whole PEF-assisted extraction applied on grape pomace, utilizing protocols to optimize all the factors involved in the PEF pre-treatment and in the subsequent SLE.

Therefore, this study was focused on the optimization of the pulsed electric fields (PEF)-assisted extraction process using response surface methodology (RSM) with the aim to sustainably intensify the extractability of phenolic compounds from white and red grape pomace. The cell disintegration index (Zp) was used as response variable to identify the optimal PEF pre-treatment conditions of grape pomace in terms of field strength (E = 0.5 - 5 kV/cm) and energy input (W_T = 1 - 20 kJ/kg), to be applied prior to the subsequent solid-liquid extraction (SLE) process. SLE for both untreated and PEF-treated samples was optimized to determine the most effective combination of extraction temperature (20-50 °C), extraction time (30-300 min), and solvent concentration (0–100% ethanol in water). Total phenolic content (TPC), flavonoid content (FC), and antioxidant activity (FRAP) of the obtained extracts from red and white grape pomace, and total anthocyanin content (TAC) and tannin content (TC) from red grape pomace, were determined. The extracted compounds from untreated and PEF-treated samples at the optimal conditions were analyzed via HPLC-PDA analysis.

Results revealed that, at a fixed extraction temperature (50 °C), the application of PEF at optimal processing conditions (E=4.6 kV/cm, WT=10 kJ/kg - E=3.8 kV/cm, WT=10 kJ/kg, for red and white grape pomace, respectively) and prior to SLE has the potential to reduce, the solvent consumption (3-12%) and shorten the extraction time (23-103 min) to achieve the same recovery yield of phenolic compounds. Under optimized conditions, the extracts obtained from PEF-treated red and white grape pomace showed higher TPC (2-8%), FC (24-31%), and FRAP (9-36%) values, TAC (19%), and TC (30%) for red grape pomace, as compared to the control extraction. HPLC analyses revealed that epicatechin, p-coumaric acid, and quercetin were among the main phenolic compounds extracted, and no degradation phenomena occurred due to PEF application.

Overall, these results encourage the integration into a winery of the PEF technology as a suitable pretreatment of grape pomace, demonstrating the feasibility of the innovative approach in developing a sustainable extraction process and supporting the possible reintegration of grape processing by-products in the food supply chain.

Barba, F. J., et al. (2015a) Effect of Alternative Physical Treatments (Ultrasounds, Pulsed Electric Fields, and High-Voltage Electrical Discharges) on Selective Recovery of Bio-compounds from Fermented Grape Pomace. Food and Bioprocess Technology 8, 1139–1148. doi:10.1007/s11947-015-1482-3.

Barba, F. J., et al. (2015b) New Approaches for the Use of Non conventional Cell Disruption Technologies to Extract Potential Food Additives and Nutraceuticals from Microalgae. Food Engineering Reviews 7, 45–62. doi:10.1007/s12393-014-9095-6.

Boussetta, N., et al. (2009) Electrically assisted extraction of soluble matter from chardonnay grape skins for polyphenol recovery. Journal of Agricultural and Food Chemistry 57, 1491–1497. doi:10.1021/jf802579x.

Brianceau, S., et al. (2015) Combined densification and pulsed electric field treatment for selective polyphenols recovery from fermented grape pomace. Innovative Food Science and Emerging Technologies 29, 2–8. doi:10.1016/j.ifset.2014.07.010.

Caldas, T. W., et al. (2018) Phenolic compounds recovery from grape skin using conventional and non-conventional extraction methods. Industrial Crops and Products 111, 86–91. doi:10.1016/j.indcrop.2017.10.012.

Chowdhary, P., et al. (2021) Current trends and possibilities for exploitation of Grape pomace as a potential source for value addition. Environmental Pollution 278, 116796. doi:10.1016/j.envpol.2021.116796.

Corrales, M., et al., (2008) Extraction of anthocyanins from grape by-products assisted by ultrasonics, high hydrostatic pressure or pulsed electric fields: A comparison. Innovative Food Science and Emerging Technologies 9, 85–91. doi:10.1016/j.ifset.2007.06.002.

Frontuto, D., et al. (2019) Optimization of Pulsed Electric Fields-Assisted Extraction of Polyphenols from Potato Peels Using Response Surface Methodology. Food and Bioprocess Technology 12, 1708–1720. doi:10.1007/s11947-019-02320-z.

Kwiatkowski, M., et al. (2020) Response surface parallel optimization of extraction of total phenolics from separate white and red grape skin mixtures with microwave-assisted and conventional thermal methods. *Journal of Cleaner Production* 251, 119563. doi:10.1016/j.jclepro.2019.119563.

Melo, P. S., et al. (2015) Winery by-products: Extraction optimization, phenolic composition and cytotoxic evaluation to act as a new source of scavenging of reactive oxygen species. *Food Chemistry* 181, 160–169. doi:10.1016/j.foodchem.2015.02.087.

OIV, International Organization of Vine and Wine (2019). Statistical report on world vitiviniculture. 2019 Statistical Report on World Vitiviniculture, 23. http://www.oiv.int/public/medias/5029/world-vitiviniculture-situation-2016.pdf. [Accessed March 10, 2022].

Pataro, G., et al. (2020) Recovery of lycopene from industrially derived tomato processing by-products by pulsed electric fields-assisted extraction. Innovative Food Science and Emerging Technologies 63, 102369. doi:10.1016/j.ifset.2020.102369.

Sirohi, R., et al. (2020) Green processing and biotechnological potential of grape pomace: Current trends and opportunities for sustainable biorefinery. Bioresource Technology 314, 123771. doi:10.1016/j.biortech.2020.123771.