Isolation of high-added-value products from grape marc of the plant Vitis vinifera L.

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INTRODUCTION

During vinification and the treatment of *Vitis vinifera L.* species fruits, byproducts such as grape marc and wine bottom sludges are produced in large quantities. More specifically, grape marc, consisted of the skin and seeds of the grapes, is usually disposed to the environment without any further treatment [1-3]. The high organic load contained in the grapes, combined with the large amounts produced every year, make necessary their treatment before its disposal to the environment. On the other hand, grapes toxicity is attributed to their high content in phenolics, known for their high antioxidant activity. Several treatment techniques, including physicochemical processes, are used to reduce the organic load of grapes with simultaneous isolation of phenolics, which are of high interest and high added value for cosmetic industry, food industry and pharmaceutics [4].

The present work is focused on the experimental investigation of the parameters during the extraction of phenolic compounds from grapes from the Merlot variety, minimizing the amount of extracted carbohydrates. The experimental results obtained from the parametric analysis of the extraction process, were tested in a pilot scale experiment using a membrane system consisting of an Ultrafiltration (UF), a Nanofiltration (NF), and a Reverse Osmosis (RO) membrane. More specifically, the product obtained from the extraction, was further treated with the pilot-scale membrane system and the final product was characterized considering its Total Phenolic Content (TPC), Total Sugar Content (TSC) and antioxidant capacity with the FRAP method.

MATERIALS AND METHODS

In the first stage, the extraction conditions of the TPC and TSC from Merlot grapes were investigated. The investigated parameters concerned the successive extractions of the raw material and their effect on the recovery of phenolic compounds combined with the reduced content in sugars. The parameters that led to the optimum results were applied to the pilot-scale extraction and the resulting extract was further processed using a membrane system implementing a UF membranes, two NF membranes with different molecular weight cut-off (MWCO), namely NF600 and NF300, and finally an RO.

Considering the results of previous parametric studies [4], the effect of successive extractions of the same raw material was investigated as well as the possibility of enriching the ethanolic extract by using it for the extraction of phenols from an additional amount of raw material. Thus, the effect of adding a new solid to the same solvent (a process in which the extract is enriched) and the combination of extractions, initially with water and then with 50% ethanol, were studied. The goal of this type of extraction was the removal of carbohydrates at the first stage, through aqueous extraction and the isolation of an ethanolic extract with an improved TSC/TPC ratio. When investigating the enrichment of the extract, the raw material was initially extracted with a solid/solvent ratio of 1/10 (w/v), during which, 30 g of grape marc were extracted with 300 mL EtOH 50% v/v, with an extraction time of 60 min, at ambient temperature and under 150 rpm stirring. Then, the ethanolic extract was obtained by removing the solids from the mixture. An amount of solid was then added again in 225 mL of the ethanolic extract, at a ratio of 1/10 w/v (22.5 g of grape marc) and re-extraction was carried out. At the end of the extraction, the solid was removed, while the extract (II-ethanol) was used for a third extraction of 18 g of grape marc, under the same extraction conditions. After the third extraction, the extract (III-ethanol) with a volume of 131 mL was obtained. TPC and TSC were measured in all three extracts. Based on these results, the extraction of grape marc was carried out in a pilot scale.

RESULTS AND DISCUSSION

During the parametric study for the enrichment of the ethanolic extract, the aim was to investigate the ability of a certain amount of solvent to extract the phenolic components of the grape marc after three consecutive additions of solid. The rationale for this study was to minimize the use of ethanol. The main results can be observed in Figure 1.

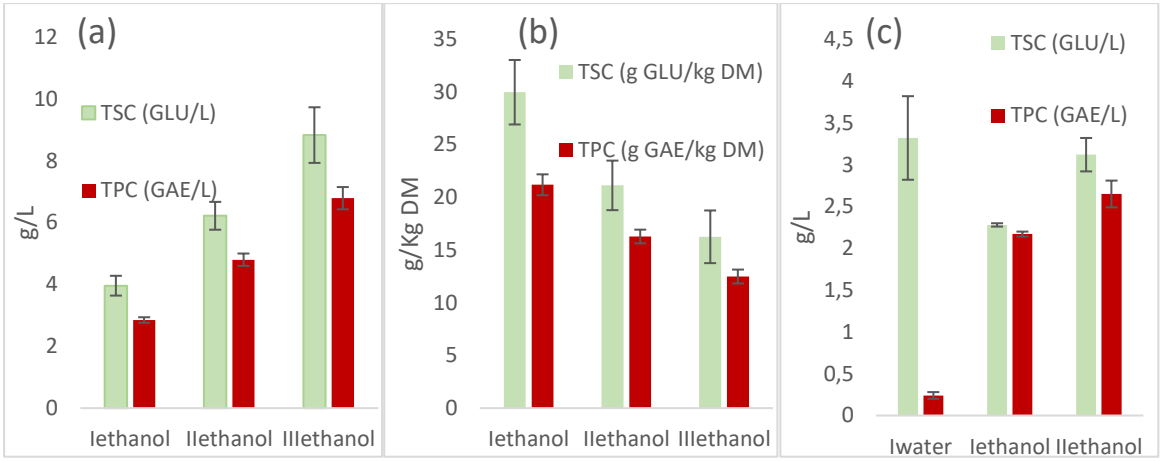

Figure 1: Concentration of TPC (red) and TSC (green) (a) after 3 step extraction, (b) g/Kg DM values in each extraction separately, and (c) values after successive extractions with water, EtOH 50% and EtOH 50%.

The successive use of the same solvent for the extraction of three batches of fresh grape marc led to the enrichment of the solvent to the extracted phenols (Figure 1.a). On the other hand, when the results are analyzed in the efficiency of each extraction step individually, the successive extraction steps slightly diminished the quantity of phenols extracted from the grape marc, as it was expected, due to the increased concentration of the extracted compounds in the bulk of the solvent. Finally, the use of water as an extraction solvent seems to favor the removal of carbohydrates, leading to higher purities of extracted phenols on the next stages of the processes, when ethanol is used for the extraction.

Using the above-mentioned process, the extraction was carried out at pilot scale. The total extract obtained after the three-step ethanol extraction, had a volume of 18 L. On further processing of the 18 L of ethanolic extract through pilot-scale membranes, the extract was diluted to 127 L to reduce the concentration of ethanol and facilitate the use of the large-scale equipment. 127 L was the volume of the UF feed stream (UF initial). Leading to 29 L UF retentate, 20 L of NF600 retentate, 15 L of NF300 retentate, were collected. 10 L of RO retentate and 64 L of RO permeate were obtained. Some discrepancies in the volume balance of the process were attributed to the water retention in the piping system of the pilot-scale equipment.

CONCLUSION

The phenolic compounds contained in grape marc of the Merlot variety were successfully extracted using ethanolic solutions as solvent, with successive uses of the same solvent to extract three fresh batches of grape marc increasing the concentration of the extracted phenols. As expected, the efficiency of extraction per step was slightly reduced in terms of extracted phenols per g of grape marc. The extraction process was implemented in pilot scale and the final extract was fractionated using in-line filtration (UF, NF600, NF300, RO).

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