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Optimization of the procedure for the liquid-liquid extraction (LLE) of phenolic compounds from extra-virgin olive oil

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Abstract

Olive oil possess several minor components with important activities from the biological point of view, like the phenolic compounds, which are capable of scavenging free radicals. The aim of this work was to optimize the technique of liquid-liquid extraction of the phenolic compounds from olive oil, employing methanol:water and ethanol:water as extraction solvents, and also to compare both mixtures to find the one able to extract the highest phenolic contents. Two binary mixture designs were performed, one for the methanol:water mixture and the other for the ethanol:water mixture. The total phenolic content was used as the designs' response. The optimal extraction condition was a ratio of 100:0 (v/v) for the methanol:water as well as for the ethanol:water mixture. After the extraction of an olive oil sample with both the optimal conditions, it was obtained a total phenolic content of 101.06 ± 6.54 $mg.kg^{-1}$ for the methanol:water mixture and of 79.95 ± 0.01 $mg.kg^{-1}$ for the ethanol:water mixture. Therefore, it was determined that the mixture of methanol:water in a 100:0 (v/v) ratio was able to extract the highest total phenolic content in olive oil samples.

Key words: Olive oil, phenolic compounds, optimization.

Introduction

Extra-virgin olive oil is a product of high nutritional quality and its minor chemical constituents are related to several health benefits. Among these components, the phenolic compounds possess high capacity of scavenging free radicals. For the analysis of phenolic compounds in olive oils, the most employed extraction technique is the liquid-liquid extraction (LLE), which works by adding hexane to the sample and then performing the phenolics extraction through a partition with a methanol:water solution (60:40, v/v). Despite its wide use, there are no studies which evaluate if other solvent ratios could be more effective, as well as if other less toxic solvents, like ethanol, could yield to similar results. Therefore, the aim of this work was to optimize the phenolic compounds extraction of olive oil, employing methanol:water and ethanol:water as extraction solvents, and also to compare both combinations to obtain the one able to extract the highest phenolic contents.

Results and Discussion

The extraction optimization was realized through the binary mixture design, which allow the obtainment of mathematical models to calculate the optimal extraction condition. Two designs of 13 experiments each were performed, one for the methanol:water mixture and the other for the ethanol:water mixture. Total phenolic content (Folin-Ciocalteu method) was evaluated as the designs' response. Only the linear model for the total phenolic content of the design with methanol presented significant regression and absence of lack of fit, and then could be used to predict the optimal condition. After the optimization procedure, the optimal condition predicted by the model for the methanol:water mixture was 100:0 (v/v). The optimal condition for the ethanol:water mixture was obtained by the surveillance of the empirical results, and also corresponded to a 100:0 (v/v) ratio. Both optimized conditions were tested again in the olive oil sample, presenting a total phenolic content of 101.06 ± 6.54 $mg.kg^{-1}$ for the methanol:water mixture (100:0, v/v) and of 79.95 ± 0.01 $mg.kg^{-1}$ for the ethanol:water mixture (100:0, v/v). Thus, it was determined that the methanol:water mixture, in a 100:0 (v/v) ratio, is able to extract the highest content of phenolic compounds, and then should be used for the extraction of these compounds from samples of olive oils.

Conclusions

The use of binary mixture designs allowed the optimization of the phenolic compounds extraction of olive oils with mixtures of methanol:water and ethanol:water, and the procedure with 100% of methanol was the most efficient, resulting in the highest total phenolic contents extraction.

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