Analytical evaluation of fining treatments for white wines contaminated by Ochratoxin A

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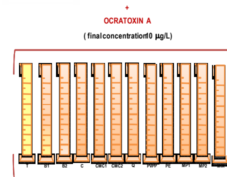
INTRODUCTION

Mycotoxins are toxic secondary metabolites produced by certain molds. Ochratoxin A (OTA) is one of the most relevant. Its chemical structure is a dihydro-isocoumarin connected at the 7-carboxy group to a molecule of L-β-phenylalanine via an amide bond. OTA in wine is a risk to consumer health [1]. According to the Regulation No. 123/2005 of the European Commission, the maximum limit for OTA in wine is 2 μg/kg [2]. Then, it is important to control its occurrence. So, the aim of this work was to know the effect of different fining agents on OTA removal from white wine.

MATEREAL AND METHODS



Wine	2013
Alcohol (% v/v)	10.4
Volumic mass at 20°C (g/mL)	0.9917
Total acidity (g/L tartaric acid)	6.8
pH	3.14
Volatile acidity (g/L acetic acid)	0.16

+ OCRATOXIN A (final concentration 10 μg/L)

Fining experiments - 11 commercial fining agents (sodium bentonite, calcium bentonite; potassium caseinate; carboxymethylcellulose; chitosan; polyvinylpolypyrrolidone-; pea protein ; mannoproteins ; mixture composed by gelatin, bentonite and activated carbon) were used to get new approaches on OTA removal from wine. Trials were performed in wines artificially supplemented (at a final concentration of 10 μg/L) with OTA.

OTA analysis - was performed according to [3]. Briefly, 2 mL of the centrifuged (4000 rpm, 15 min). supernatant were collected and added of an equal volume of acetonitrile/methanol/acetic acid (78:20:2 v/v/v). The solid fractions obtained after fining, were centrifuged (4000 rpm, 15 min), the resulting supernatant discarded, and the pellet extracted with 1 mL of the above solution and 1 mL of H_2O. After 12 h, the extracts were filtered through a syringe filter (0.45 μm) and stored at 4 °C until analyzed by HPLC with fluorescence detection. The chromatographic separation was performed on a C18 reversed-phase YMC-Pack ODS-AQ analytical column (250 x 4.6 mm I.D., 5 mm), fitted with a pre-column with the same stationary phase. The samples were eluted at a flow rate of 1 mL/min during 20 min with a mobile phase consisting of water/acetonitrile/acetic acid (99:99:2 v/v/v). The injection volume was 50 μL and parameters for detection: λexc = 333 nm, λem = 460 nm and gain = 1000. The OTA retention time was approximately 16 min. The OTA concentration in the samples was determined by comparison of peak areas with a calibration curve made with standards of OTA (Sigma-Aldrich).

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RESULTS AND DISCUSSION

The most effective fining agent in removing OTA was a commercial formulation that contains activated carbon, a well-known adsorbent of mycotoxins (Figure 1). Removals between 10-30% were obtained with potassium caseinate, mannoproteinsand pea protein. With bentonites, carboxymethylcellulose, polyvinylpolypyrrolidone and chitosan no considerable OTA removal was verified.

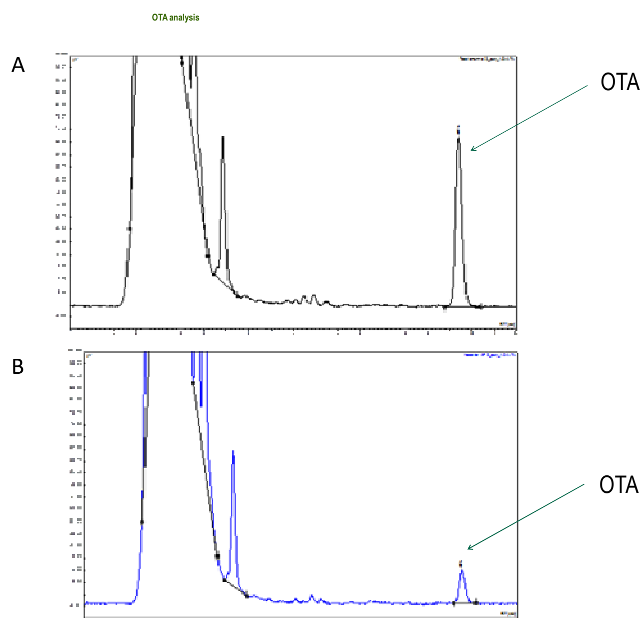

Figure 1. (A) Chromatogram from the white wine without treatment (B) chromatogram from the white wine treated with a commercial formulation that contains activated carbon.

These results may provide useful information for winemakers, namely in the selection of the most appropriate enological product for OTA removal, reducing the toxicity and simultaneously enhancing food safety and wine quality.

ACKNOWLEDGEMENTS

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