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An effective approach to mitigating ochratoxin A (OTA) levels in wine with minor impact on wine quality

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Abstract. Ochratoxin A (OTA) levels in wine are well-documented, with red wines typically having the highest concentrations, followed by rosé and white wines. In Europe, wine is the second-largest source of OTA intake after cereals. The maximum allowable OTA limit in wine is 2 µg/L, according to Regulation (EC) No. 1881/2006. While activated carbon shows potential for reducing OTA, its efficiency in wine, especially in red wine, is limited by the complexity of the wine matrix. In white wine, up to 98.3% OTA reduction was achieved with 1 g/L activated carbon. In red wines, the reduction ranged from 32% to 61%, depending on the physicochemical properties of the carbons. This study evaluates different enological deodorizing activated carbons for OTA removal, with a focus on their impact on wine quality. Complete OTA removal was achieved in white wines with most activated carbons, whereas only one type of carbon was effective in red wines, likely due to anthocyanins competing for adsorption sites. The results suggest that optimizing pore size distribution in activated carbon can effectively remove OTA from both white and red wines with minimal impact on wine color.

1. Introduction

Mycotoxins, naturally produced by fungi, can cause toxic effects when ingested through contaminated food. These compounds have attracted global attention due to their significant economic impact, particularly concerning human health and international food trade [1,2]. Ochratoxin A (OTA), one of the most widely distributed mycotoxins, is produced as a secondary metabolite by certain filamentous fungi from *Aspergillus* and *Penicillium* genera. In Europe, wine is the second most significant source of OTA exposure, following cereals [3]. Due to its various toxic effects, including carcinogenic, genotoxic, immunotoxic, and hepatotoxic properties [4,5], OTA has been classified by the International Agency for Research on Cancer [6] as a possible human carcinogen (Group 2B). Consequently, as a leading wine producer and consumer, Europe has established a maximum OTA limit of 2 µg/L in wine, as outlined in European Commission Regulation 1881/2006 and subsequent regulations [7].

Fungal invasion occurs when berries are damaged by injuries, cracks, or lesions caused by powdery mildew or esca, leading to splitting. *Aspergillus* and *Penicillium* species are commonly found in grapes [8]. These fungi produce ochratoxins, particularly OTA, which can subsequently be detected in wine [9]. The primary source of OTA contamination in grapes, raisins, and wine is fungi from the *Aspergillus* genera, including species like *A. carbonarius*, and *A. niger* [10]. Several factors, such as temperature, pH, moisture content, and water activity, influence the growth of OTA-producing fungi. Temperature is especially critical, with the optimal ranges for OTA production being 10–20 °C for *A. carbonarius* and 20–25 °C for *A. niger* [11,12].

Due to its better adaptation to extreme high temperatures and dry conditions, *A. niger* is expected to become more prevalent than *A. carbonarius* with climate change [13]. Although *A. niger* produces OTA less frequently [14,15], rising temperatures in moderate climates are likely to result in higher OTA concentrations.

The concentration of OTA in wine is influenced by multiple factors, including climate (especially temperature and relative humidity in the month leading up to harvest), grape-growing practices such as the use of fungicides, the extent of berry damage before maceration, and the maceration process itself [16,17]. Furthermore, OTA levels in wine have been found to vary with the latitude of the production region, with wines made from grapes grown at lower latitudes showing higher incidence and concentration of OTA contamination [16]. Enological practices and good manufacturing processes during winemaking are also critical in determining the final OTA levels in wine [17,18]. Certain wine characteristics, such as whether a wine is dry or sweet, and whether it is red or white, have also been observed to influence OTA levels. Studies indicate that red wines typically have higher OTA levels [16,19,20], often due to the extended contact between grape skins and juice during alcoholic fermentation, which is longer in red wines compared to white wines [20,21,22]. Similarly, sweet wines tend to have higher OTA levels, likely due to their later harvest and extended time on the vine, increasing the potential for exposure to OTA-producing fungal contaminants [19].

To effectively prevent fungal growth in plants and protect consumer health, it is essential to implement good agricultural practices that focus on preventing OTA formation. However, if OTA contamination does occur, one strategy for its removal is the use of adsorbing agents. Various enological products have been tested for OTA removal in wine, including activated carbon, bentonite, potassium caseinate, casein, egg albumin, chitin, chitosan and zeolite [23,24]. Despite these efforts, OTA reduction has generally been relatively low, and in many cases, the treatment negatively impacted wine quality, affecting parameters such as color, phenolic compounds content, and anthocyanins [17,24,25,26].

Among the various adsorbents studied by Dumeau and Trione [27], activated carbon and potassium caseinate have shown the highest effectiveness. Activated carbon can remove approximately 90% of OTA from red wine, while potassium caseinate achieves an 82% reduction. However, caution is needed when using activated carbon, as it also removes anthocyanins and other polyphenols from the wine. Activated carbon derived from cherry stones has been found to reduce OTA levels in wine by up to 50% without significantly affecting the overall polyphenolic index or color intensity [28].

Adsorption using physicochemical agents has emerged as a promising strategy for controlling OTA contamination, offering significant advantages in mycotoxin management [23,24,29]. However, the challenge of selective adsorption remains a potential limitation.

The study assesses various enological deodorizing activated carbons for their effectiveness in removing OTA, with a particular focus on their impact on wine quality.

2. Material and Methods

2.1. Wine

A commercial white wine and red wine were used in the study, with their physicochemical characteristics outlined in Table 1.

Table 1. Wine physicochemical parameters.

	White wine	Red wine
Alcohol strengths (%v/v)	10.4	13.4
Titratable acidity (g/L tartaric acid)	6.8	5.0
pH	3.14	3.49

2.2. Vegetable-Origin Activated Carbons

Activated carbons derived from vegetable sources, labeled C1 through C7, were supplied by SAI Enology Lda. Their characteristics, including surface area, porosity, and pore-size distribution, are described in detail by Filipe-Ribeiro et al. [30,31].

2.3. Experimental Design

White and red wines were spiked with OTA to a final concentration of 10 µg/L. Activated carbons were then applied at a dosage of 1 g/L, the maximum allowed for white wines according to the International Code of Enological Practices. Control samples of red and white wines without added activated carbon were also prepared. All experiments were conducted in duplicate.

2.4. OTA Analysis by HPLC with Fluorescence Detection

After treatment with the various activated carbons, wine samples were centrifuged at 4000 rpm for 15 minutes. A 2 mL portion of the supernatant was then mixed with an equal volume of acetonitrile/methanol/acetic acid (78:20:2, v/v/v) and left to stand for 12 hours. The extracts were filtered through a 0.45 µm nylon syringe filter, stored at 4 °C, and analyzed by HPLC with fluorescence detection. The HPLC system used consisted of a Varian Prostar 210 pump, a 410 autosampler, a Jasco FP-920 fluorescence detector, and a YMC-Pack ODS-AQ C18 column. Elution was carried out at a flow rate of 1 mL/min for 20 minutes using a water/acetonitrile/acetic acid (99:99:2 v/v/v) mixture. Detection parameters were set λex = 333 nm and λem = 460 nm, with a gain = 1000. The OTA retention time was approximately 12 minutes. OTA concentrations were calculated using a calibration curve ranging from 0.05 to 6.0 µg/L. Recovery rates were 97.2% for white wine and 92.4% for red wine. The limits of detection (LOD) and quantification (LOQ) were 0.05 µg/L and 0.09 µg/L for white wine, and 0.08 µg/L and 0.11 µg/L for red wine. All analyses were performed in duplicate.

2.5. Color, Pigments, Chromatic characteristics, Total phenols, Flavonoids and Non-flavonoids

The color of the white wine and the color intensity and hue of the red wine were quantified according to O.I.V. methods [32]. The concentration of total and colored anthocyanins, as well as total and polymeric pigments in the red wine, were determined using the Somers and Evans method [33]. Wine chromatic characteristics were calculated using the CIELab method as outlined by the OIV [32]. Chroma [$C^* = [(a^*)^2 + (b^*)^2]^{1/2}$] and the hue-angle [$h^o = tang^{-1}(a^*/b^*)$] were also calculated. To more precisely differentiate color, the color difference was determined using the following expression: $\Delta E^* = [(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2]^{1/2}$. The phenolic content of the wines was measured by absorbance at 280 nm, both before and after the precipitation of favonoid phenols using formaldehyde, as described by Kramling and Singleton [34] and Ribéreau-Gayon et al. [35]. This allowed for the quantification of flavonoid, non-favonoid, and total phenols in the wines, with results expressed as gallic acid equivalents based on calibration curves with standard gallic acid. All analyses were performed in duplicate.

2.6. Anthocyanins, Catechin and Phenolic acids by HPLC

Analyses were performed using an Ultimate 3000 Dionex HPLC system equipped with a PDA-100 photodiode array detector and an Ultimate 3000 Dionex pump. Separation was carried out on a C18 column (250 mm × 4.6 mm, 5 µm particle size, ACE, Scotland) at a flow rate of 1 mL/min and a temperature of 35 °C. The injection volume was 50 µL, with detection wavelengths ranging from 200 to 650 nm. The mobile phase consisted of 5% aqueous formic acid (A) and methanol (B). The gradient elution was programmed as follows: 5% B from 0 to 5 min, followed by a linear increase to 65% B until 65 min, and then a decrease back to 5% B from 65 to 67 min [30,31, 36].

2.7. Statistical Analysis

Partial Least Square (PLS) analysis was employed to identify the key characteristics of activated carbons for OTA removal from white and red wines by establishing a structure-efficiency relationship between the pore volume size distributions of the activated carbons and their OTA removal efficiencies. This method was also used to evaluate the removal of anthocyanins and simple phenolic compounds by the same activated carbons. PLS regression is advantageous because it uses the information in the Y variables to build prediction models, can handle more variables than objects in the data, and avoids issues of collinearity among variables. Cross-validation was used to determine the optimal number of components to include, based on the number of PLS components with the lowest prediction error sum of squares (PRESS). PLS-R calculations were performed using the XLSTAT-v2006.06 software package (Addinsoft, Inc).

3. Results and discussion

The effectiveness of OTA removal by activated carbons varied depending on the wine matrix. All carbons, except C2, completely removed OTA from white wines. In contrast, the carbons were less efficient in red wines, with only C3 achieving full removal (Figure 1). This suggests that the structural properties of the carbons are particularly important in the more complex red wine matrix. A significant correlation was found between OTA removal efficiency and mesopore volume ($r = 0.889$, $p < 0.0031$). The high efficiency of activated carbon C3 can be attributed to its distinct physicochemical properties, notably its higher mesopore volume compared to most other activated carbons, except for C7. Although C7 had a similar mesopore volume to C3, its OTA removal efficiency was lower, indicating that mesopore volume alone does not fully explain C3 superior efficiency. The correlation between OTA removal and mesopore volume suggested that adsorption capacity is influenced by the accessibility of OTA to the inner surface of the adsorbent, which depends on pore size and competition with other components for these available pores. The higher efficiency observed in white wine compared to red wine, both containing 10 µg/L of OTA, may be due to the higher phenolic content, especially anthocyanins, in red wine, which can compete with OTA for adsorption sites.

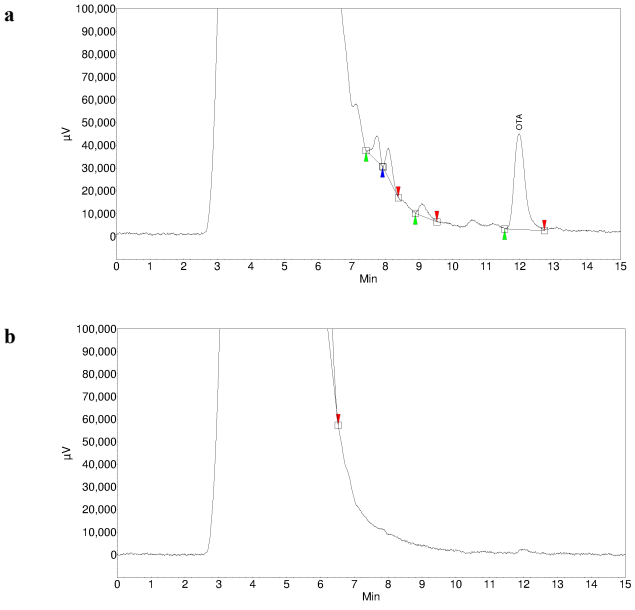


Figure 1. RP-C18 chromatogram and fluorescence detection of OTA in spiked (10 µg/L) red wine (a) and after treatment with activated carbon C3 (b).

To evaluate the impact of different activated carbons on wine quality after OTA removal, the color, chromatic properties, and phenolic composition of both white and red wines were analyzed.

In white wines, the use of activated carbons led to a significant reduction in color, with C3 and C7 causing the most substantial decrease. This reduction in color correlated with lower levels of total phenols and non-flavonoid phenols (Table 2). Both C3 and C7 produced a color difference greater than 4. However, since reducing

brown-yellow hues is often beneficial for white wine from a sensory perspective, the use of these activated carbons is unlikely to adversely affect its visual appearance.

Table 2. Significant correlations between the color and chromatic characteristics of white wines and their phenolic composition after treatment with the different activated carbons.

	White wine color (Abs $_{420\ nm}$)	White wine L*	White wine b*
Total Phenols	r=0.887 *p*<0.0033	r=-0.741 *p*<0.035	r=0.890 *p*<0.0031
Non-flavonoid Phenols	r=0.767 *p*<0.026	r=-0.780 *p*<0.022	r=0.780 *p*<0.022

L* (lightness), b*(yellowness)

In red wines, although the activated carbons were primarily intended for deodorization, a significant reduction in color intensity was observed across all types, particularly with C3 and C7. This reduction in color was associated to decreases in total phenols, flavonoid phenols, total anthocyanins, colored anthocyanins, and polymeric pigments (Table 3). The application of C3 and C7 resulted in the most pronounced color difference approximately 7. While these changes might be noticeable to the human eye and could be perceived as unfavorable by consumers, the overall color intensity of the wines decreased by only 24%.

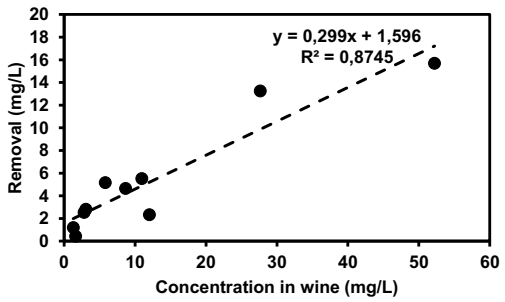
Table 3. Significant correlations between the color and chromatic characteristics of red wines and the phenolic composition after treatment with the different activated carbons.

	Red wine color intensity	Red wine L*	Red wine a*
Total Phenols	r=0.799 p<0.017	r=-0.844 p<0.0084	r=-0.859 p<0.0063
Flavonoid Phenols	r=0.815 p<0.014	r=-0.860 p<0.0062	r=-0.874 p<0.0045
Total Anthocyanin's	r=0.918 p<0.0013	r=-0.915 p<0.0014	r=-0.915 p<0.0014
Colored Anthocyanin's	r=0.941 p<0.00049	r=-0.959 p<0.00017	r=-0.950 p<0.00030
Polymeric Pigments	r=0.999 p<0.00001	r=-0.985 p<0.00001	r=-0.981 p<0.000017

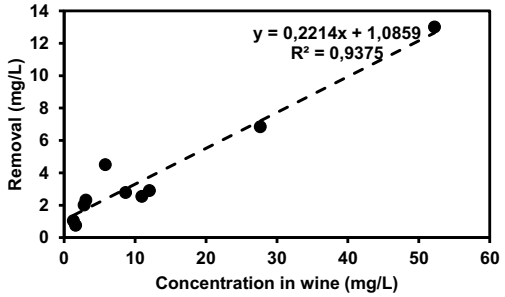
L* (lightness), a*(redness)

The phenolic compound most significantly affected in white wines was *trans*-caftaric acid, followed by coutaric acid. Together, these two compounds accounted for 41% (with C4) to 62% (with C3) of the total phenolic compounds removed. The removal extent of various phenolic acids and catechin by the activated carbons was correlated with their initial concentration in the white wine (Figure 2).

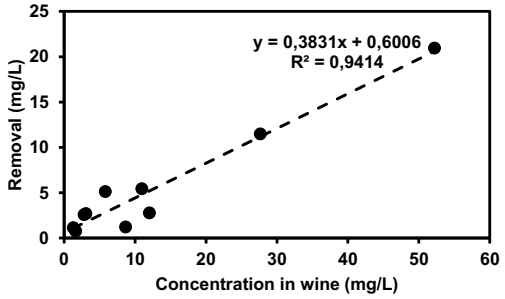
C1



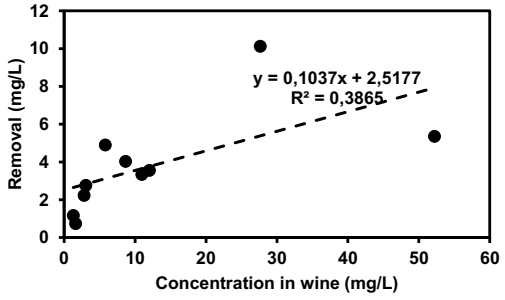
C2



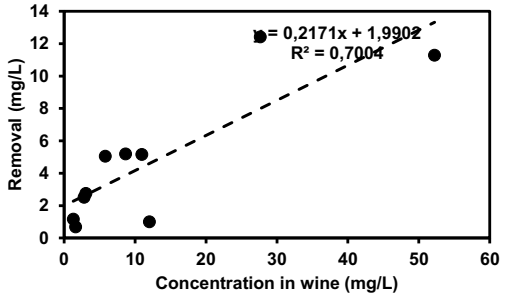
C3



C4



C5



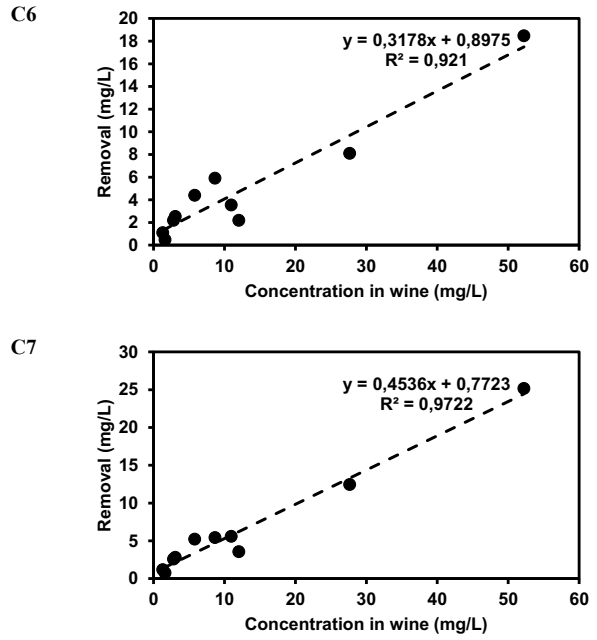
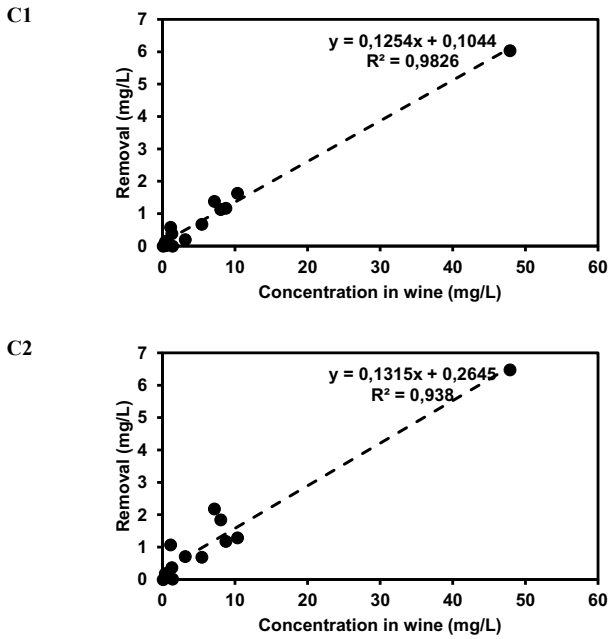


Figure 2. Removal of individual phenolic acids and catechin from white wine by activated carbons (C1-C7) relative to their initial concentrations in the wine.

In red wines, the reduction of phenolic compounds by activated carbons was less pronounced than in white wines. However, smaller quantities of phenolic acids, catechin, and anthocyanins were still affected. Similar to the findings in white wines, the extent of removal for these compounds in red wines was correlated with their initial concentrations (Figures 3 and Figure 4).



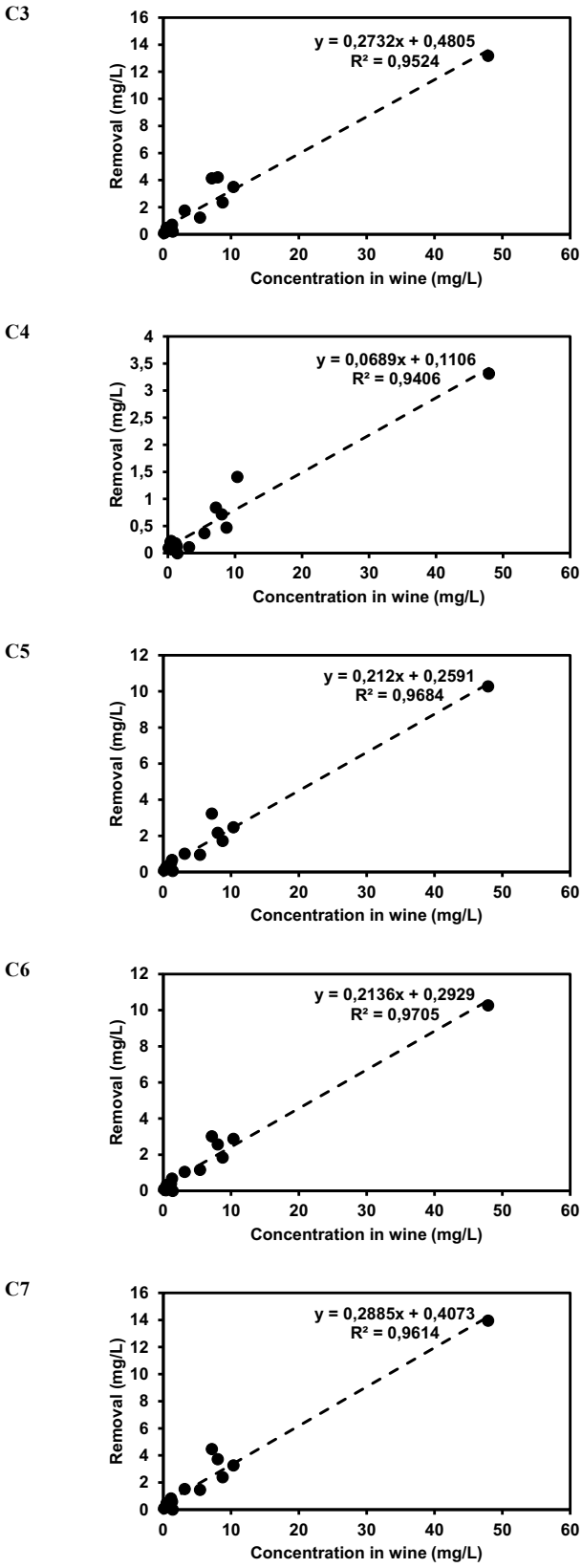


Figure 3. Removal of individual anthocyanins from red wine by activated carbons (C1-C7) based on their concentrations in the wine.

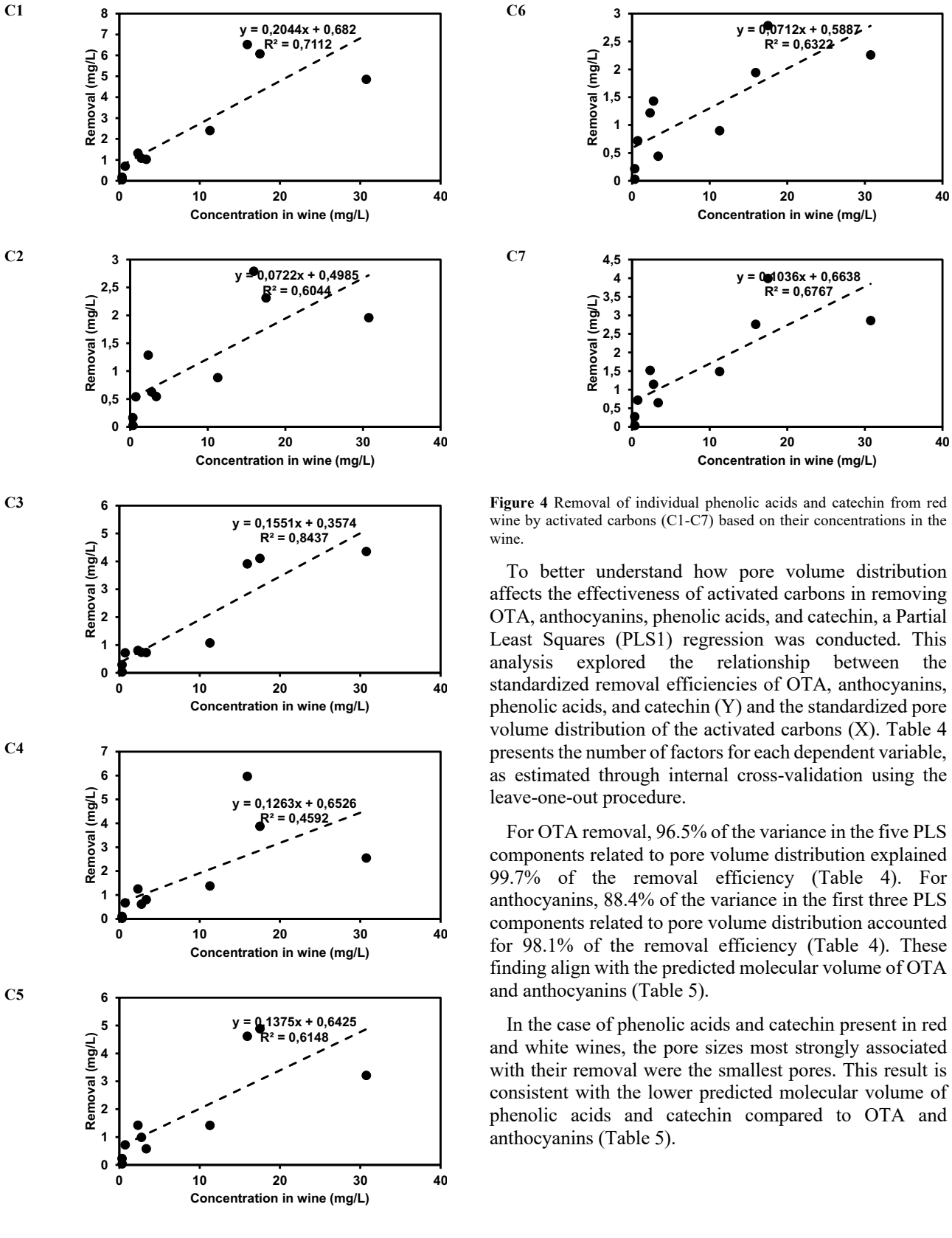


Figure 4 Removal of individual phenolic acids and catechin from red wine by activated carbons (C1-C7) based on their concentrations in the wine.

To better understand how pore volume distribution affects the effectiveness of activated carbons in removing OTA, anthocyanins, phenolic acids, and catechin, a Partial Least Squares (PLS1) regression was conducted. This analysis explored the relationship between the standardized removal efficiencies of OTA, anthocyanins, phenolic acids, and catechin (Y) and the standardized pore volume distribution of the activated carbons (X). Table 4 presents the number of factors for each dependent variable, as estimated through internal cross-validation using the leave-one-out procedure.

For OTA removal, 96.5% of the variance in the five PLS components related to pore volume distribution explained 99.7% of the removal efficiency (Table 4). For anthocyanins, 88.4% of the variance in the first three PLS components related to pore volume distribution accounted for 98.1% of the removal efficiency (Table 4). These finding align with the predicted molecular volume of OTA and anthocyanins (Table 5).

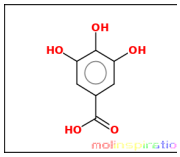
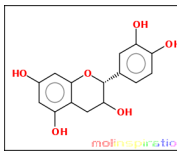
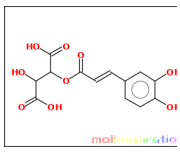
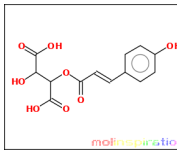
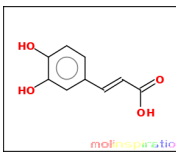
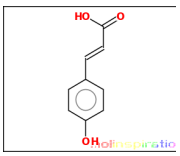
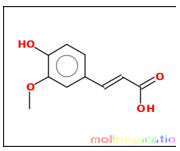
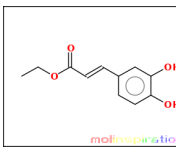
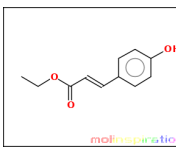
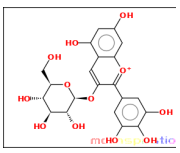
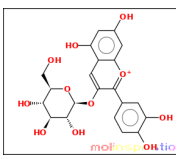
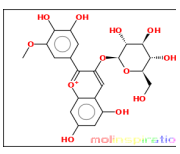
In the case of phenolic acids and catechin present in red and white wines, the pore sizes most strongly associated with their removal were the smallest pores. This result is consistent with the lower predicted molecular volume of phenolic acids and catechin compared to OTA and anthocyanins (Table 5).

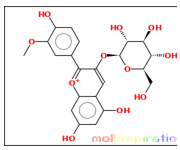
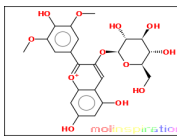
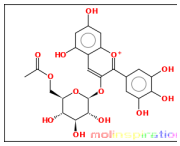
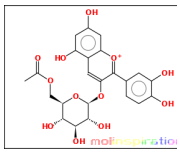
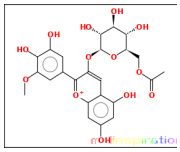
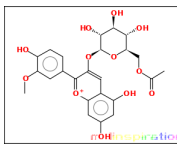
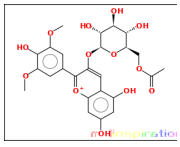
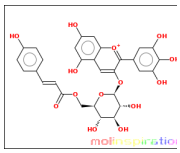
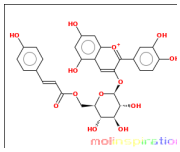
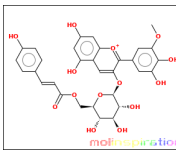
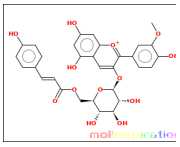
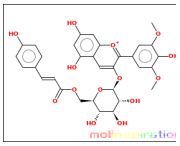
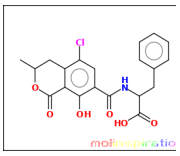
Table 4. PLSl regression of individual variables *versus* the pore volume distribution descriptive variables

	PC#	OTA	Anthocyanins	Phenols Red	Phenols White
Nº PC		5	3	3	3
% Var Y	1	75.29	77.31	68.79	78.13
	2	13.60	18.49	15.04	7.78
	3	4.52	2.38	2.94	2.67
	4	5.52	-	-	-
	5	1.61	-	-	-
Total		99.74	98.12	86.76	88.58
%Var X	1	87.27	87.80	71.57	83.11
	2	3.30	2.16	5.92	5.69
	3	2.60	2.63	2.04	1.48
	4	2.06	-	-	-
	5	1.26	-	-	-
Total		96.49	92.58	79.52	90.28
Q^2 (cum)		0.896	0.884	0.821	0.491

[a] Optimal number of principal components for construction of the PLS model determined by leave-one-out cross validation

Table 5. Predicted molecular volume, octanol-water partition coefficient (P), Topological polar surface area (TPSA) and Equivalent spherical radius using Molinspiration software (https://www.molinspiration.com/)

Name	Structure	Properties
Gallic acid		MW = 170.12 g/mol LogP = 0.59 TPSA = 97.98 Volume = 135.10 Å^3 Spherical radius=3.18 Å
Catechin		MW = 290.27 g/mol LogP = 1.37 TPSA = 110.37 Volume = 244.14 Å^3 Spherical radius = 3.88 Å
Caftaric acid		MW = 312.23 g/mol LogP = -0.61 TPSA = 161.59 Volume = 251.14 Å^3 Spherical radius = 3.91 Å
Coutaric acid		MW = 296.23 g/mol LogP = -0.12 TPSA = 141.36 Volume = 243.12 Å^3 Spherical radius=3.87 Å
Caffeic acid		MW = 180.16 g/mol LogP = 0.94 TPSA = 77.75 Volume = 154.50 Å^3 Spherical radius = 3.33 Å
p-Coumaric acid		MW = 164.16 g/mol LogP = 1.43 TPSA = 57.53 Volume = 146.48 Å^3 Spherical radius = 3.27 Å^3
Ferulic acid		MW = 194.19 g/mol LogP = 1.25 TPSA = 66.76 Volume = 172.03 Å^3 Spherical radius = 3.45 Å
Ethylcaffeate		MW = 208.21 g/mol LogP = 1.93 TPSA = 66.76 Volume = 188.83 Å^3 Spherical radius = 3.56 Å
Ethylcoumarate		MW = 192.21 g/mol LogP = 2.42 TPSA = 46.53 Volume = 180.81 Å^3 Spherical radius = 3.51 Å
Delphinidin-3-glucoside		MW = 465.39 g/mol LogP = -3.08 TPSA = 211.69 Volume = 374.95 Å^3 Spherical radius = 4.47 Å
Cyanidin-3-glucoside		MW = 449.39 g/mol LogP = -2.79 TPSA = 191.46 Volume = 366.93 Å^3 Spherical radius = 4.44 Å
Petunidin-3-glucoside		MW = 479.41 g/mol LogP = -2.78 TPSA = 200.70 Volume = 392.48 Å^3 Spherical radius = 4.54 Å

Peonidin-3-glucoside		MW = 463.42 g/mol LogP = -2.49 TPSA = 180.47 Volume = 384.46 Å^3 Spherical radius = 4.51 Å
Malvidin-3-glucoside		MW = 493.44 g/mol LogP = -2.47 TPSA = 189.70 Volume = 410.00 Å^3 Spherical radius=4.61 Å
Delphinidin-3--(6''-acetylglucoside)		MW = 507.42 g/mol LogP = -2.38 TPSA = 217.77 Volume = 411.46 Å^3 Spherical radius = 4.61 Å
Cyanidin-3--(6''-acetylglucoside)		MW = 491.43 g/mol LogP = -2.09 TPSA = 197.54 Volume = 403.44 Å^3 Spherical radius=4.58 Å
Petunidin-3--(6''-acetylglucoside)		MW = 521.45 g/mol LogP = -2.07 TPSA = 206.77 Volume = 428.99 Å^3 Spherical radius = 4.68 Å
Peonidin-3--(6''-acetylglucoside)		MW = 505.45 g/mol LogP = -1.78 TPSA = 186.54 Volume = 420.97 Å^3 Spherical radius = 4.65 Å
Malvidin-3-(6''-acetylglucoside)		MW = 535.48 g/mol LogP = -1.76 TPSA = 195.78 Volume = 446.52 Å^3 Spherical radius = 4.74 Å
Delphinidin-3-(6''-*p*-coumaroyl) glucoside		MW = 611.53 g/mol LogP = -0.72 TPSA = 238.00 Volume = 501.74 Å^3 Spherical radius=4.93 Å
Cyanidin-3-(6''-*p*-coumaroyl) glucoside		MW = 595.53 g/mol LogP = -0.43 TPSA = 217.77 Volume = 493.72 Å^3 Spherical radius = 4.90 Å
Petunidin-3-(6''-*p*-coumaroyl) glucoside		MW = 625.56 g/mol LogP = -0.41 TPSA = 227.00 Volume = 519.27 Å^3 Spherical radius = 4.99 Å
Peodinin-3-O-(6''-*p*-coumaroyl) glucoside		MW = 609.56 g/mol LogP = -0.12 TPSA = 206.77 Volume = 511.25 Å^3 Spherical radius = 4.96 Å
Malvidin-3-O-(6''-*p*-coumaroyl) glucoside		MW = 639.59 g/mol LogP = -0.10 TPSA = 216.01 Volume = 536.80 Å^3 Spherical radius=5.04 Å
Ochratoxin A		MW = 403.82 g/mol LogP = 1.74 TPSA = 112.93 Volume = 336.34 Å^3 Spherical radius=4.31 Å

4. Conclusions

The results of this study demonstrate that activated carbons, when used within the application levels authorized by the International Code of Enological Practices for wine, can effectively eliminate OTA in both white and red wines, provided they possess the appropriate structural characteristics. The structural requirements for complete OTA removal are more critical in red wines than in white wines due to the presence of anthocyanins, which compete with OTA for access to the mesopores in the activated carbon. Therefore, selecting activated carbons with an optimized pore size distribution is a highly promising strategy for mitigating the safety risks posed by this mycotoxin in wines, even at elevated OTA levels. This approach can effectively remove OTA from both white and red wines while having minimal impact on wine color.

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