Food Chemistry 268 (2018) 463–467

Contents lists available at ScienceDirect

Food Chemistry

journal homepage: www.elsevier.com/locate/foodchem

Bottle capsules as a barrier against airborne 2,4,6-trichloroanisole

Andrii Tarasov[a,*], Doris Rauhut[b], Rainer Jung[a,*]

[a] Department of Enology, Hochschule Geisenheim University, Von-Lade-Straße 1, 65366 Geisenheim, Germany
[b] Department of Microbiology and Biochemistry, Hochschule Geisenheim University, Von-Lade-Straße 1, 65366 Geisenheim, Germany

ARTICLE INFO

Keywords:
Wine
Capsule
2,4,6-Trichloroanisole (TCA)
Barrier
Ethylene vinyl alcohol copolymer (EVOH)

ABSTRACT

The possibility of 2,4,6-trichloroanisole (TCA) migration through synthetic stoppers and into wine from highly contaminated air was shown by several authors. However, those experiments were usually conducted without bottle capsules, which are a common part of wine packaging. In the current study, we demonstrated that the presence of capsules (without open holes) above synthetic stoppers can reduce wine contamination by airborne d_5-TCA by about 10 times or more. Generally, metallic capsules revealed better barrier properties than polyvinyl chloride counterparts. Application of EVOH film on the external surface of the polyvinyl chloride capsules usually resulted in a lower level of wine contamination. Additionally it was demonstrated, that relatively short exposure (3 months) of the bottles to highly contaminated air could cause a considerable absorption of d_5-TCA by synthetic stoppers, which can subsequently lead to wine contamination after 12 months.

1. Introduction

Organoleptic taints in wine cause considerable annual losses in the wine industry. Musty/mouldy defect remains among the most numerous wine faults nowadays. The presence of various compounds could lead to this wine fault, e.g.: geosmin, 2-methylisoborneol, 1-octene-3-one, 1-octene-3-ol, 2-methoxy-3,5-dimethylpyrazine (Sefton & Simpson, 2005). However, the main contributors to the musty/mouldy wine taint usually are haloanisoles, particularly, 2,4,6-trichloroanisole (TCA). The importance of TCA as a cause of this wine defect was identified in the early 1980s (Buser, Zanier, & Tanner, 1982; Tanner, Zanier, & Buser, 1981). This compound is characterized by extremely low perception threshold in wine starting from 1.5 to 2 ng/l (Sefton and Simpson, 2005; Soleas, Yan, Seaver, & Goldberg, 2002). Other haloanisoles such as 2,4-dichloroanisole, 2,6-dichloroanisole, 2,3,4,6-tetrachloroanisole, pentachloroanisole possess higher perception thresholds in wine and rarely play a key role in the musty/mouldy taint (Chatonnet & Labadie, 1995; Sefton & Simpson, 2005). Nevertheless, these contaminants in combination with TCA can magnify the perception of the wine defect.

Cork stoppers are the most recognized and studied source of TCA and other haloanisoles in wine. Being formed according to different pathways (Simpson & Sefton, 2007), haloanisoles are extracted by wine from the cork material during the aging process. Therefore, "cork taint" became a conventional name for the musty/mouldy wine fault. Precursors of TCA and haloanisoles in cork material are halophenols such as 2,4,6-trichlorophenol (TCP) and pentachlorophenol (PCP). These substances are still abundant in the nature because of the intense utilization of PCP-based biocides in the past. Nowadays these biocides are forbidden in many countries around the world. Analysis of haloanisoles and halophenols content in cork material can be performed by different chromatographic, bioanalytic and sensorial methods (Tarasov, Rauhut, & Jung, 2017).

Formation of haloanisoles in cork material usually occurs at moist conditions by various filamentous fungi as a result of *O*-methylation of halophenols. *Penicillium*, *Fusarium* and *Trichoderma* strains were found to be able to carry out this bioconversion at high and moderate levels (Álvarez-Rodríguez et al., 2002; Coque, Álvarez-Rodríguez, & Larriba, 2003). Besides cork material, formation of haloanisoles according to this mechanism can take place also in different wooden objects inside a wine cellar: roof constructions, pallets, wooden barrels etc. (Chatonnet, Bonnet, Boutou, & Labadie, 2004; Chatonnet, Guimberteau, Dubourdieu, & Boidron, 1994). In addition to agricultural biocides, wood preservatives and fireproofing agents could serve as a source of halophenols in the wooden materials, including 2,4,6-tribromophenol. The latter is a precursor of 2,4,6-tribromoanisole (TBA), which was also identified as a contributor to the musty/mouldy wine taint (Chatonnet et al., 2004). Besides that, application of chlorine containing agents, which are still in use in some wine cellars, could cause a formation of halophenols and haloanisoles in the wooden materials (Chatonnet, 2004; Jung & Schaefer, 2010).

Once TCA and other haloanisoles are formed in the wood, they can easily migrate to the air inside the wine cellar and be absorbed by equipment and different enological materials: plastic hoses, filter

* Corresponding authors.
E-mail addresses: andrii.tarasov@hs-gm.de (A. Tarasov), doris.rauhut@hs-gm.de (D. Rauhut), rainer.jung@hs-gm.de (R. Jung).

https://doi.org/10.1016/j.foodchem.2018.06.118
Received 17 February 2018; Received in revised form 21 June 2018; Accepted 23 June 2018
Available online 23 June 2018
0308-8146/

sheets, bentonite, bottle closures etc. Subsequently, wine can contact these materials and extract TCA during the production process (Schaefer, 2014). Even minor migration of contaminant to the wine can cause problems because of the very low perception threshold of TCA. The importance of this “non-cork” pathway of musty/mouldy wine contamination by haloanisoles was underestimated until recent time.

The possibility of wine contamination by the air pollutants already after the bottling was studied by different authors (AWri, 2011; Lopes et al., 2011; Pereira et al., 2013). This mechanism involves migration of exogenous TCA (or another taint compound) from the contaminated atmosphere through the closures to the wine. The mentioned studies showed similar results. Cork and agglomerated stoppers revealed excellent barrier properties against exogenous TCA. No contaminant was found in the corresponding wines even after 30 months of bottles storage in the d_5-TCA polluted atmosphere (Pereira et al., 2013). Also, direct application of d_5-TCA solution on the top of the cork and agglomerated stoppers did not lead to wine contamination (Capone, Skouroumounis, & Sefton, 2002) At the same time, synthetic stoppers, BVS screw caps and crown caps were able to permeate certain amounts of haloanisoles to the wine, which often exceeded the perception thresholds (AWri, 2011; Lopes et al., 2011; Pereira et al., 2013). Despite that, the concentration of TCA in the air during the described experiments was usually higher than is expected under real conditions, these studies revealed permeation properties of various closures in principle. However, the conducted experiments normally did not involve the usage of capsules on the bottle necks sealed with the stoppers.

Capsules are a common part of packaging of commercial wine bottles with all types of stoppers. Therefore, in the current work we aimed to study the ability of capsules to serve as an additional barrier against airborne TCA. The experiment was performed with synthetic stoppers since they were shown to be able to permeate certain amount of exogenous contaminant. Three types of commercially available and most common capsules were examined: polyvinyl chloride (PVC) capsules, aluminum-polylaminate (Al-pol) capsules and tin capsules. Additionally, the efficiency of EVOH protective film application on the external surface of PVC capsules was tested.

2. Materials and methods

2.1. Chemicals and other materials

The following chemicals were used for experiments and analyses: d_5-TCA (CDN Isotopes, Canada); ethanol (Martin und Werner Mundo OHG, Germany); sodium chloride (Roth, Germany); ethylene vinyl alcohol (EVOH) copolymer Excavel HR-3010 (Kuraray Europe GmbH). Metallic containers of about 125 l capacity for the bottles storage were supplied by Bayern Fass, Germany. Parafilm “M”® was purchased from Carl Roth Karlsruhe, Germany.

2.2. Wine and materials for bottling

White wine from Rheingau region (Germany) 2015 vintage with following characteristics was used: alcohol content 12.2% (v/v), titratable acidity 7.7 g/l, sugar content 1.9 g/l, pH 3.1.

Bottles made of green transparent glass (750 ml) with the finish type “Cork” produced by “Verallia” were supplied by Saint Gobain – Oberland, Germany. Commercially available synthetic stoppers of one type produced by extrusion technology with the following parameters were used for the experiment: diameter 23.5 mm, length 43 mm, “medium + ” oxygen transmission rate. Three types of capsules were employed: polyvinyl chloride (PVC) capsules (65 mm length; two holes on the top), aluminum-polylaminate Al-pol capsules (65 mm length; two holes on the top); tin capsules (65 mm length; two holes on the top).

2.3. Preparation of EVOH solution

20 g of dry EVOH copolymer Excavel HR-3010 was placed into a 300 ml flask and 200 ml of water was added. Then mixture was gradually heated up to 95 °C with a reflux condenser and intensive stirring on the magnetic stirrer. After 2 h the heat source was switched off and the transparent solution of polymer was slowly cooled down to 40–45 °C with a slow stirring. Then 50 ml of EVOH water solution was transferred to the 100 ml flask and 25 ml of ethanol was added by little portions (about 2 ml per minute) with a stirring on the magnetic stirrer in order to prevent polymer agglomeration. The solution that was obtained was stored in the sealed flask. The solution was used for the experiment during the first week before any changes occurred. After about 1 month light haze in the polymer solution could be formed and intensified over time.

2.4. Bottling process

The bottling process was performed on the Hochschule Geisenheim University facilities (Germany) in December 2015. Treatment of the bottles with SO_2 solution preceded the bottling. 60 Bottles were filled with the wine at level 55 ± 10 mm from the top of the bottle necks. Afterwards synthetic stoppers were inserted without prior application of inert gases or vacuum using a corker machine GAI 4040.

2.5. Application of capsules on the bottle necks

48 bottles were sealed with three types of capsules: 24 bottles with PVC capsules; 12 bottles with Al-pol capsules and 12 bottles with tin capsules. PVC capsules were applied by a standard technique which implies shrinking of the capsule around the bottle neck under the action of a hot air (machine produced by Clemens). Al-pol and tin capsules were fastened by the rolling machine Otto Sick (VDE 530).

All the capsules possessed holes on their tops. Each hole on all the capsules was covered by a small aluminum folia piece (3–4 mm × 3–4 mm) and placed under the layer of EVOH solution served as a glue (Fig. 1). When EVOH film was formed, local treatment of the covered holes with EVOH solution was repeated until good sealing was achieved. Additionally, the whole external treatment of the capsules by EVOH solution was done for a group of 12 bottles with PVC capsules. Application of EVOH solution on the capsules was performed by means of a paintbrush. After several hours when a solid EVOH film was formed, the complete surface treatment of the same 12 PVC capsules was repeated until a solid film was obtained. Then all 60 bottles were kept in a vertical position until the next day when the installation of the experiment was assigned.

2.6. Design of the experiment

Four metallic containers of about 125 l capacity were used as a

Fig. 1. Sealing of the holes on the top of the capsules by aluminum folia pieces and EVOH polymer (example of Al-pol capsule).