Effect of Closure, Packaging and Storage Conditions on Impact Odorants of Wine

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Abstract: 3-alkyl-2-methoxypyrazines (MPs) represent an important and potent class of odor-active compounds associated with wine quality. 30 ng/l each of 3-isobutyl-2-methoxypyrazine, 3-isopropyl-2-methoxypyrazine and 3-secbutyl-2-methoxypyrazine were added to a Riesling and Cabernet Franc wine and monitored with HS-SPME-GC-MS over 18 months to investigate the effects of various closure and packaging options as well as light and storage temperature on MPs. Other impact odorants were monitored using SPE-GC-FID. Changes in MP concentrations during bottle aging varied with closure/packaging option, with the greatest decrease evident in Tetrapak® cartons. We observed similar changes in other impact odorants to previous studies, with synthetic corks displaying an increased capacity for sorption compared to natural corks and screwcaps. MPs did not vary consistently over time with light or temperature conditions. Acetate esters decreased, regardless of light or temperature conditions, while phenethyl acetate and isoamyl acetate decreased at a greater rate in ambient temperature conditions compared with 12°C. Free and bound SO_2 retention was higher in light-excluded conditions and influenced by bottle hue.

Keywords: methoxypyrazines; packaging; wine closures; Tetrapak; ladybug taint; *Harmonia axyridis*; aging; bottle hue; storage temperature; cellaring; wine quality; wine flavour

INTRODUCTION

Odor-active compounds are important determinants of wine flavour and overall quality. MPs are potent odorants found in many wines, with sensory thresholds as low as 320 pg/l (Pickering *et al*. 2007). 3-isopropyl-2-methoxypyrazine (IPMP), 3-secbutyl-2-methoxypyrazine (SBMP) and 3-isobutyl-2-methoxypyrazine (IBMP) are secondary grape metabolites that contribute to the desired varietal character of certain wines (Parr *et al*. 2007) but, at higher levels, are responsible for vegetative and herbaceous aromas considered detrimental to wine quality (Allen *et al*. 1994). Another source of MPs in wine is the inadvertent incorporation of lady beetles (Coleoptera: *Coccinellidae*) with grapes during harvest operations, which can result in a wine defect known as ladybug taint (LBT, Pickering *et al*. 2004) that has been attributed to IPMP derived from *Coccinellidae* haemolymph (Pickering *et al*. 2005). Regardless of the source of MPs, there is much interest in understanding how they change during wine processing and aging.

Wine closure/packaging options and storage conditions offer opportunities to alter a wine's odorant profile. The capacity for closures or packaging to directly remove volatile compounds through sorptive processes is termed flavor scalping (FS), and has been shown to vary with closure type for some wine odorants (e.g. Brajkovich *et al*. 2005). Interestingly, we are unaware of any peer-reviewed literature on Tetrapak® cartons and FS, surprising, given their widespread use in the wine industry. Light exposure after bottling may also affect constituents of alcoholic beverages, particularly 350 nm–500 nm wavelengths (D'Auria *et al*. 2003). Clear, green and amber are common wine bottle hues, and transmit 95%, 50%, and 10%

of 350 nm–550 nm light, respectively (Selli *et al.* 2002). Additionally, temperature may also affect bottle-ageing through mediation of reaction rates, with higher storage temperatures producing "quick-ageing" effects associated with advanced oxidation, and altering volatile composition (Marais & Pool 1980). The objective of this study was to examine the effects of commonly used closure/packaging options, light and storage temperature on MPs and other impact odorants in a white and red wine over 18 months of bottle-aging.

MATERIAL AND METHODS

Bulk, commercial Riesling and Cabernet Franc wine from the 2006 Niagara Peninsula vintage was used. In order to ensure sensorially relevant concentrations sufficient for quantification over this longitudinal study, 30 ng/l of IPMP, SBMP and IBMP was added to the base wines before bottling. Wines were closed under four cork-type closures (natural cork – NatC, agglomerate cork – Agl, extruded synthetic cork – Syn-Ex, moulded synthetic cork – Syn-M), a roll-on-tamper-evident (ROTE) screwcap (Scap), or a Tetrapak® carton (Tpk). 750 ml glass Bordeaux bottles were used for the bottle treatments. Three chambers were prepared for storage of a sub-set of these wines under specific lighting and temperature conditions: Condition 1 (light + ambient temp), for examining the influence of clear, green and amber bottle hues at 22°C, Condition 2 (dark + ambient temp) and Condition 3 (dark + cellar temp (12°C)). Duplicate samples were retrieved from the cellar and storage chambers 3, 6, 12 and 18 months after bottling and analysed. MPs were determined in duplicate using a stable isotope dilution method that uses headspace-solid-phase-microextraction (HS-SPME) coupled to gas chromatography-mass spectrometry (GC-MS) as detailed in Kotseridis *et al.* (2008). Other indicator compounds were chosen for analysis that represent the most important classes of wine volatiles, specifically, phenethyl acetate, isoamyl acetate, ethyl hexanoate, ethyl caprylate (ethyl octanoate), ethyl caprate (ethyl decanoate), phenyl ethanol, and octanoic acid. Indicator volatiles were determined at 3 and 12 months using solid-phase-extraction (modification of Pickering *et al.* 2005) coupled to GC-FID (modification of Ortega *et al.* 2001) and three internal standards (3-ethyl-2-hydroxy-valerate (for esters), 3-octanol (for phenyl ethanol) and heptanoic acid (for octanoic acid)). General wine chemistry parameters were determined at bottling and after 12 months using the methods of Iland *et al.* (2004). Full details of the treatments, the physico-chemical composition of the base wines, and the analytical methods are given in Blake *et al.* (2009a, b).

RESULTS AND DISCUSSION

Closure/packaging study

IPMP concentration varied between closure/packaging options, and tended to be lower after 12 and 18 months in Cabernet Franc for all closures. The greatest sustained decrease from initial concentrations was with Tpk, where values were 23% and 41% lower in Riesling and Cabernet Franc wine, respectively. Closure/packaging types affected SBMP concentration in a similar way in both wines over time. Concentrations of SBMP were only lower than bottling levels in Tpk wines (average decrease of 27%). IPMP and SBMP concentrations increased at some time points for some closures, perhaps suggesting contribution from the closure itself. Concentrations of IBMP in both Cabernet Franc and Riesling responded similarly to closure/packaging types, and overall decreased significantly in all conditions. After 18 months, the greatest decrease was observed in Tpk and Syn-M and the smallest change was in NatC (Riesling) and Scap (Cabernet Franc). For the non-MP impact odorants, and under all conditions, acetate esters decreased the most during aging, ethyl esters and octanoic acid either increased or decreased slightly, and phenyl ethanol remained relatively stable. Tpk wines had the lowest concentration of acetate esters (Riesling wine) and the highest concentration of ethyl esters after 12 months, but showed no clear trend for other volatiles. After 12 months, Agl (Cabernet Franc) and Syn-Ex (Riesling) show the lowest concentrations of ethyl hexanoate, ethyl caprylate (Riesling) isoamyl acetate, phenyl ethanol (Cabernet Franc), and octanoic acid. These results generally agree with the literature, with synthetic corks showing an increased capacity for volatile sorption compared to natural corks and screwcaps, although the Agl closure also displayed potential FS capacity. Tetrapak® cartons appear to have allowed a greater ingress of oxygen into the wines than bottles, as

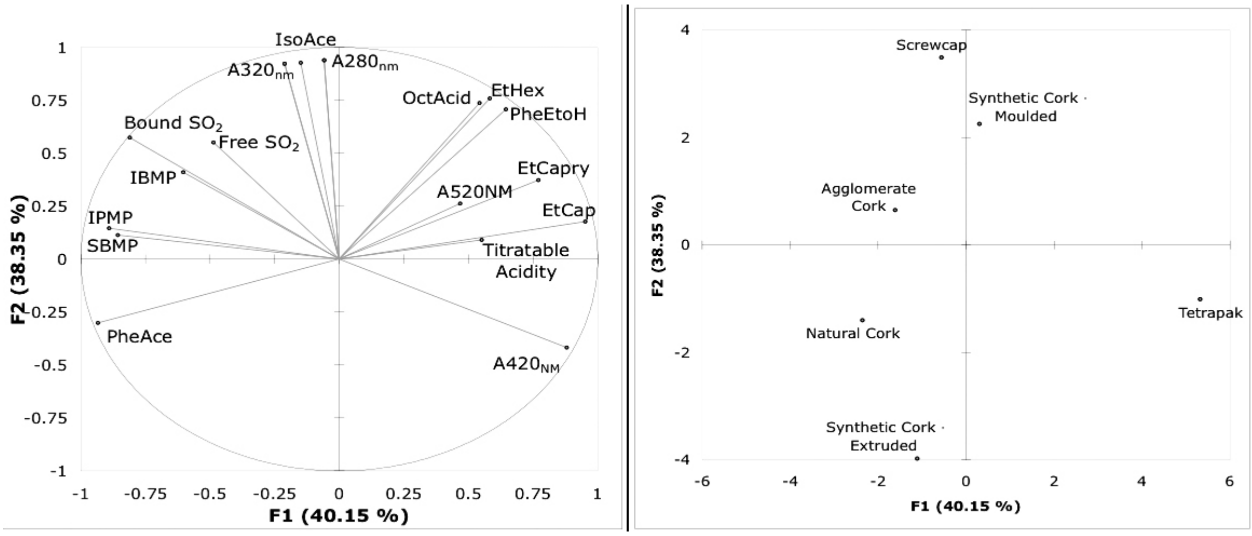

Figure 1. Principal component biplot for Riesling wine after 12 months bottle age (closure trial) (adapted from Blake *et al*. 2009a)

IPMP: 3-isopropyl-2-methoxypyrazine, SBMP: 3-sec-butyl-2-methoxypyrazine, IBMP: 3-isobutyl-2-methoxypyrazine, PheAce: phenethyl acetate, EtCap: ethyl caprate, EtCapry: ethyl caprylate, EtHex: ethyl hexanoate, IsoAce: isoamyl acetate, PheEtoH: phenyl ethanol, OctAcid: octanoic acid

evidenced by spectrophotometric measurements and changes in free and total SO_2 (Figure 1).

Storage condition study

IPMP concentration was relatively stable or showed a small decrease, and was not consistently affected by light or temperature conditions. However, after 12 months IPMP tended to be higher in light-excluded Riesling wines, and, within light treatments, higher in amber bottles compared with other hues for both wines. SBMP concentration was unaffected by light or temperature. IBMP decreased with time in both wine styles regardless of light or temperature condition, which did not

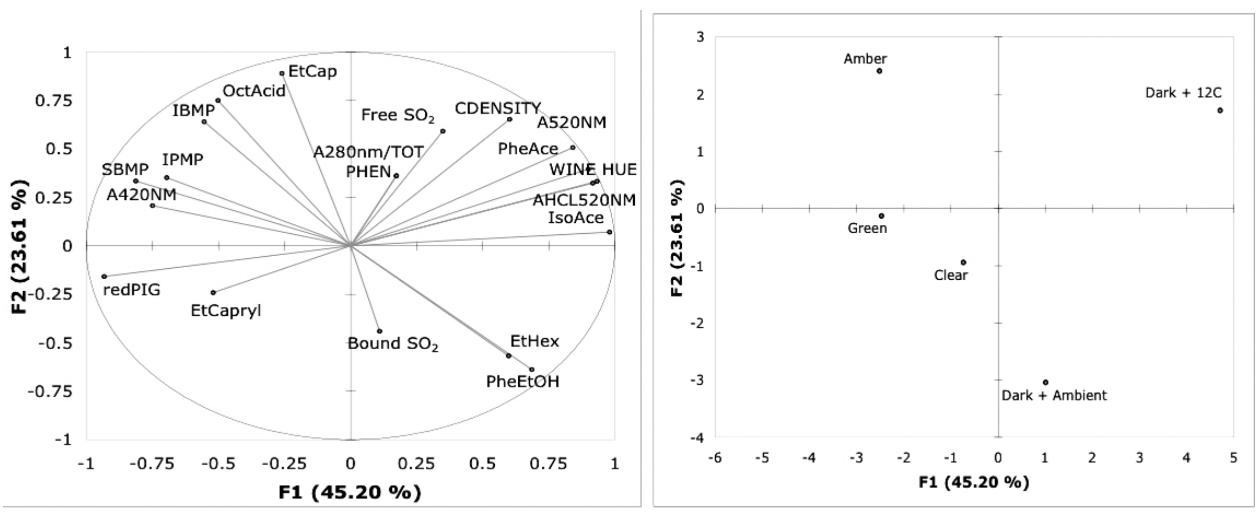

Figure 2. Principal component biplot for Cabernet Franc wine after 12 months (storage trial) (adapted from Blake *et al*. 2009a)

IPMP: 3-isopropyl-2-methoxypyrazine, SBMP: 3-secbutyl-2-methoxypyrazine, IBMP: 3-isobutyl-2-methoxypyrazine, PheAce: phenethyl acetate, EtCap: ethyl caprate, EtCapryl: ethyl caprylate, EtHex: ethyl hexanoate, IsoAce: isoamyl acetate, PheEtOH: phenyl ethanol, OctAcid: octanoic acid, CDensity: wine colour density, RedPig: Degree of Red Pigmentation consistently affect concentrations. Concentrations of acetate esters tended to decrease with storage time, regardless of condition. However, the acetate esters were most affected by storage conditions, with phenethyl acetate and isoamyl acetate decreasing at a greater rate in ambient temperature conditions compared with 12°C. Ethyl esters, phenyl ethanol and octanoic acid tended to be similar for all treatments and remain relatively stable over time independent of storage conditions, although ethyl caprylate in Riesling after 12 months was significantly higher in the dark + ambient temperature condition. Bottle hue did not significantly influence these analytes. Free and bound SO_2 retention was higher in light-excluded conditions, while temperature during storage did not affect SO_2 preservation. Bottle hue also influenced free SO_2 concentration, with retention (averaged across both wine styles) greatest in amber (56%), intermediate with green (43%), and lowest in clear bottles (34%). Our results suggest that differences in gas permeability and contribution from the different closure and packaging types associate with changes in wine composition during ageing, and the combination of light-exclusion and cooler storage conditions tend to associate with increased retention of compounds indicative of higher wine quality.

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