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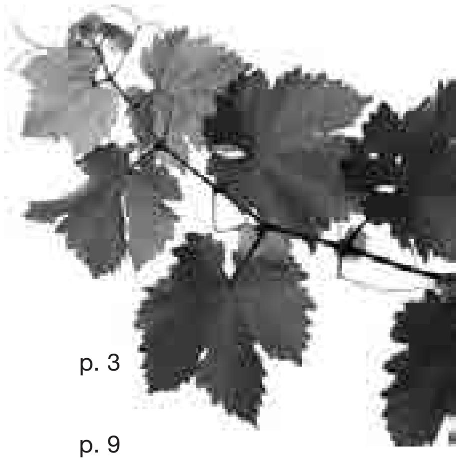

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Review of processing technology to reduce alcohol levels in wines

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Abstract: Lower ethanol content wines are becoming an important style in the range of beverages offered for sale by many wineries as consumers become more attuned to societal attitudes that govern alcohol consumption. The removal of ethanol using various engineering solutions is an important approach for the production of beverages that are more acceptable for certain consumers. Common approaches for ethanol removal include the spinning cone column, reverse osmosis, osmotic distillation, nanofiltration and evaporative perstraction. Each approach has specific advantages and disadvantages and the best approach for ethanol removal from wine is largely determined by the required ethanol reduction, production volumes, capital and operating costs. This review highlights the important features of the commonly used processing techniques for removal of ethanol from wine.

Keywords: alcohol reduction, osmotic distillation, reverse osmosis, spinning cone

1. Introduction

The production of wines with reduced ethanol concentration is an important aspect of wine production that has gained considerable attention over the past 10 years or so. Consumer demand for wines that are perceived as healthier, more favourable excise rates for lower alcohol products and changing attitudes of consumers regarding the social consequences of excessive ethanol consumption are just some of the attitude drivers for lowering ethanol levels in wine. Significant consumer demand is apparent for wines with lower ethanol levels.

Producers may also wish to marginally reduce alcohol concentrations (e.g. 1 or 2% reduction) in wine to correct balance and maintain consistency of style from one vintage to another. Depending upon the required adjustment to ethanol concentration a portion of the base wine may be extensively dealcoholised and used for blending purposes thereby avoiding the requirement to treat the entire wine blend.

A range of methods have been reported in the literature that have application for the removal or moderation of ethanol in wine (Schmidtke *et al.*, 2012). Certain techniques are somewhat theoretical and have limited practical application since the wine matrix is substantially modified, e.g. the use of glucose oxidase to convert fermentable carbohydrate to gluconic acid (Pickering *et al.*, 1999); require considerable energy inputs such as freeze thawing of juice or wine (Vella, 1984); or involve highly specialized plant and equipment with little opportunity for other winery applications such as supercritical liquid extraction (Seidlitz *et al.*, 1991).

Viticultural approaches such as rootstock selection, manipulation of vine leaf to crop ratio (Stoll *et al.*, 2009; Etchebarne *et al.*, 2010), optimizing varietal selection for specific climatic conditions, early grape harvest (Kontoudakis *et al.*, 2011) and altering vineyard management practices which impact and moderate the imbalance between grape sugar accumulation and the development of grape derived flavour precursors are attracting interest and offer solutions that are suitable for moderating ethanol levels to some degree. Moreover, these are very acceptable approaches from a consumer perspective.

After harvest, grape juice can be nanofiltered to concentrate fermentable carbohydrate, with the by-product being a juice with lower potential alcohol that may also been employed for the production of reduced alcoholic wines (García-Martín *et al.*, 2010).

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The selection of novel or modified yeast strains, or manipulating fermentation conditions, so that carbohydrate is biochemically diverted through alternative pathways rather than glycolysis and subsequent ethanol production are also approaches that have potential to moderate the final ethanol concentration in wine (Kutyna *et al.*, 2010). Post fermentation, the most common winery production techniques utilized for ethanol removal rely upon membrane processes and/or thermal distillation. It is feasible to remove ethanol during fermentation using non-membrane techniques without substantial loss of yeast metabolic activity (Wright & Pyle, 1996; Aguera *et al.*, 2010), however the added complexity of operations during the peak labour demands often means that technological approaches to ethanol removal are applied post fermentation.

This review will focus on the commonly used processing technological approaches for removal of fermentable carbohydrate or ethanol from wine, namely membrane based and thermal distillation techniques.

2. Membrane Based Technologies

Membrane based technologies have become a popular tool for wine processing due in part to the flexible configuration of equipment, size, portability and, compared to other technologies, the relatively modest capital requirements for establishing this capacity within wineries. All membrane processes result in the partitioning of the wine into permeate (i.e. passes through the membrane), and retentate (i.e. retained in the feed) streams. Several membrane based techniques have been developed for ethanol removal from beverages. These rely upon different driving forces to transport ethanol across a semi-permeable barrier. The driving forces are: transmembrane pressure (reverse osmosis), partial pressure differential (pervaporation) or a vapour pressure gradient (osmotic distillation). Each technique relies upon the molecular permeation of ethanol from the feed stock with high concentration to a stripping phase with low concentration.

A significant advantage of membrane based technologies is the relatively low cost of operations, and with most operations taking place at low to moderate temperatures, thermal damage to wine aroma by chemical reactions or aroma degradation is limited. Besides this, membranes have been developed that are reasonably selective for ethanol, limiting the loss of volatile aroma compounds from the beverage (Catarino *et al.*, 2007). Disadvantages of membrane technologies are the capital investment required (e.g. membranes, housings etc) and aroma losses may still occur during the membrane processes that affect wine sensory properties. In the case of reverse osmosis there is the added disadvantage of needing to incorporate a second process to separate aroma compounds and water from ethanol in the reverse osmosis permeate (Meier, 1992; Pyle, 1994; Massot *et al.*, 2008). The removal of ethanol from wine using membrane process can be a relatively slow process that requires the base wine to 'pass' the membrane numerous times in order to achieve the desired adjustment of ethanol concentration. A comparison of the basic approaches for ethanol removal using reverse osmosis, osmotic distillation and pervaporation is shown in figure 1, and a summary of these methods appears in table 1.

2.1. Reverse Osmosis process

The basic operating principle of reverse osmosis (RO) units is the application of high pressure to the wine so that specific substances will migrate across a semi-permeable membrane from high to low concentration. Dealcoholisation of wine by RO is performed at high pressures to obtain sufficient permeation rates. The application of such high pressures inevitably leads to increases of temperature at the membrane surface. Prevention of excessive temperature exposure of the wine during operations necessitates the use of ancillary heat exchangers. RO is a widely used process for ethanol removal from wine and was the first membrane based technology commercially used for ethanol removal from beverages (Meier, 1992). There are two main modes of RO operations: dead end and cross-flow. In dead end mode, the feed stock flows directly towards the filter, the permeate passes through the RO membrane, and the retentate remains on the feed side of the membrane throughout the dealcoholisation process. In cross-flow mode, the feed material flows tangentially to the membrane surface, and a portion of the feed material selectively passes through the membrane (permeate). The remainder of the feed material remains on the feed side of the membrane (retentate). The retentate is swept away by the inflow of fresh feed material, and is collected continuously at the downstream side of the reverse osmosis unit.

The membranes typically used for RO ethanol removal are asymmetric, flat sheet membranes composed of cellulose acetate or cellulose triacetate.

Fine hollow fibres of aromatic poly amides or cellulose triacetate and thin film composites, where an extremely fine layer of a highly hydrophilic polymer has been placed on a microporous support, usually made from polysulphone, are also utilised (Noble & Stern, 1995).

Table 1. Membrane separation processes of relevance for moderating ethanol concentration

Separation Process	Approximate size range	Separation mechanism	Driving force	Application	Selected References
Nanofiltration	0.5-5 nm	Sieving and charge effects	Pressure	Controlling juice sugar concentration	[A, B]
Reverse osmosis	0.1-1 nm	Transfer through a semi-permeable membrane due to pressure	Trans Membrane Pressure	Ethanol removal	[A, C]
Osmotic distillation (evaporative perstraction/membrane contator)	0.03-0.5 µm	Transport of volatile component	Vapour pressure gradient	Controlling juice sugar concentrations Ethanol removal	[F, G]
Pervaporation	Nonporous permselective membrane	Partial vaporization	Difference in partial pressure	Dealcoholization of wine Aroma recovery	[D, E]

A: (Cuperus & Nijhuis, 1993); B: (Echavarría *et al.*, 2011); C: (Catarino *et al.*, 2007); D: (Diban *et al.*, 2008) E: (Hogan *et al.*, 1998); F: (Karlsson & Trägårdh, 1996); G: (Catarino & Mendes, 2011)

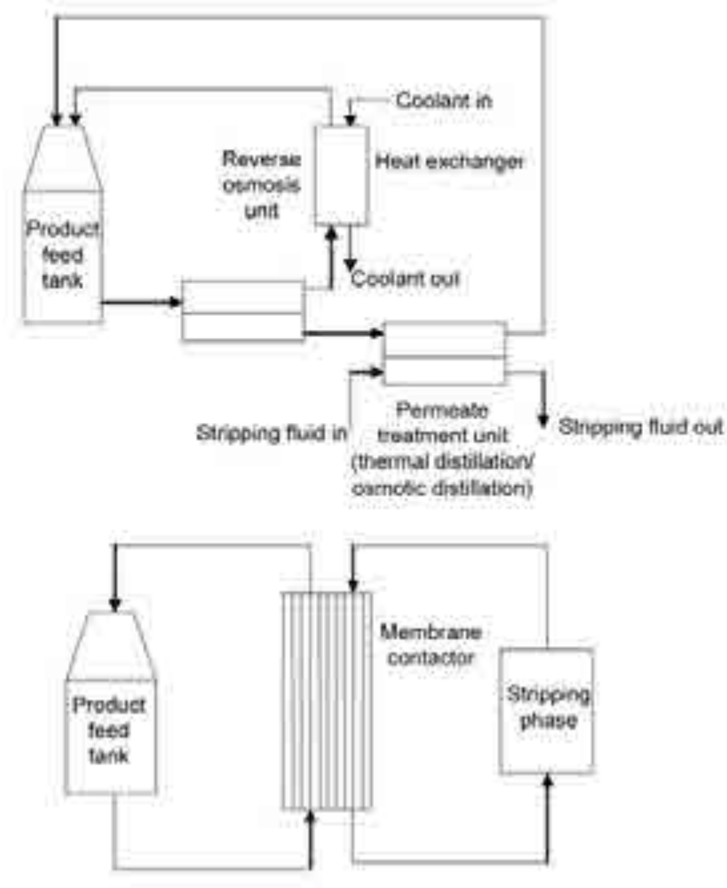

Figure 1. Schematic figure comparing the basic approaches of reverse osmosis (top) and osmotic distillation (bottom) separation approaches for removal of ethanol in wine.

Composite membranes with a high strength polymer supporting layer are most commonly used. These arrangements provide the necessary permeation rates, selectivity and have the capacity to be cleaned and back-flushed to remove any contaminating material that has collected upon the membrane surface. Cellulose acetate or cellulose triacetate thin film membranes are commonly used for ethanol removal. These membranes have a high water and ethanol permeability and good rejection of compounds with high molecular weight such as proteins, polyphenols and carbohydrates. For the ethanol reduction, the best membrane should have the highest permeate flux and lowest ethanol rejection.

Operating parameters such as feed pressure, temperature and flow rates, influence the efficacy of ethanol removal and it is therefore necessary to determine the optimum condition for ethanol permeation and retention of other wine components (Catarino *et al.*, 2007). The effects of these process parameters are summarized as:

- Increasing feed pressure results in higher solvent permeation and thus higher ethanol flux, but also increases the permeation of aroma compounds.
- Ethanol and aroma compound permeation also increases with temperature.

Several membrane configurations have been developed including flat sheet (plate and frame), tubular, hollow fibre and spiral wound. The spiral wound configuration is amongst the most efficient in terms of space. It is essentially a large flat membrane that has been rolled around a hollow retentate collection tube with separate alternating membrane layers. These are separated by a feed spacer, allowing the feed to enter the membrane housing with the flow directed longitudinally. The space between the membranes is directed to the retentate collection tube.

Concentration polarization is an inevitable consequence of membrane separation processes in which a reversible and direct decline of permeate flux across the membrane occurs. This phenomena arises as a build up in the concentration of retained molecules on the pressure side of membrane creating additional resistance to solvent permeation (Cuperus & Nijhuis, 1993). The detrimental effects of concentration polarization and fouling is greater in dead end filtration in comparison to cross flow devices in which accumulated retentate is removed from the membrane surface with the flow of feed stock. Regular back flushing during operations can alleviate to some degree the effects of membrane fouling and restore membrane performance.

The RO permeate stream is usually around 0.7-1.5 % v/v ethanol and therefore contains substantial wine derived water. The consequence of water removal is significant as concentration of wine components arises during processing. Wine must be restored to the original water concentration and this is usually accomplished by the continuous treatment of the permeate to separate the water and ethanol components, with the water redirected back to the treated wine during operations. Permeate treatment using downstream processes of thermal distillation, membrane contactor or pervaporative extraction are required to obtain a high ethanol by-product and water fractions (Massot *et al.*, 2008). The relative low ethanol concentration in the permeate therefore dictates that all of the wine must be ‘passed’ across the membrane several times to achieve significant alcohol reduction which increases the overall processing time and exposure of the wine to the process.

Reverse osmosis does has advantages over the other dealcoholization process. It has low energy consumption, minimizes thermal degradation of aroma compounds and so preserves the sensory characteristics of wine along with ethanol removal (Catarino *et al.*, 2007; Labanda *et al.*, 2009). On the other hand, some researchers argue that the production of low ethanol beverages by reverse osmosis units is not commercially feasible because the production cost and energy consumption increases with incremental increases in osmotic pressure (Pilipovik & Riverol, 2005).

2.2. Osmotic distillation

Osmotic distillation (OD), also referred to as isothermal membrane distillation or evaporative perstraction, is a promising membrane based technique for low to moderate rates of ethanol removal from beverages. It can be used to reduce the ethanol content in alcoholic beverages with minimal effect on the organoleptic properties of the product. The driving force of the process is the partial or vapour pressure differences between volatile solute feed and stripping solutions (Varavuth *et al.*, 2009). In OD the feed stock is circulated through a hydrophobic hollow fibre membrane contactor with the stripping liquid flowing on the opposing side of the membrane. In the removal of ethanol from wine, the stripping fluid is degassed water with hydrophobic membranes (polypropylene or polyvinylidenefluoride) employed to create the pressure differential (Varavuth *et al.*, 2009; Diban *et al.*, 2013). A significant advantage of OD is that most processing can be conducted at ambient temperatures, without the requirement for high pressure. This helps allow the process to be relatively cost effective.

Two streams flow through a hydrophobic hollow fibre membrane contactor, with volatile compounds moving from the liquid with high vapour pressure into that of lower vapour pressure. Microporous hydrophobic membranes create a vapour gap between the two liquid phases. Volatile compounds from the feed (high concentration) solution are free to migrate, by convection or diffusion, to the stripping solution. This configuration can avoid the aqueous solution penetrating into the pores (Diban *et al.*, 2008). Therefore the application of a closed loop OD without downstream processing of the permeate can clearly result in the removal of ethanol from wine until an equilibrium is achieved and the desired ethanol reduction is achieved (Hogan *et al.*, 1998). Replacement of the stripping water and batching the OD process can result in the complete removal of ethanol from wine although significant aroma losses also occur when wine is processed in this manner (Liguori *et al.*, 2013).

Common applications of osmotic distillation are the concentration of fruit juices (Varavuth *et al.*, 2009), and ethanol removal from wine or beer (Hogan *et al.*, 1998; Liguori *et al.*, 2013). OD is suitable for ethanol removal from wine since:

- ethanol is the most concentrated volatile component and the most rapidly diffusing species across the membrane

- the vapour pressure of the aroma compounds is low and so the osmotic distillation flux is also low

The rate of ethanol removal from the feed stream can be manipulated by varying processing conditions (e.g. feed velocity, stripping solution velocity and temperature), however water flux from the strip side of the membrane to that of the feed may also arise (Varavuth *et al.*, 2009). However, some aroma losses do occur with increasing operating time and temperature. For low rates of ethanol (<2 % v/v) removal from wine, OD is reported to have a relatively modest impact upon the concentration of aliphatic acids, monoterpenes and some alcohols. However ethyl esters were substantially reduced (Fedrizzi *et al.*, 2013) and these observations are generally in agreement with previous results (Diban *et al.*, 2008; Varavuth *et al.*, 2009; Diban *et al.*, 2013).

2.3. Pervaporation

Pervaporation is a separation technique in which a liquid feed is separated by partial vaporization through a nonporous selectively permeable membrane. The feed mixture flows on one side of membrane and part of the feed vaporizes passing through the membrane to the opposite side where a carrier gas, or vacuum, removes the permeate whereupon it is condensed to liquid. An important aspect of pervaporation is a phase transfer of the diffusing compounds from the feed into the permeate. Mass transfer in pervaporation occurs in three consecutive steps (Karlsson & Trägårdh, 1996):

- Selective absorption into the membrane at the feed side;

- Selective diffusion through the membrane;

- Desorption into the carrier gas on the permeate side.

The membrane composition used for pervaporation will determine the overall selectivity of mass transfer and hence the application of the process. Typically membranes are dense non-porous and may be hydrophilic or hydrophobic (organophilic). Cellulose acetate or polyvinyl alcohol materials have hydrophilic properties and thus water permeation is favoured when these membrane are used. Conversely polydimethylsioxane, polyoctylmethylsiloxe or polytrimethylsilypropane membranes favour the permeation of organic substances (Catarino *et al.*, 2009). Hydrophobic pervaporation membranes can be used for dealcohlisation as ethanol permeates these membranes more readily than water (Brüschke, 1990). Unfortunately, aroma compounds are also organic and significant aroma losses may arise when pervaporation is applied to wine. The permeability of hydrophilic membranes is highest for water, intermediate for ethanol and low for aroma compounds. However, if the sweeping gas contains water vapour, the flux of water through the membrane is reduced, but ethanol is still passed across the membrane. Consequently a high proportion of ethanol can be separated from a water-ethanol mixture using a hydrophilic membrane. Pervaporation performance is affected by several factors including permeate pressure, feed concentrations, interactions between components in the feed, mass transfer resistance and most importantly temperature (Karlsson *et al.*, 1995). Pervaporation can be operated at low or ambient temperatures although most alcohol removal procedures have been conducted at temperatures at or exceeding 30°C (Tan *et al.*, 2005; Takács *et al.*, 2007).

The most obvious use for pervaporation is the recovery of aroma compounds from beverages and the techniques has been applied to both wine and beer before alcohol reduction using another process (Catarino & Mendes, 2011). The rate of pervaporation of the major classes of aroma compounds varies depending on operating conditions and so optimization of these parameters is necessary to achieve reduced ethanol wine with desirable aroma.

3. Spinning cone

The spinning cone column (SCC) has wide applications in the food and beverage industries for flavour recovery, to preserve the freshness or taste of processed food or drinks, and preparation of concentrates. The SCC can be operated with either liquid or slurry mixes and can therefore be readily used for ethanol removal during fermentation without a requirement for clarification or stabilization of the wine (Wright & Pyle, 1996). The column contains a rotating vertical central shaft fitted with upward facing cones alternating with sets of fixed downward conical baffles attached to the casing of the column (Pyle, 1994). The liquid feed enters at the top of the column and flows down, while the stripping gas (steam or inert gas) is fed into the base of the column and flows up (Pyle, 1994). To avoid regulatory issues in wine production the stripping vapour can be generated by redirecting a portion of the product discharge through a heater prior to reinjection into the base of the column. As the liquid flows downwards a thin film is created by the centrifugal force of the rotating cone and the liquid migrates to the top of the rotating vane whereupon it drops onto the underneath fixed cone and migrates back towards the centre of the column. This liquid flow path is repeated for the number of rotating and fixed cones thereby creating a large surface area to facilitate mass transfer of volatile components from liquid to the gaseous phase. The SCC operates under vacuum so volatile aroma components are transferred to the gas phase at relatively high vacuum and low temperatures (Harders & Sykes, 1999). Vapour phase turbulence is promoted by fins on the undersurface of the rotating cones and this enhances mass transfer whilst preventing pressure gradient formation within the column. Coupled with reasonable clearances between alternating cones, the fins also ensure that a constant pressure and therefore constant temperature is maintained through the entire length of the column. The cone arrangements and a schematic diagram illustrating the SCC setup for ethanol removal from wine is shown in figure 2.

Ethanol removal from wine using the SCC is typically a two-stage process. In the first stage, the more delicate aroma components are removed at moderate to high vacuum (~0.04 atmosphere) and low temperature (~26-28 °C) (Belisario-Sánchez *et al.*, 2009) with these components typically collected in a high strength ethanol stream that represents approximately one percent of the original wine volume. The second stage in which ethanol is removed from the base wine is conducted at a higher temperature, usually around 38 °C and results in an alcohol concentrate that is typically above 50 % v/v along with the dealcohlised base wine. The concentrated alcohol stream can therefore be used for alternative products without significant additional downstream processing and water is not removed from the original base wine.

It is possible to reduce the ethanol concentration from 15 % v/v to less than 1 % v/v using the SCC. Adjustment of the ethanol concentration in a blend to achieve sensory balance is usually conducted by extensively treating a portion of the final volume. The final dealcoholised wine is produced by blending the recovered aroma with the wine produced by the second stage (Pyle, 1994). Total residency time of the wine is between 10-20 seconds, for each pass of the feed stock, depending upon the size of the SCC and operating conditions with flavour and ethanol removal occurring in a single pass for each fraction.

A number of ancillary devices are required for the SCC; namely heat exchangers to warm the product feed to operating temperatures, pumps and condensers to collect the gaseous vapour and collect the removed fraction. In terms of cost the SCC has a relatively high requirement for capital outlay and operating expense, however the advantage of high throughput, flexible operating conditions, hygiene and application to the production of juice concentrate make the SCC a suitable technology for large wineries.

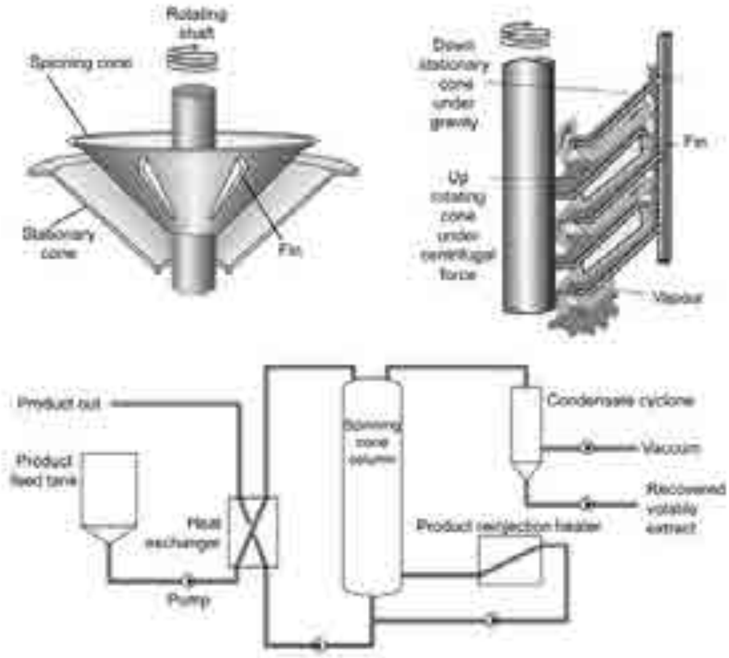

Figure 2. Rotating and fixed stationary cones produce a large surface area for liquid to vapour phase transfer. Product reinjection to create the stripping vapour is used for wine processing. Courtesy Flavourtech, Lenehan Rd, Griffith, NSW.

4. Conclusion

The method chosen by wine producers to moderate ethanol levels are often determined by consideration of the important factors of wine style, production volume, the level of ethanol to be removed from the product, a desire to retain natural or organic association of the product, capital outlay, operating expenses, flexibility for use of equipment and staff training requirements. For many wine producers it will be important to implement strategies that target the entire wine production process, commencing with vineyard site and varietal selection, management practice, careful control of fermentation parameters along with judicial use of appropriate processing technologies for producing wines with lower ethanol concentrations. Several processing technologies are available for ethanol removal from wine; however, no single approach is likely to produce a significant alcohol reduction without substantial alteration to the sensory properties of the product. Such changes to the sensory properties of the product arise from aroma loss and alteration to mouth feel properties such as body, heat, sweetness and perception of bitterness and acidity. Importantly, the perceived change of aroma may not always be associated with the actual removal of aroma active compounds from the wine. The presence of absence of ethanol in wine substantially alters the polarity of the wine base and this impacts the volatility of aroma compounds (Aznar *et al.*, 2004; Villamor & Ross, 2013). The extent of sensory change in reduced alcohol wines is very much a factor of the presence or absence of specific classes of compound responsible for the varietal aroma and the extent of ethanol removal. The behaviour of different aroma compounds and aroma classes with regard to retention or permeation through membranes, modification or degradation with thermal exposure and alterations to volatility remain largely unknown. It is likely that the best processing approach for ethanol removal from specific wines will therefore be determined by the varietal composition and with careful optimization of operating conditions such as temperatures, flow rates, stripping, condensation and aroma recovery rates. Clearly significant research into the behaviour of flavour and sensorially important compounds is required to optimize wine processing technologies for ethanol removal.

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