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ORIGINAL RESEARCH ARTICLE

Quantitative analysis of sugars extracted from French oak (*Quercus petraea*) chips: Effect of toasting temperature

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ABSTRACT

In this study, we conducted extraction experiments using model wine on French oak chips toasted at various temperatures to investigate the extracted sugars. A certain amount of sugars were extracted from the oak chips, and the quantity significantly varied depending on the toasting temperature. Analysis of the composition of the extracted sugars revealed the presence of substantial amounts of xylose and glucose, with the composition showing significant differences based on the toasting temperature. Furthermore, it was elucidated that the extracted sugars included monosaccharides and oligo or polysaccharides. This research represents the first comprehensive analysis correlating the toasting temperature with the extraction of a substantial amount of sugars from toasted chips.

KEYWORDS: Sugar, oak chip, toasting, wine

INTRODUCTION

The use of barrels in winemaking is important because it adds to the complexity of wine flavour and improves wine quality (Garde-Cerdán and Ancín-Azpilicueta, 2006). Wine barrels are generally made of oak and assembled by coopers. One essential process in wine barrel making is the heat treatment called "toasting," in which the inside of the wood is toasted over an open fire (Jackson, 2008). Different chemical changes occur inside the wood depending on the degree of toasting (Watrelot *et al.*, 2018). The degree of toasting is mainly classified into light toasting, medium toasting, and heavy toasting, and has a significant effect on the sensory characteristics of wine as different substances are extracted into wine from the barrels (Chira and Teissedre, 2014).

The compounds extracted into wine from oak barrels can be broadly classified into volatile and non-volatile components. Volatile compounds affect wine aroma and include typical compounds such as oak lactone, eugenol, and vanillin, which are aroma compounds characteristic of oak wood (Bautista-Ortín *et al*., 2008; Pérez-Prieto *et al*., 2002). Non-volatile compounds have also been studied intensively in the past several years, focusing on phenolic compounds that affect wine taste, colour stability, and mouthfeel (García-Estévez *et al*., 2017; Morata, 2019). It has been shown that the amount of ellagitannin, a typical non-volatile component, is reduced by toasting (Cadahía *et al*., 2001). Therefore, because of the significant impact of barrel toasting on wine quality, research on these barrel-derived compounds has been vigorously conducted (Carpena *et al*., 2020; Singleton, 1995).

Several studies have focused on the extraction of sugars from barrel wood. Wood is composed of natural polymers such as cellulose and hemicellulose, which have a sugar-linked structure (Fengel and Wegener, 1983). It is, therefore, not surprising that these natural polymers are thermally decomposed by toasting and extracted as sugars. In fact, the extraction of sugars from barrels has been reported in several studies of spirits. Experiments on brandy aged in oak barrels for 30 years showed that up to 500 mg/L monosaccharides could be extracted (Viriot *et al*., 1993). Another experiment showed that the sugar content in brandy aged in oak barrels for 40 years increased to 2000 mg/L (Lafon, 1971). Belchior performed paper chromatography on brandy aged in Limousin oak barrels for 6 months and found that arabinose, glucose, xylose, rhamnose, and fucose were extracted (Belchior, 1972). Unfortunately, limited scholarly attention has been given to the extraction of sugars from wine barrels. Alañón *et al*. (2010) quantified monosaccharide anhydrate extracted from oak wood under strong toasting conditions using model wine (Alañón *et al*., 2010). Del Álamo *et al*. (2000) showed that storage in barrels increases sugar extraction, but it is not clear whether this sugar is derived from the barrels (Del Alamo *et al*., 2000). Le Floch *et al*. suggested that polysaccharides may be extracted from wood barrels into wine (Le Floch *et al*., 2015). However, the types and amounts of sugars extracted from the barrels under wine barrel ageing conditions remain unclear.

In this study, experiments were carried out with model wines to analyse the sugars extracted from French oak chips and the effect of toasting temperature on sugar content. To our knowledge, this study is the first to evaluate the extraction of sugars from oak chips quantitatively.

MATERIALS AND METHODS

1. Reagents and chemicals

D-(+)-Galacturonic acid (GalA) monohydrate and D-glucuronic acid (GlcA) were purchased from Sigma-Aldrich (St. Louis, USA). Tetrahydrofuran (THF, GPC grade), acetonitrile (HPLC grade), trifluoroacetic acid, D-(+)-galactose (Gal), D-(+)-mannose (Man), L-(+)-arabinose (Ara) and D-glucose (Glc) were from Fujifilm Wako Pure Chemical Industries (Osaka, Japan). D-(+)-xylose (Xyl) was from Tokyo Chemical Industry Co., Ltd. (Tokyo, Japan). L-(−)-fucose (Fuc) and L-(+)-rhamnose (Rha) were from Nacalai Tesque Inc. (Kyoto, Japan). An ABEE labelling kit was purchased from MGC Woodchem Corp. (Tokyo, Japan). Polystyrenes of different molecular masses [(Mp) 162, 370, 580, 1180, 4870, 9310, 28,440, and 75,050] were from Agilent (Santa Clara, CA, USA). TLC silica gel 60 F254 (20 cm × 20 cm) was purchased from Merck. (Darmstadt, Germany). Other chemicals were of analytical grade.

2. Preparation of oak chip samples

Heartwood staves of French oak (*Quercus petraea*) grown in Allier, France, were used. The staves (approximately 30 kg) were chipped using a chipper shredder (KCM181D, Kioritz Corp., Tokyo, Japan). The dimensions of oak chips selected for this work were approximately 1.5 cm × 1.0 cm × 0.1 cm (width × length × thickness). Oak chips were toasted at 180 °C, 200 °C, 220 °C, 240 °C, 260 °C, and 280 °C for 10 minutes in a muffle furnace (del Alamo Sanza *et al*., 2004; Jackson, 2008) (Yamato FP313, Tokyo, Japan), and cooled at room temperature. Oak chips without toasting treatment (Untoasted, UT) were also used as control samples.

3. Measurement of weight loss during toasting

The oak chips (approximately 6 g) were weighed before toasting (initial weight). After the toasting treatment, the chips were allowed to stand in a room and weighed several times until their weight became constant. The percentage of weight loss from the initial weight was calculated. The experiment was conducted in five replications.

4. Extraction and sampling preparation of oak chips with model wine

All model wines used in this study contained 5 g/L potassium tartrate and 12 % (v/v) ethanol and were adjusted to pH 3.6 using HCl. Six grams of oak chips were added to 1 L of a model wine in a glass bottle, and the headspace was replaced with N_2 gas. Subsequently, extraction experiments were conducted under dark conditions at 25 °C with shaking

at 80 rpm using a shaker (Yamato Scientific Co., Ltd., BW400, Tokyo, Japan). On days 0, 1, 3, 5, 7, 10, 13, 16, and 19 of the extraction experiments, 3 mL samples were collected and subjected to neutral sugars analysis. After sampling, the headspace was replaced with N_2 gas. After 21 days of extraction, the remaining liquid was placed in a rotary evaporator to remove ethanol and then freeze-dried to obtain powder, and the powder was used as the freeze-dried extract for gel permeation chromatography (GPC) analysis. All extraction experiments were performed in triplicate.

5. Determination of neutral sugars

Neutral sugars were determined by the phenol-sulfuric acid method, as described by Dubois *et al*. (1956). The amounts of neutral sugars were calculated as xylose equivalent. All measurements were performed in triplicate.

6. HPLC analysis of sugars

The sugars extracted from French oak chips were determined separately for monosaccharide and total sugar composition. Total sugar composition was analysed by hydrolysing samples with 8M TFA followed by ABEE derivatisation according to the method of Gomis *et al*. (Gomis *et al.*, 2003) using an ABEE labelling kit (MGG Woodchem Corporation, Tokyo, Japan). Monosaccharides were analysed using the same method, omitting the acid hydrolysis process. The amounts of oligo- or polysaccharides were determined by calculating the difference between the amounts of monosaccharides and the total sugar content. The HPLC system was equipped with a pump (PU-2089 Plus), a column oven (CO-2065 Plus), an autosampler (AS-2055 Plus), and a UV detector (UV-2075 Plus), which were all from JASCO Corp. (Tokyo, Japan). Samples were passed through a 0.45 µm PTFE syringe filter (Lab Lab Company Co., Ltd., Tokyo, Japan). Aliquots of 10 µL were injected into the column (Honenpak C18, 75 mm × 4.6 mm i.d., MGC Woodchem Corp., Tokyo, Japan). ABEE-derivatised sugars were detected by measuring the absorbance at 305 nm. The temperature for the chromatographic separation was set at 30 °C. Sugars in the samples were quantified using calibration curves prepared using standard solutions of two acidic sugars (GlcA, GalA) and seven neutral sugars (Gal, Man, Glc, Ara, Xyl, Fuc, Rha). All measurements were performed in triplicate.

7. Gel permeation chromatography (GPC)

GPC was measured using the method described by Vera-Loor *et al.* (2023). The HPLC system was equipped with two pumps (PU-2080 Plus), a column oven (CO-2065 Plus), an autosampler (AS-2057 Plus), and an RI detector (RI-4030), which are all from JASCO Corp. (Tokyo, Japan). The freeze-dried extract was dissolved in THF (200 mg/mL), and an aliquot of the dissolved extract was passed through a 0.45 µm Nylon syringe filter (Lab Lab Company, Tokyo, Japan). Then, 100 µL of the filtrate was injected into the column (Agilent PLgel MIXED-D, 30 cm × 7.5 mm i.d., Santa Clara, CA, USA). The mobile phase was THF, the flow rate was 1.0 mL/min, and the column temperature was set at 35 °C. The standard used in the measurements for column calibration was polystyrene. Molecular weight was estimated with this calibration curve ($r^2 = 0.998$). The molecular weight for each peak was estimated using polystyrene as the standard. Each measurement was taken only once.

8. Statistical analysis

Statistical analysis was performed by Tukey's honestly significant difference test with JMP (SAS Institute Inc., Cary, NC, USA)

RESULTS AND DISCUSSION

1. Change in chip weight after toasting

The percentages of weight loss were 4.7 %, 5.2 %, 6.2 %, and 9.8 % after toasting at 180 °C, 200 °C, 220 °C, and 240 °C, respectively, showing that the weight loss increased with increasing toasting temperature (Figure 1).

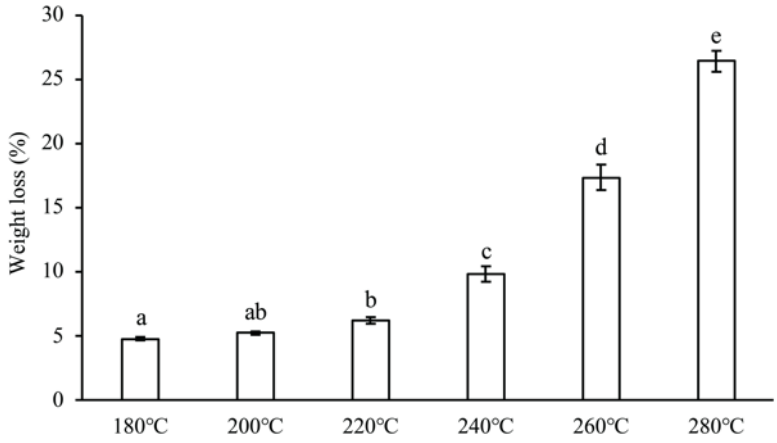


FIGURE 1. Weight change of oak chips after toasting. Bars indicate the standard deviation. Different letters indicate significant differences between samples ($p < 0.05$).

On the other hand, severe weight losses of 17.3 % and 26.4 % were observed at 260 °C and 280 °C, respectively. Previous papers have shown that wood components undergo several stages of chemical changes depending on the heat treatment temperature (Esteves and Pereira, 2008). At low temperatures ranging from room temperature to 150 °C, drying occurs primarily, initially with the loss of free water in the wood and finally with the removal of bound water. At heat treatment temperatures of 180 °C to 250 °C, natural polymers in wood undergo gradual and irreversible chemical changes, and at temperatures above 250 °C, carbonisation of wood occurs, forming carbon dioxide and pyrolysis products. Therefore, we speculated that the significant weight loss at 260 °C and 280 °C, as shown in Figure 1, was due to the carbonisation of the oak chips caused by toasting at high temperatures, which gasified the wood components and made the chips lighter.

2. Neutral sugar content in the extract

Neutral sugars extracted from the oak chips into the model wines were quantified by the phenol-sulfuric acid method and progress curves were generated (Figure 2).

For all toasting conditions, rapid sugar extraction was observed within a week (Figure 2). Thereafter, neutral sugar extraction was slow until day 21. Figure 1 shows that the percentage of weight loss increases as the toasting temperature increases. Thus, more oak chips are added at higher toasting temperatures than at lower toasting temperatures. The amounts of neutral sugars extracted per gram of oak original untoasted chips on Day 21 were calculated (Figure 3), considering the weight loss of chips as the raw material due to heat treatment as described in 3.1.

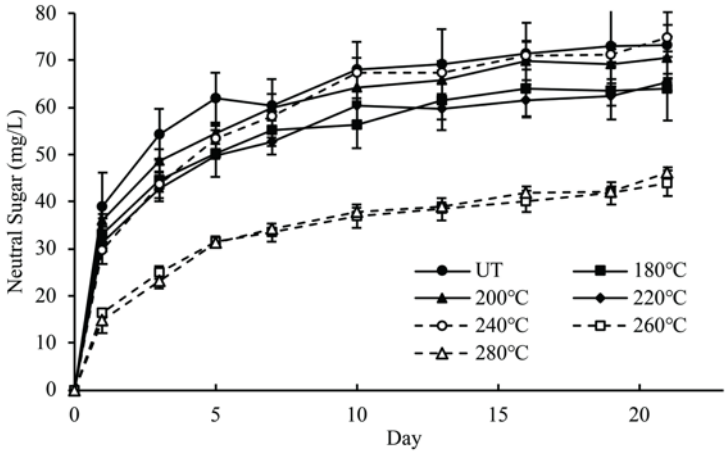


FIGURE 2. Amounts of neutral sugars extracted from oak chips untoasted or toasted at different temperatures over time. Bars indicate the standard deviation.

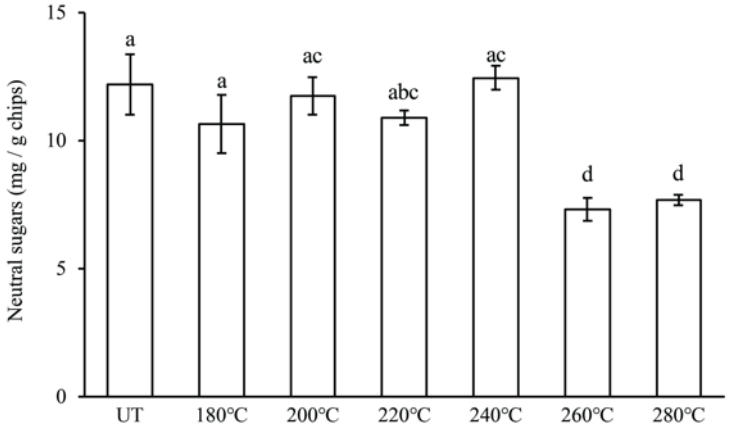


FIGURE 3. Amounts of neutral sugars extracted from oak chips untoasted or toasted at different temperatures on Day 21. Values were corrected by the weight loss rate, which is the amount extracted relative to the amount in oak chips before toasting.

Bars indicate the standard deviation. Different letters indicate significant differences between samples ($p < 0.05$).

Sugars extracted from chips were about 12 mg/g of original chips and were constant from UT to 240 °C toasting conditions. However, a significant reduction was shown for the chips treated more than 260 °C. This suggests that, through toasting at temperatures exceeding 260 °C, alterations in the structure of polysaccharides occurred in chips that led to a decrease in the extracted sugar content in addition to the changes in bound water content in the chips. Moreover, in our preliminary extraction experiment using commercial oak chips, the amount of sugars extracted was more than two-fold that of those in this study (data not shown). Therefore, the amount of sugar extracted may vary between chip makers.

The wood used for barrels usually undergoes a seasoning process, during which the wood is exposed to rain and wind for two to three years while slowly drying naturally. Therefore, water-soluble components such as sugars are expected to be washed away during this seasoning process. However, we found that 12.2 mg/g-chips of sugar were extracted from UT chips. It is speculated that the sugars present inside the staves before chipping were hardly removed during the seasoning process and were exposed after chipping and dissolved out. Further research is required to confirm this speculation.

3. Analysis of sugar content and composition

The contents and compositions of monosaccharides and oligo or polysaccharides extracted into model wines were measured on day 21, and the results are shown in Figures 4 and 5, respectively.

Figure 4 shows that monosaccharides were extracted into the model wines from untoasted (UT) oak chips and oak chips toasted at different temperatures.

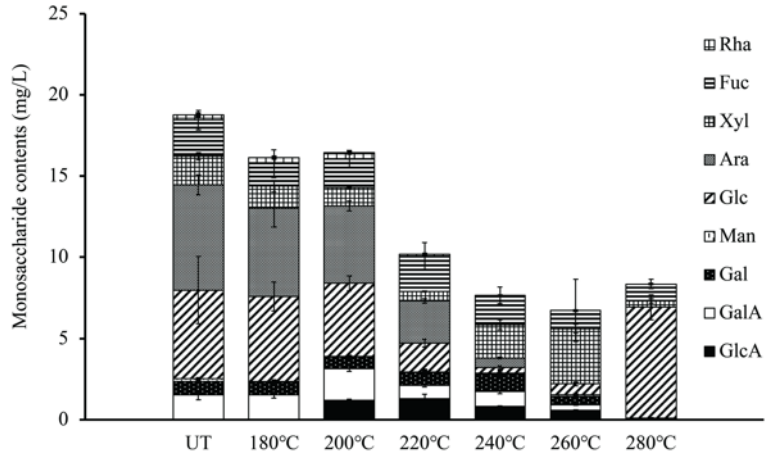


FIGURE 4. Contents and compositions of monosaccharides extracted from oak chips. Bars indicate the standard deviation.

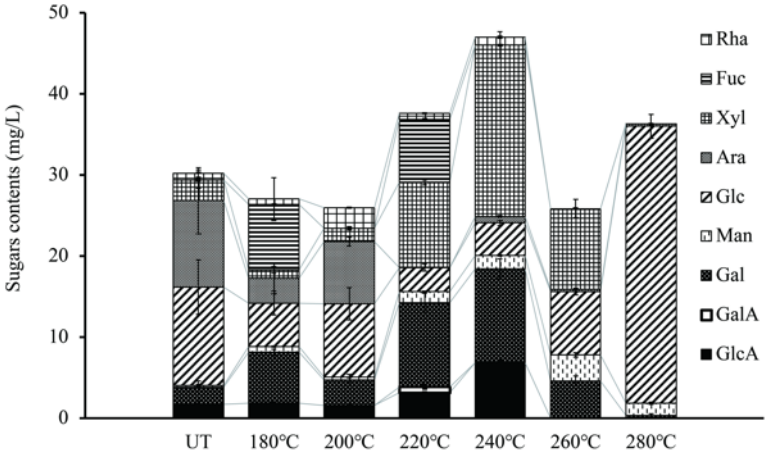


FIGURE 5. Contents and compositions of oligo or polysaccharides extracted from oak chips. Bars indicate the standard deviation.

Consistent with the results of this experiment, it has been reported that monosaccharides such as Ara, Xyl, Gal, and Glc are extracted from barrel wood (Alañón *et al.*, 2010). The amounts of monosaccharides extracted into the model wines from UT oak chips and oak chips toasted at 180 °C and 200 °C showed slight differences, namely, they were 18.77 mg/L, 16.12 mg/L, and 16.45 mg/L, respectively. In addition, no significant differences in the composition of sugars extracted into the wines were observed for the three oak chip samples. On the other hand, the amounts of monosaccharides extracted into the model wines decreased steeply for oak chips toasted at 220 °C or higher temperatures; for oak chips toasted at 260 °C, it was the lowest at 6.73 mg/L. In the UT sample and the oak chip samples toasted at 180 °C and 200 °C, Glc and Ara were detected in high amounts in the model wines. On the other hand, the amounts of Glc and Ara gradually decreased, whereas the amount of Xyl increased in the model wines as the oak chip toasting temperature increased from 220 °C to 260 °C. The amount of Xyl rapidly decreased, and that of Glc significantly increased in the model wine for the oak chips toasted at 280 °C.

Figure 5 shows that the amounts of oligo or polysaccharides extracted into the model wines from the UT sample and the oak chip samples toasted at 180 °C and 200 °C did not change much, being 30.17 mg/L, 27.05 mg/L, and 25.22 mg/L, respectively.

However, the composition of sugars extracted into the model wines from these oak chip samples differed significantly at different toasting temperatures. Glc and Ara were extracted in high amounts from the UT sample but were extracted in low amounts from oak chips toasted at 180 °C. On the other hand, the amounts of Gal and Fuc extracted from oak chips toasted at 180 °C increased. The amount of Xyl extracted increased as the oak chip toasting temperature increased from 180 °C to 240 °C, and the highest amount was extracted from oak chips toasted at 240 °C (18.99 mg/L). The amounts of sugars extracted increased for oak chips toasted at 200 °C to 240 °C, and the highest amount was extracted from oak chips toasted at 240 °C (44.80 mg/L). On the other hand, the amounts of oligo or polysaccharides extracted from oak chips toasted at 260 °C and 280 °C decreased to 23.39 mg/L and 20.80 mg/L, respectively. Furthermore, the amounts of Gal and Xyl extracted decreased rapidly from oak chips toasted at 260 °C, whereas the amount of Glc increased. Therefore, it is clear that a significant amount of oligo/polysaccharide is extracted from the oak chips. The oligosaccharides present in wine have been studied in recent years, and their abundance and roles are becoming better understood (Apolinar-Valiente *et al*., 2021). However, the effects of barrel-derived oligosaccharides on wine are not clear, and further research is needed. It is not known if polysaccharides can affect flavour, but they may have some effect, such as an effect on tartaric acid precipitation suppression. If these sugars are polysaccharides, the amount extracted is 1/3 to 1/10 of that from grape skins or yeast (Gawel *et al*., 2018). However, a preliminary extraction experiment with commercial oak chips confirmed that the amount of polysaccharides extracted was about twice that of the sugars obtained here (data not shown).

Even though the amounts of neutral sugars extracted from the UT sample and the oak chip samples toasted up to 240 °C remained constant, as shown in Figure 3, the compositions of sugars extracted from the oak chip samples toasted at different temperatures were significantly different, as shown in Figure 5. Furthermore, as the oak chip toasting temperature increased, the amount of monosaccharides extracted decreased, whereas the amount of oligo- or polysaccharides extracted increased. The experiments detected two acidic sugars (GlcA and GalA) and seven neutral sugars (Gal, Man, Glc, Ara, Xyl, Fuc, Rha). The major polysaccharides in wood are cellulose and hemicellulose (Fengel and Wegener, 1983). In particular, hemicellulose is mainly composed of pentoses (Xyl and Ara), hexoses (Man, Glc, and Gal), and uronic acids (GlcA and GalA). Based on the types of sugars detected in this study, it is highly likely that sugars derived from wood polysaccharides were extracted. In general, the wood cell wall is composed of microfibrils, which are crystalline structures made up of aggregates of cellulose molecules, and an amorphous matrix composed of hemicellulose and lignin that fills the space between the crystalline structures (Wang *et al.*, 2021). Amorphous hemicellulose is less thermally stable than crystalline cellulose and is, therefore, more susceptible to thermal decomposition by toasting (Mburu *et al*., 2008; Özgenç *et al*., 2017). In this regard, amorphous hemicellulose portions may be preferentially degraded. Sikora *et al*. disclosed that the pyrolysis of hemicellulose is temperature-dependent because the contents of Man, Gal, and Ara, which are sugars constituting hemicellulose, decrease with increasing heat treatment temperature (Sikora *et al*., 2018). From Figure 5, it is inferred that the large difference in sugar composition from 180 °C to 240 °C is due to the different ways pyrolysis of polysaccharides in the wood occurs at each toasting temperature. On the other hand, the amounts of sugars extracted were significantly reduced at the toasting temperatures of 260 °C and 280 °C. These results were consistent with neutral sugars quantified by the phenol-sulfuric acid method. Furthermore, Glc accounted for the majority of the total sugars detected at 260 °C and 280 °C (13.36 mg/L and 15.09 mg/L, respectively), whereas the amount of Xyl, which was abundant at 240 °C, was greatly reduced at these temperatures. Brito *et al*. stated that complete degradation of hemicellulose in wood occurs at a heat treatment temperature of 250 °C or higher (Esteves and Pereira, 2008). From the observed decrease in the amounts of sugars extracted and the sugar composition, it is considered that the sugars derived from hemicellulose are denatured into other components for the oak chips toasted at 260 °C and 280 °C. Furthermore, there are glycosidic precursors in oak chips that are involved in oak-derived aromas (Slaghenaufi *et al*., 2013). The amount of these may vary with toasting conditions, potentially affecting wine quality. In this experiment, it is not known how glycosidic precursors are formed and hydrolysed in oak chips. However, the amount and composition of extracted sugars vary with the toasting conditions; this could indirectly affect the oak-derived volatile components.

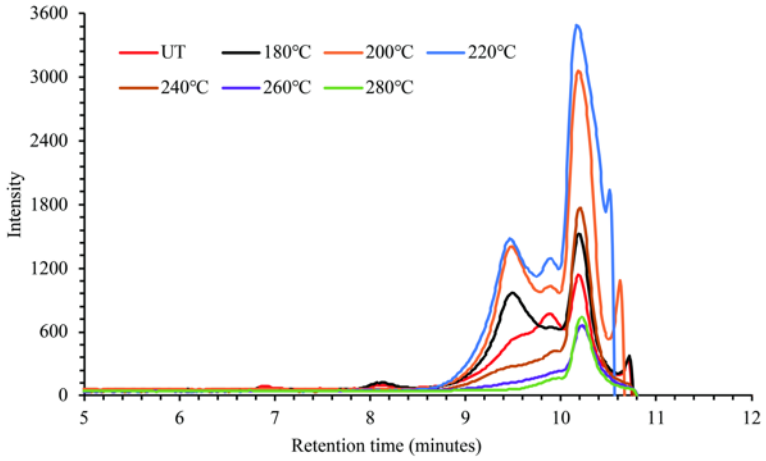


FIGURE 6. GPC chromatograms of extracted components from oak chips untoasted or toasted at different temperatures.

TABLE 1. Estimated molecular weights of the freeze-dried extracts from oak chips untoasted or toasted at different temperatures.

Sample	Peak 1	Peak 2	Peak 3	Peak 4	Peak 5	Peak 6
UT	71,000	10,400	1060	573	356	156
180 °C		9690	1120	551	356	154
200 °C			1100	573	356	154
220 °C			1130	582	365	212
240 °C			1110	544	351	
260 °C				551	342	
280 °C				502	342	

A comparison of the sugar composition extracted from the oak chips toasted at 260 °C and 280 °C revealed that the amount of Glc was significantly higher at 280 °C than at 260 °C (Figures 4 and 5). Cellulose has a crystalline structure in wood and is not easily decomposed by heating (Poletto *et al.*, 2012). Therefore, we considered that amorphous hemicellulose and crystalline cellulose were decomposed simultaneously at 260 °C and 280 °C. Monosaccharides, oligosaccharides, and polysaccharides were extracted from the oak chips toasted at 280 °C (Figures 4 and 5). Yu *et al.* showed that the pyrolysis of cellulose at 100–300 °C resulted in the production of sugars and anhydrous sugar oligomers with varying degrees of polymerisation ranging from 1 to 10 (Yu *et al.*, 2012). The results of this study inferred that these sugars were extracted into model wine from the oak chips.

4. GPC analysis

In addition to the amount and composition of sugars determined in this study, we were interested in the molecular size of these sugars. Therefore, GPC measurements were carried out to estimate the molecular weights of the sugars extracted from the oak chips (Figure 6).

The estimated molecular weights of the GPC peaks are shown in Table 1. Six peaks were identified. Peaks 1 and 2 were identified for the UT sample, and peak 2 was identified for the oak chip sample toasted at 180 °C. Peaks 3 and 4 likely indicate the molecular weights of the hexamer and the trimer sugars, respectively. Peak 3 was identified for the oak chip samples toasted at 180 °C to 240 °C but not for the samples toasted at 260 °C or higher temperatures. Peak 5 indicates the molecular weight of the dimer, and peak 6 is the molecular weight of the monomer. As monosaccharides were identified through sugar analysis using HPLC (Figure 4), and so because the estimated molecular weight closely matched that of monosaccharides (180), peak 6 was considered as the monosaccharides. It is important to highlight that corresponding spots for monosaccharides were also observed in TLC analysis; however, these results were excluded from this study due to the low resolution of the analysis (data not shown). On the other hand, the extracted components from the oak chips include phenolic compounds and polysaccharides as well as their thermal decomposition products, which are likely to be detected as peaks. Therefore, the molecular

weights of the sugars extracted as oligo or polysaccharides could not be estimated.

CONCLUSIONS

This study's extraction experiments using model wines revealed that sugars were extracted as monosaccharides and oligo or polysaccharides from French oak chips. Analysis of sugar composition revealed that Xyl and Glc were extracted in large amounts, suggesting that these sugars were produced by the thermal decomposition of cellulose and hemicellulose in the oak chips. Moreover, it was clarified that the toasting temperature significantly affects the amount, composition, and size of extracted sugars. The extraction of sugars from oak chips is presumably due to the thermal decomposition of the natural polymers in the wood, but further experimentation with barrels is needed as it is quite possible that the same thing could happen in actual oak barrels. The seasoning process may also significantly impact the concentrations of sugars in wines. GPC revealed the extraction of sugars whose molecular weights correspond to the monomers. The sugars extracted from the oak chips are not expected to affect the taste of wine because their amounts are low. Further experiments should be conducted to determine the effect of the sugar extracted from the oak chips on the wine.

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