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Effects of different pectinase maceration pre-treatments on physicochemical properties of raspberry juice and wine

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Abstract: This study presented the effects of different pectinase maceration pre-treatments on the basic chemical properties, the content of 13 phenolic compounds, and their sensory evaluation of raspberry juice and wine made from Polka and Heritage cultivars. Thermo pectinase maceration significantly ($P < 0.05$) increased phenolic amounts, turbidity, and colour intensity in raspberry juice samples. But from juice to wine, the least reduction of phenolic amounts, turbidity, and colour intensity occurred in cold maceration (CM) treatments. In addition, the highest amount of phenolic compounds, the lowest turbidity, and the highest sensory scores were found in CM final wines from the Heritage cultivar. The study showed CM treatment was the most efficient pre-treatment maceration method for raspberry winemaking for its better phenolic maintenance and colour stability, and the Heritage cultivar was more suitable for winemaking than Polka for its higher phenolic content and lower turbidity.

Keywords: raspberry wine; phenolic profiles; pectinase; maceration

Red raspberry (*Rubus idaeus*) was referred to as golden fruit with good flavour, attractive colour, and potential health benefits (Yu et al. 2019; Yang et al. 2020). Fermentation into raspberry wine was regarded as an economical way for postharvest preservation and value-add of fruits (Guo et al. 2018; Jiang et al. 2020).

Wine quality is an overall balance of numerous factors, such as colour, flavour, body, and chemical stability, which is closely related to the profile and concentration of phenolic compounds (Ye et al. 2014; Guo et al. 2018). In the case of red wine production, the application of pectinase maceration could not only lead to higher juice yields, shorter maceration and settling time, but also offer qualitative benefits, such as improvement of colour, tannin, and flavour extraction (Liu et al. 2017; Claus and Mojsov 2018; Jiang et al. 2020). However, in some cases, the use of pectinase only resulted in a very slight improvement in the phenolic composition (A'lvarez et al. 2006). It was reported that the interaction between the extracted phenolic compounds and the suspended cell walls component could be responsible for the phenolic content in the final wine when maceration enzymes were used. As the components of the cell walls show high affinity for phenolic compounds and adsorb them into their structure, a large part of the extracted phenolic compounds will not become part of the final wine's phenolic composition, affecting its chromatic and other physicochemical characteristics (Renard et al. 2017; Pedro et al. 2020; Osete-Alcaraz et al. 2022).

Cold maceration (CM) was usually applied to increase wine colour stability as well as improve taste and flavour (Wang et al. 2016; Aleixandre-Tudo and Du Toit 2018). High temperature applied during thermo-maceration (TM) also could promote ruptures on the fruit cell wall, facilitating the extraction of phenolic compounds (Aguilar et al. 2016; Liu et al. 2017; Wojdyłoa et al. 2021). As enzymatic hydrolysis was accompanied

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by the maceration process, maceration temperatures and duration time played an important role in enzymatic activities and maceration effects, which would lead to the different phenolic extraction and their maintenance in the final wine (Martino et al. 2013; Aleixandre-Tudo and Du Toit 2018). Raspberries are soft fruits containing high quantities of pectin, so pectinase maceration is necessary for juice yield and winemaking. However, pectinase maceration conditions of raspberry are usually determined only by the juice yield (He et al. 2017; Wang et al. 2021), and the information is scarce on the pectinase macerating technologies on the physicochemical property of the raspberry juice and wine. In addition, different cultivars could make different responses to the pectinase maceration process (González--Neves et al. 2013; Zhang et al. 2018).

Therefore, this study aimed to investigate the effects of different pectinase maceration conditions on the physicochemical characteristics, phenolic profiles, and sensory attributes of those raspberry juice and wine obtained from Polka and Heritage cultivars. Results will help us better understand the effects of pectinase maceration on the physicochemical property, and also establish a foundation for quality improvement of raspberry wine and acceleration of its industrialisation.

MATERIAL AND METHODS

Plant material. The material used in this study was Heritage and Polka cultivar collected of raspberry at over 80% bright red maturity at Damugua village, Fansi County, Shanxi Province, China. The Heritage was a red colour raspberry cultivar with lower pectin content. The Polka was a pink colour cultivar with higher acid and firmer texture (Dragišić Maksimović et al. 2013). Raspberry was stored in frozen conditions (BC/BD-447SH; AUCMA, China) before being processed.

Chemical reagent. Folin-Ciocalteu reagent was obtained from Solarbio (China). Thirteen standard samples, cyanidin-3-O-glucoside, proanthocyanidins, quercetin, catechin, epicatechin, ellagic acid, gallic acid, vanillic acid, caffeic acid, salicylic acid, chlorogenic acid, rutin, quercetin, kaempferol, were purchased from Shanghai Anpu Biotechnology Co., Ltd. (China).

Preparation for pectinase treatment and fermentation. For each variety, raspberry was thawed for 24 h with 30 ppm potassium metabisulfite, then they were ground by hands with pectinase preparation: Rohavin® Color (RCO) (AB, Germany), which was used at the dosage of 30 mg L^{-1} recommended by the manufacturers. This maceration pectinase contains endo-polygalacturonase (PG), pectinesterase (PE), pectin lyase (PL), and cellulose, with a declared minimum activity of PE 630 unit (U) g^{-1}.

Pectinase maceration was carried out (LRH-150; Shanghai YiHeng, China) at 50 °C for 1 h (TM), 30 °C for 4 h [normal maceration (NM)], and 10 °C for 12 h (CM). Maceration temperature and time were determined by previous experiments according to the juice yield. After press, and then the six variants of juice samples were labelled as HT-J, PT-J, HN-J, PN-J, HC-J, PC-J, as shown in Table 1.

The juice sample was allowed to settle for over 12 h at a low temperature (5 °C) (FCD-238SE; Haier, China), and then take the supernatant for fermentation. Fermentation was performed after being adjusted to 20°Brix by addition of glucose (ameliorated), and inoculated with a commercial yeast *Saccharomyces cerevisiae* VP5 (Voson, Italy) at a dose of 0.2 g L^{-1}, and was conducted in an incubator room (LRH-150; Shanghai YiHeng, China) at 15 °C for 5 days. After fermentation finished, the wine was added with 50 ppm potassium metabisulfite and matured at 15 °C for 4 months (Lim et al. 2012). The wine samples were labelled as HT-W, PT-W, HN-W, PN-W, HC-W, and PC-W for analysis, as shown in Table 1. All treatments were run in triplicate.

Determination of physicochemical parameters. Raspberry juice and wine were analysed for basic physicochemical characteristics, such as titratable acid (TA), pH, sugar content, and alcohol content, in the way described by Wojdyłoa et al. (2021). Turbidity was measured with a turbidimeter Turbiquant 3000T (Merck, Germany). Colour intensity was calculated as the sum of absorbance at 620, 520, and 420 nm (Osete-Alcaraz et al. 2022).

Table 1. Labels of raspberry juice and wine samples

Treatments of different pectinase macerations	Juice (J)		Wine (W)	
	Heritage (H)	Polka (P)	Heritage (H)	Polka (P)
TM (50 °C, 1 h)	HT-J	PT-J	HT-W	PT-W
NM (30 °C, 4 h)	HN-J	PN-J	HN-W	PN-W
CM (10 °C, 12 h)	HC-J	PC-J	HC-W	PC-W

TM – thermo-maceration; NM – normal maceration; CM – cold maceration

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The total phenolic content (TPC) and the total anthocyanin content (TAC) were determined by Folin-Ciocalteau assay and modified pH shift assay respectively (Zhang et al. 2018). All measurements were run in triplicate.

Quantitative analysis of individual phenolic compounds by high performance liquid chromatography-mass spectrometry (HPLC-MS). Extraction of phenolic compounds was carried out in neutral and acidic conditions, as described by Ye et al. (2014). Juice or wine sample (10 mL) was extracted by 120 mL of ethyl acetate at pH 7.0 and pH 2.0, respectively. The combined organic phase was evaporated to dryness on a vacuum rotary evaporator at 35 °C (RE--2000A; YingYu, China). The dry residue was dissolved with 10 mL of methanol and then filtered through a 0.22 μm organic membrane filter (Merck, Germany) before injection into the high performance liquid chromatography-mass spectrometry (HPLC-MS) system. The detection system was carried out on a QTRAP 4500 LC/MS (AB SCIEX Inc., US) with a Thermo Hypersil GOLD C18 column (50 mm × 2.1 mm, 1.8 μm particle size). Water with 0.1 % formic acid and acetonitrile was used as mobile phase A and phase B, respectively, with a flow rate of 0.30 mL min^{-1}. The gradient program of phenolic compounds separation was as follows (time, % phase B): 0 min, 5%; 0.5 min, 5%; 3 min, 95%; 4 min, 95%; 4.1 min, 5%; 5 min, 5%. The mass spectrometer was done through an electrospray interface (ESI) in negative ionisation mode for the individual phenolics. The capillary temperature was 600 °C, the sheath gas flow rate was 60 arbitrary units (arb), the auxiliary gas flow rate was 10 arb, and the spray voltage was set to 5 500 V. Spectra was acquired in full-scan mode with 100–1 000 m/z. The quantity of all individual compounds was calculated using the calibration curve designed by reading the peak areas of standard solutions at six different concentrations (10, 20, 50, 100, 500, 1 000 μg L^{-1}) and expressed as mg L^{-1}.

Sensory evaluation. Raspberry wine was evaluated for sensory qualities on the basis of colour/appearance, flavour/aroma, body, taste, and overall acceptability on 10 points hedonic scale. A taste panel that consisted of trained judges (7–9 members at a time) evaluated the samples, who were university staff familiar with wine consumption. Prior to evaluation, a session was held to take the commercial raspberry wine as a reference to familiarise the panellists with the product. The judges were asked to read through the questionnaires and the meaning of each attribute, and they were not allowed to discuss their scores during the evaluation sessions. The average scores for each attribute would be the final result.

Statistical analysis. Analysis of variance (ANOVA) and principal component analysis (PCA) was carried out using SPSS 20.0 statistical package for Windows. All analyses were done in triplicate.

RESULTS AND DISCUSSION

Physicochemical properties of raspberry juice and wine. Tables 2, 3 showed the effects of different pectinase maceration conditions on the physicochemical properties of raspberry juice and wine. The juice recovery of all treatments was over 72%. TM treatment significantly increased TPC, turbidity, TAC, and colour intensity in raspberry juice samples. This result was consistent with the results reported in the literature (Martino et al. 2013; Wang et al. 2016). It could be explained by multiple effects of temperature on the mass-transfer process such as improved diffusion, denaturation of the plant matrix, and improvement of solvent characteristics in terms of penetration and solubility of anthocyanins (Zhu et al. 2016).

However, a significant decline in TPC, turbidity, TAC, and colour intensity was found in all treated wines after fermentation and ageing. Most importantly, from juice to wine, the maximum reduction of turbidity (72.2%), colour intensity (14.4%), TPC (31.5%) happened to the TM wine samples, while the minimum reduction of turbidity (36.8%), colour intensity (7.5%), TPC (9.3%) and TAC (4.4%) were obtained by CM wine samples. It showed that CM juice is more stable than TM juice, and the highest phenolic amount and the lowest turbidity were obtained in CM wines. In addition, CM increased the colour stability of raspberry wines, as for the minimum reduction of colour intensity.

One reason for low phenolic attendance from juice to wine in TM samples might be the interaction between the extracted phenolic compounds and the cell wall polysaccharides suspended in the juice. Osete--Alcaraz et al. (2022) demonstrated that the cell wall polysaccharides components showed a high affinity for phenolic compounds and adsorbed them into their structure. In our study, TM juice samples showed the greatest turbidity and TPC, indicating that the greatest amount of suspended cell wall polysaccharides components and the phenolic compounds were generated. Once the binding occurs between the extracted phenolic compounds and the polysaccharides components, these interactions are difficult to reverse and their complex would be precipitated during the fermentation and ageing period (Renard et al. 2017; Pedro et al. 2020). This also can affect its chromatic characteristics.

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Table 2. Physicochemical properties of raspberry juice obtained in different conditions (mean ± SD; n = 3)

Sample	Yield (%)	TA (g L^{-1})	pH	Total sugar (g L^{-1})	Turbidity (NTU)	Colour intensity	TPC (g L^{-1})	TAC (mg L^{-1})
PT-J	76.29 ± 3.81^{a}	14.54 ± 0.68^{c}	3.15 ± 0.09^{b}	78.40 ± 3.92^{bc}	185.53 ± 9.28^{a}	5.15 ± 0.08^{c}	2.19 ± 0.06^{b}	361.13 ± 18.06^{d}
PN-J	75.81 ± 3.84^{a}	15.38 ± 0.71^{b}	3.16 ± 0.02^{b}	86.27 ± 2.81^{c}	72.62 ± 3.63^{c}	4.70 ± 0.14^{d}	1.89 ± 0.04^{d}	312.61 ± 15.63^{e}
PC-J	72.93 ± 3.65^{c}	16.82 ± 0.45^{a}	3.18 ± 0.06^{b}	99.22 ± 4.96^{a}	40.85 ± 1.54^{e}	4.38 ± 0.12^{e}	1.75 ± 0.02^{e}	273.79 ± 13.67^{f}
HT-J	77.35 ± 3.87^{a}	13.55 ± 0.43^{d}	3.22 ± 0.10^{a}	69.30 ± 3.47^{d}	164.27 ± 8.21^{b}	5.72 ± 0.06^{a}	2.35 ± 0.03^{a}	633.04 ± 51.65^{a}
HN-J	74.13 ± 3.71^{b}	13.87 ± 0.39^{bc}	3.23 ± 0.04^{a}	81.37 ± 4.07^{b}	43.59 ± 2.18^{d}	5.50 ± 0.09^{b}	2.26 ± 0.03^{a}	589.20 ± 29.46^{b}
HC-J	72.03 ± 1.60^{c}	14.61 ± 0.53^{b}	3.25 ± 0.06^{a}	95.33 ± 5.07^{a}	27.82 ± 0.89^{f}	5.11 ± 0.16^{c}	2.03 ± 0.05^{c}	470.28 ± 23.66^{c}

$^{a–f}$Values in the same column with different letters showed statistically significant differences (P < 0.05) according to the Duncan test (n = 3); SD – standard deviation; TA – titratable acid; NTU – unit of scattered turbidity; TPC – total phenolic content; TAC – total anthocyanin content; for an explanation of the samples' abbreviations see Table 1

Table 3. Physicochemical properties of raspberry wine obtained in different conditions (mean ± SD; n = 3)

Sample	Alcohol content (V%)	pH	TA (g L^{-1})	Total sugar (g L^{-1})	Turbidity (NTU)	Colour intensity	TAC (mg L^{-1})	TPC (g L^{-1})
PT-W	8.65 ± 0.38^{b}	3.13 ± 0.02^{b}	12.86 ± 0.59^{c}	4.12 ± 0.21^{b}	55.06 ± 2.75^{a}	4.56 ± 0.23^{a}	318.81 ± 10.94^{e}	1.52 ± 0.03^{d}
PN-W	8.95 ± 0.40^{b}	3.12 ± 0.01^{b}	13.49 ± 0.32^{b}	4.42 ± 0.21^{b}	35.44 ± 1.77^{c}	4.42 ± 0.22^{c}	264.84 ± 19.24^{d}	1.62 ± 0.03^{bc}
PC-W	9.47 ± 0.42^{a}	3.10 ± 0.02^{b}	14.35 ± 0.65^{a}	4.58 ± 0.38^{b}	39.36 ± 1.97^{b}	4.25 ± 0.21^{d}	283.33 ± 18.17^{d}	1.65 ± 0.03^{b}
HT-W	7.81 ± 0.34^{c}	3.20 ± 0.02^{a}	11.64 ± 0.23^{e}	5.09 ± 0.25^{a}	42.74 ± 3.14^{b}	4.73 ± 0.23^{a}	550.32 ± 32.52^{a}	1.59 ± 0.04^{c}
HN-W	8.83 ± 0.39^{b}	3.18 ± 0.03^{a}	12.33 ± 0.57^{d}	5.23 ± 0.26^{a}	20.14 ± 0.21^{d}	4.55 ± 0.23^{ab}	482.87 ± 29.14^{b}	1.65 ± 0.02^{b}
HC-W	9.28 ± 0.41^{a}	3.17 ± 0.02^{a}	13.05 ± 0.60^{bc}	5.12 ± 0.31^{a}	11.43 ± 0.57^{e}	4.50 ± 0.13^{b}	446.10 ± 17.31^{c}	1.77 ± 0.03^{a}

$^{a–e}$Values in the same column with different letters showed statistically significant differences (P < 0.05) according to the Duncan test (n = 3); SD – standard deviation; TA – titratable acid; NTU – unit of scattered turbidity; TPC – total phenolic content; TAC – total anthocyanin content; for an explanation of the samples' abbreviations see Table 1

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Therefore, it is worthwhile to understand which phenolic compounds could be responsible for the effects of different pectinase maceration.

Contents of phenolic compounds of raspberry juice and wine. Phenolic profiles make a great contribution to the organoleptic quality, health benefits, and chemical stability of wine (Ye et al. 2014; Liu et al. 2017; Guo et al. 2018; Jiang et al. 2020). As shown in Tables 4, 5, 13 kinds of individual phenolic compounds in the raspberry juice and wine were determined. Regarding those identified individual phenolic profiles, the flavan-3-ols was the most dominant group, accounting for more than 75% of total phenols. Wojdyłoa et al. (2021) reported flavan-3-ols accounted for the majority of total phenolics (TPs) (63–90%) in Dornfelder red wine. But in persimmon wine, gallic acid accounts for more than 75% of TPs content (Liu et al. 2017).

The effects of different pectinase maceration on the phenolic profiles of raspberry juice and wine samples were investigated (Tables 4, 5). MS chromatogram of individual phenolic compounds standards (Figure S1) and those compounds in the real wine samples (Figure S2) are provided in the electronic supplementary material (ESM; for ESM see the electronic version). TM juice samples obtained significantly higher levels of TPC, which was mainly contributed by the markedly increases of the flavan-3-ols. But from juice to wine, the reduction rate of the total identified phenolics of TM samples was 59.2%, only 27.6% for CM samples. In addition, the highest amount of total identified phenolics was in CM wine samples. Those results were in accordance with the changes in TAC and TPC as shown in Tables 2, 3. Furthermore, TM increased the turbidity of juice and wine samples which was in accordance with the high content of flavan-3-ols. It was reported that noncovalent interactions between proanthocyanidins and cell wall material were responsible for the 'natural fining' occurring spontaneously during wine production (Aguilar et al. 2016).

Colour plays an important role in red wine quality. Cyanidin-3-glucoside was one of the main anthocyanins in raspberry fruit (Yang et al. 2020). As shown in Table 4, cyanidin-3-O-glucoside in TM juice samples was highest but decreased a lot by a scale of about 38.3% from juice to wine. CM samples showed the best stability in colour intensity for the least reduction of 22.2%. Under cold pectinase maceration conditions, anthocyanins could undergo copigmentation by non-covalent physiochemical association with a colourless phenolic cofactor, which could help to stabilise

Table 4. Phenolics profiles of raspberry juice obtained in different treatments (mg L^{-1}) (mean ± SD; n = 3)

Profiles	PT-J	PN-J	PC-J	HT-J	HN-J	HC-J
Cyanidin-3-O-glucoside	3.23 ± 0.16^{b}	2.58 ± 0.13^{d}	2.16 ± 0.11^{e}	5.20 ± 0.26^{a}	3.30 ± 0.16^{b}	2.72 ± 0.19^{c}
Total of anthocyanins	**3.23 ± 0.16^{b}**	**2.58 ± 0.13^{d}**	**2.16 ± 0.11^{e}**	**5.20 ± 0.26^{a}**	**3.30 ± 0.16^{b}**	**2.72 ± 0.19^{c}**
Proanthocyanidins	123.17 ± 7.55^{b}	103.74 ± 6.68^{c}	76.69 ± 4.83^{d}	131.91 ± 7.64^{a}	108.27 ± 5.23^{c}	81.02 ± 3.04^{d}
Catechin	2.91 ± 0.15^{a}	2.31 ± 0.12^{b}	1.85 ± 0.07^{c}	2.83 ± 0.07^{a}	1.73 ± 0.09^{c}	1.40 ± 0.07^{d}
Epicatechin	2.79 ± 0.14^{a}	2.21 ± 0.09^{b}	1.73 ± 0.07^{c}	2.74 ± 0.14^{a}	1.71 ± 0.09^{c}	1.43 ± 0.07^{d}
Total of flavan-3-ols	**128.87 ± 7.58^{b}**	**108.26 ± 6.84^{c}**	**80.27 ± 4.68^{d}**	**137.48 ± 6.56^{a}**	**111.71 ± 5.31^{c}**	**83.85 ± 2.39^{d}**
Ellagic acid	2.37 ± 0.12^{c}	3.54 ± 0.20^{b}	4.63 ± 0.19^{a}	3.55 ± 0.18^{b}	3.89 ± 0.19^{b}	4.42 ± 0.12^{a}
Gallic acid	0.48 ± 0.01^{c}	0.49 ± 0.02^{c}	0.31 ± 0.01^{d}	0.83 ± 0.04^{a}	0.55 ± 0.03^{b}	0.32 ± 0.02^{d}
Vanillic acid	1.06 ± 0.05^{a}	0.97 ± 0.05^{b}	0.85 ± 0.04^{c}	0.74 ± 0.04^{c}	0.69 ± 0.04^{d}	0.73 ± 0.04^{c}
Caffeic acid	0.26 ± 0.01^{c}	0.35 ± 0.02^{b}	0.40 ± 0.02^{a}	0.33 ± 0.02^{bc}	0.39 ± 0.02^{b}	0.41 ± 0.02^{a}
Salicylic acid	0.18 ± 0.01^{a}	0.14 ± 0.01^{c}	0.18 ± 0.00^{a}	0.16 ± 0.01^{b}	0.11 ± 0.01^{d}	0.12 ± 0.01^{d}
Chlorogenic acid	0.55 ± 0.03^{b}	0.24 ± 0.01^{c}	0.16 ± 0.01^{d}	0.71 ± 0.09^{a}	0.23 ± 0.01^{c}	0.17 ± 0.01^{d}
Total of phenolic acids	**4.90 ± 0.20^{d}**	**5.73 ± 0.12^{c}**	**6.53 ± 0.19^{a}**	**6.32 ± 0.22^{a}**	**5.86 ± 0.12^{c}**	**6.17 ± 0.10^{b}**
Rutin	0.19 ± 0.01^{cd}	0.21 ± 0.01^{c}	0.23 ± 0.01^{c}	0.52 ± 0.03^{a}	0.45 ± 0.02^{b}	0.47 ± 0.02^{b}
Quercetin	0.70 ± 0.04^{d}	1.26 ± 0.06^{c}	0.70 ± 0.04^{d}	1.83 ± 0.09^{a}	1.99 ± 0.10^{a}	1.64 ± 0.08^{b}
Kaempferol	0.02 ± 0.00^{c}	0.03 ± 0.00^{b}	0.01 ± 0.00^{d}	0.04 ± 0.00^{a}	0.04 ± 0.00^{a}	0.02 ± 0.00^{c}
Total of flavonols	**0.91 ± 0.05^{d}**	**1.50 ± 0.05^{c}**	**0.94 ± 0.03^{d}**	**2.39 ± 0.04^{a}**	**2.48 ± 0.04^{a}**	**2.13 ± 0.06^{b}**
Total phenols	**137.91 ± 7.40^{b}**	**118.07 ± 4.62^{c}**	**89.90 ± 3.22^{d}**	**151.39 ± 5.51^{a}**	**123.35 ± 4.40^{bc}**	**94.87 ± 4.35^{e}**

$^{a–e}$Values in the same column with different letters showed statistically significant differences ($P < 0.05$) according to the Duncan test (n = 3); SD – standard deviation; for an explanation of the samples' abbreviations see Table 1

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Table 5. Phenolics profiles of raspberry wine obtained in different treatments (mg L^{-1}) (mean ± SD; n = 3)

Profiles	PT-W	PN-W	PC-W	HT-W	HN-W	HC-W
Cyanidin-3-O-glucoside	2.32 ± 0.07^{a}	1.88 ± 0.05^{d}	1.60 ± 0.03^{e}	2.55 ± 0.08^{a}	2.03 ± 0.05^{b}	1.94 ± 0.03^{c}
Total of anthocyanins	**2.32 ± 0.07^{a}**	**1.88 ± 0.05^{d}**	**1.60 ± 0.03^{e}**	**2.55 ± 0.08^{a}**	**2.03 ± 0.05^{b}**	**1.94 ± 0.03^{c}**
Proanthocyanidins	32.65 ± 6.32^{d}	26.66 ± 13.33^{e}	38.49 ± 19.24^{d}	49.47 ± 24.73^{c}	55.72 ± 27.86^{b}	58.41 ± 1.20^{a}
Catechin	0.66 ± 0.03^{d}	0.50 ± 0.03^{e}	0.71 ± 0.02^{c}	1.01 ± 0.05^{b}	1.11 ± 0.06^{b}	1.37 ± 0.07^{a}
Epicatechin	0.63 ± 0.01^{c}	0.50 ± 0.03^{d}	0.69 ± 0.03^{c}	1.00 ± 0.05^{b}	1.03 ± 0.05^{b}	2.40 ± 0.12^{a}
Total of flavan-3-ols	**33.94 ± 1.60^{e}**	**27.66 ± 0.68^{f}**	**39.89 ± 1.31^{d}**	**51.48 ± 1.93^{c}**	**57.86 ± 1.84^{b}**	**62.18 ± 2.21^{a}**
Ellagic acid	3.22 ± 0.16^{c}	3.12 ± 0.16^{c}	4.36 ± 0.22^{b}	4.44 ± 0.12^{b}	4.42 ± 0.12^{b}	5.72 ± 0.29^{a}
Gallic acid	0.74 ± 0.04^{b}	0.51 ± 0.03^{d}	0.56 ± 0.03^{d}	0.63 ± 0.01^{c}	0.97 ± 0.05^{a}	1.03 ± 0.02^{a}
Vanillic acid	1.50 ± 0.08^{a}	1.35 ± 0.04^{b}	1.47 ± 0.07^{a}	1.20 ± 0.06^{c}	1.24 ± 0.06^{c}	1.38 ± 0.07^{b}
Caffeic acid	0.84 ± 0.04^{d}	1.14 ± 0.06^{c}	1.41 ± 0.04^{b}	1.13 ± 0.01^{c}	1.42 ± 0.07^{b}	1.58 ± 0.06^{a}
Salicylic acid	0.54 ± 0.03^{b}	0.52 ± 0.02^{b}	0.59 ± 0.03^{a}	0.24 ± 0.01^{d}	0.33 ± 0.02^{c}	0.34 ± 0.01^{c}
Chlorogenic acid	0.57 ± 0.03^{b}	0.14 ± 0.01^{c}	0.09 ± 0.00^{d}	0.64 ± 0.03^{a}	0.08 ± 0.00^{e}	0.07 ± 0.00^{e}
Total of phenolic acids	**7.41 ± 0.20^{d}**	**6.78 ± 0.22^{e}**	**8.48 ± 0.29^{b}**	**8.28 ± 0.22^{c}**	**8.46 ± 0.22^{b}**	**10.12 ± 0.20^{a}**
Rutin	0.25 ± 0.01^{a}	0.15 ± 0.01^{b}	0.13 ± 0.01^{c}	0.08 ± 0.00^{e}	0.05 ± 0.00^{f}	0.12 ± 0.00^{d}
Quercetin	2.33 ± 0.42^{b}	2.27 ± 0.67^{c}	2.05 ± 0.75^{e}	3.61 ± 0.83^{a}	2.33 ± 0.77^{b}	2.17 ± 0.66^{d}
Kaempferol	0.20 ± 0.01^{f}	0.39 ± 0.02^{a}	0.31 ± 0.02^{c}	0.33 ± 0.02^{b}	0.28 ± 0.01^{d}	0.24 ± 0.01^{e}
Total of flavonols	**2.78 ± 0.35^{c}**	**2.81 ± 0.65^{b}**	**2.49 ± 0.66^{f}**	**4.02 ± 0.74^{a}**	**2.66 ± 0.64^{d}**	**2.53 ± 0.56^{e}**
Total phenols	**46.45 ± 1.72^{e}**	**39.13 ± 1.73^{f}**	**52.46 ± 1.96^{d}**	**66.33 ± 2.80^{c}**	**71.01 ± 1.99^{b}**	**76.77 ± 1.61^{a}**

$^{a–f}$Values in the same column with different letters showed statistically significant differences ($P < 0.05$) according to the Duncan test (n = 3); SD – standard deviation; for an explanation of the samples' abbreviations see Table 1

the structure of the anthocyanin and enhance colour intensity (Zhang et al. 2018). CM wine contained the most amount of the total phenolic acid, particularly for ellagic acid, caffeic acid, which were found to interact with anthocyanins to form copigments that largely improve the colour stability of red wines. Ellagic acid was considered to be a good copigment factor because of its large planar structure (Zhang et al. 2018). In addition, caffeic acid could also be considered for improving the chromatic intensity and colour stability of wines (Zhang et al. 2021).

Averagely, the total identified phenolic content of Heritage juice and wine was higher than that of the Polka cultivar. All individual phenolic compounds followed the same trend except for vanillic acid and salicylic acid, which would be the characteristic compounds for the Polka cultivar. Furthermore, Heritage contains more flavonols than Polka. From juice to wine, the reduction rates of Heritage and the Polka were 42.9% and 60.2% respectively, indicating Heritage was more stable during fermentation and ageing processes.

Sensory evaluation. The aged wine samples obtained by different pectinase maceration treatments were scored independently by members of the tasting team, and then added and averaged to get the final tasting results. The evaluation was conducted in five aspects: colour, fragrance, flavour, taste and overall acceptability, with 10 points for each. As can be seen from Figure 1, the total scores of CM raspberry wine from the Heritage cultivar were the highest with bright red colour, good flavour, balanced palate, and good overall acceptability, which was related to the highest content of TPs.

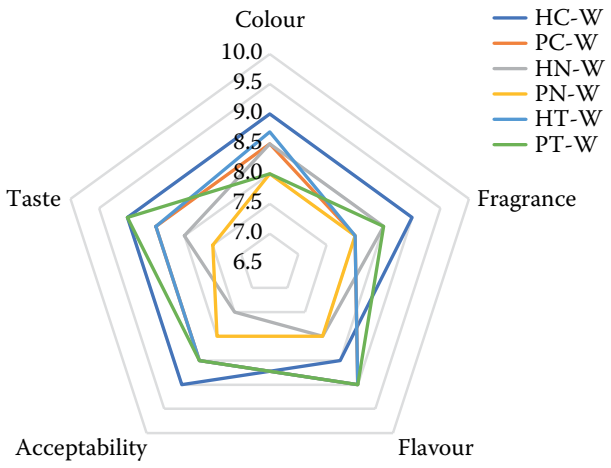

Figure 1. Sensory evaluation of different raspberry wine samples

For an explanation of the samples' abbreviations see Table 1

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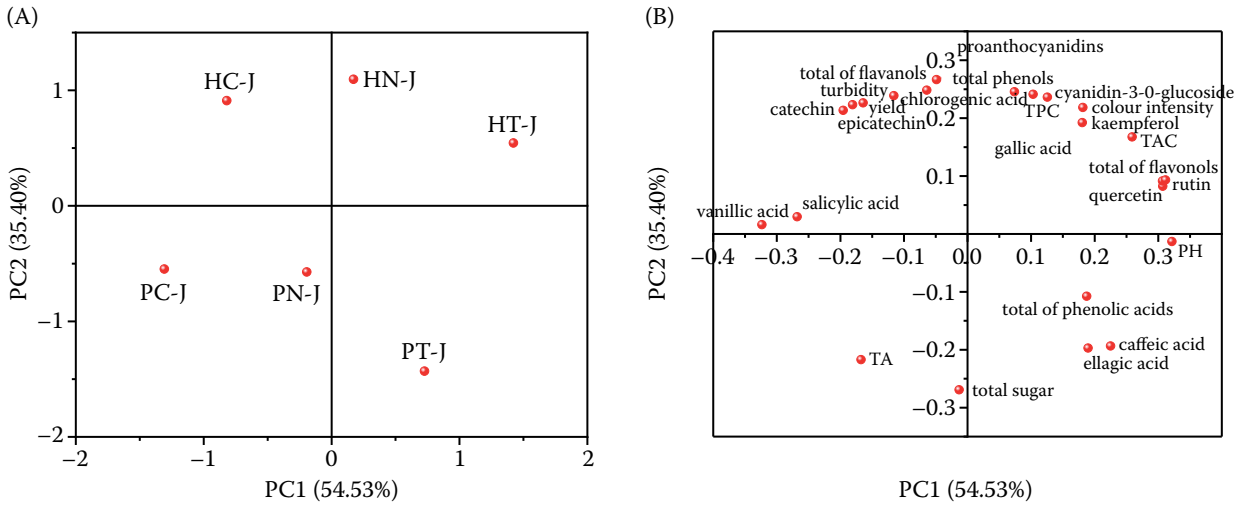

Figure 2. Principal component analysis (PCA) of different treatments as a function of physicochemical properties of raspberry juice: (A) classification of raspberry juice analysed in function of PC1 and PC2, (B) PCA loading of all analysed compounds

PC – principal component; TA – titratable acid; TPC – total phenolic content; TAC – total anthocyanin content; for an explanation of the samples' abbreviations see Table 1

Variety effect *versus* pectinase macerating conditions effect. PCA was performed to identify the special parameters for better discriminating the juice and wine with different pectinase maceration treatments, as shown in Figures 2, 3. For juice samples, the first two principal components (PCs) accounted for 89.93% of the variance, with PC1 accounting for 54.53%, and PC2 accounting for 35.40%. Pectinase maceration treatments were correctly separated on PC1, influenced mainly by the positive correlations with the total phenols content and the total flavan-3-ols. TM juice contains more flavan-3-ols, especially for proanthocyanidins, while CM juice contains more total sugar, caffeic acid and ellagic acids, which contribute to the colour stability. And PC2 separated juice into two raspberry cultivar groups, one is Polka; another is Heritage, influenced mainly by pH, the flavonols, vanillic acid and salicylic acid content. Polka juice had more vanillic acid, salicylic acid and lower pH. But

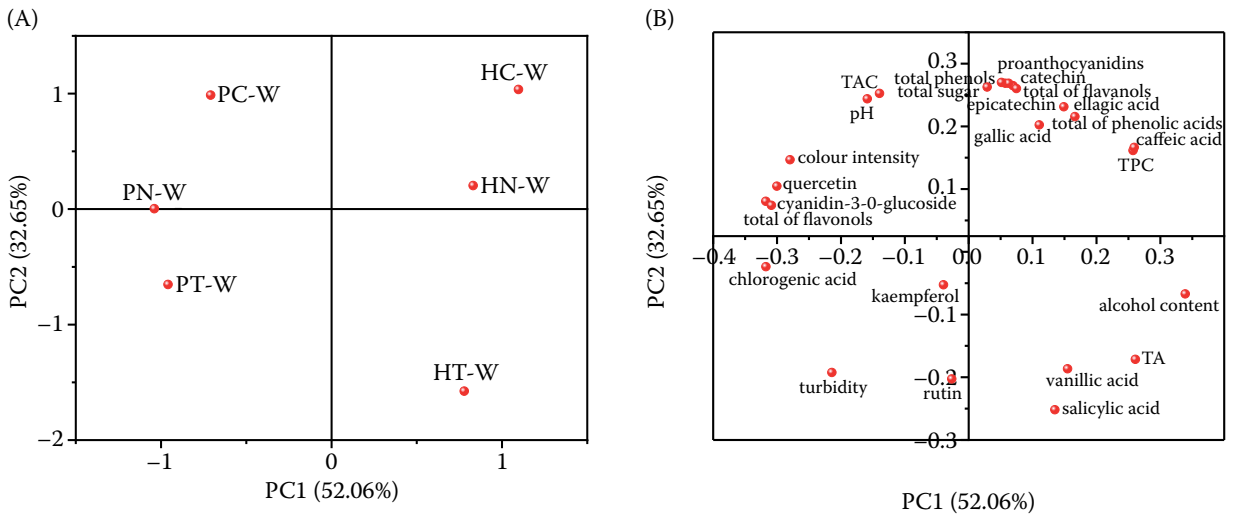

Figure 3. Principal component analysis (PCA) of different treatments as a function of physicochemical properties of raspberry wine: (A) classification of raspberry wine analysed in function of PC1 and PC2, (B) PCA loading of all analysed compounds

PC – principal component; TA – titratable acid; TPC – total phenolic content; TAC – total anthocyanin content; for an explanation of the samples' abbreviations see Table 1

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Heritage cultivar juice had more flavonols, which were responsible for high colour intensity.

For wine samples, the first two PCs accounted for 84.71% of the variance, with PC1 accounting for 52.06%, and PC2 accounting for 32.65%, indicating raspberry variety play a more important role in the final wine quality than the maceration conditions. Heritage wine had more total phenols, Polka wine had more vanillic acid and salicylic acid. But Heritage CM wine possesses the highest levels of the total phenolic acid and the total flavan-3-ols, ellagic acid and caffeic acid which contribute to the highest sensory scores among raspberry wine samples.

Therefore, pre-fermentation pectinase maceration treatment made a greater effect on juice physicochemical properties than cultivars, but its effect became less after fermentation and ageing. The effect of cultivars became more obvious in the wine differences.

CONCLUSION

Three pectinase maceration treatments (TM, 50 °C for 1 h; NM, 30 °C for 4 h; CM, 10 °C for 12 h) were tested for their effects on the quality of raspberry juice and wine obtained from Polka and Heritage cultivars. The study identified CM treatment as the most efficient pre-treatment maceration method for raspberry wine-making for its better phenolic maintenance and colour stability. Phenolic analysis showed TM markedly decreased the flavan-3-ols content, particularly for proanthocyanidins, and CM was helpful for the extraction of phenolic acid, particularly for ellagic acid and caffeic acid. The interaction between higher amounts of flavan-3-ols and cell wall polysaccharides could result in the greater loss of the phenolic compounds in the TM wine, and higher levels of elegiac acid and caffeic acid in CM samples might contribute to the colour stability. In addition, vanillic acid and salicylic acid would be characteristic compounds for the Polka variety, but the Heritage cultivar was more suitable for wine-making than Polka for its higher phenolic content and lower turbidity. These results could better understand the effects of pectinase maceration and cultivars on phenolic extraction and maintenance, and promote the establishment of optimal conditions for high-quality raspberry wine production.

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