Acta Alimentaria 52 (2023) 1, 91–101
DOI: 10.1556/066.2022.00187

AKADÉMIAI KIADÓ

Enhancement of aromatic and sensory profile of jujube wines fermented with fresh *Zizyphus jujuba* cv. Muzao at the white-ripe period

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ORIGINAL RESEARCH PAPER

Received: September 19, 2022 • Accepted: December 22, 2022
Published online: March 6, 2023

ABSTRACT

In this paper, fresh Muzao (*Zizyphus jujuba* cv. Muzao) at full-red and white-ripe periods were used as raw materials to brew low-alcohol jujube wines directly without cooking or extraction. The results showed that the contents of total acid, total phenolics, and total tannin of white-ripe jujube wines (WRJW) were significantly higher than that of full-red jujube wines (FRJW) under the same ratio of jujube fruit/water ($P < 0.05$). When the ratio of jujube fruit to water increased from 1:1 to 1:5, the total esters contents of WRJW increased from 2261.56 $\mu g\,L^{-1}$ to 3671.51 $\mu g\,L^{-1}$, but decreased in FRJW. Especially, the contents of ethyl octanoate, ethyl decanoate, and isoamyl caprylate in WRJW (1:5) were significantly higher than in other wine samples ($P < 0.05$). These three esters with a variety of aroma description characteristics can give jujube wine a more complex flavour. The sensory evaluation also showed that the WRJW (1:5) had the highest score. This wine had the following characteristics: clear and transparent, light yellow, pure elegant fruit and wine aroma, pleasant fragrance, harmonious wine body, fresh taste, sweet and sour, with typical characteristics of jujube wine. The research results can provide a theoretical basis and technical reference for the industrial production of high-quality jujube wine.

KEYWORDS

Jujube wine, *Zizyphus jujuba* cv. Muzao, maturity, aroma compounds, sensory profile

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1. INTRODUCTION

Muzao (*Zizyphus jujuba* cv. Muzao), as one of the most famous 6 geographical indications jujube species with the largest export volume, is mainly cultivated in the Yellow River Valley areas of China (Mao et al., 2022). Muzao have rich nutritional and medicinal value (Ji et al., 2018; Tang et al., 2020). Jujube wine, as a fermented product, can greatly improve the economic value of jujube, since jujube has broad market prospects and high economic and social benefits (Xu et al., 2019). Currently, the fermentation technology of jujube wine that have been reported in the articles mainly include soaking dried jujube in spirits to make red jujube wine, steaming and extracting jujube juice from dried jujube for fermentation, or imitating the maceration and fermentation technique of red wine (Cai et al., 2020). However, the red jujube wine produced by these techniques is obviously not competitive with red wine in the market. Therefore, there is an alternative way to use fresh jujube without cooking and extraction to make a clear and yellowish low-alcohol jujube wine with a strong fruity and alcoholic flavour and fresh taste, which is well-loved by consumers (Yuan et al., 2021).

Different maturity of fresh jujubes have different sugar and acid ratio, phenolic substances, and aroma components, and the variability of these compounds can lead to significant differences in nutritional value and sensory quality of red jujube wine (Lee et al., 2018). However, to date, all studies focused on ripe jujubes and very little information indicate the differences of jujube wine using different maturity of fresh jujubes. In addition, the fermentation of jujube often requires the addition of water to dissolve the large amount of soluble solids, reducing sugars, polyphenols, and tannins in the jujube, as the amount of juice obtained directly from the jujube fruit is small (Lu et al., 2002). The optimal ratio of jujube fruit to water provides a good nutritional environment for the fermentation of jujube wine.

Therefore, the present study aimed to compare the differences in jujube wine brewed from fresh Muzao (*Zizyphus jujuba* cv. Muzao) at different maturity periods with different water additions. The physicochemical properties, volatile compounds, and sensory quality of jujube wine were evaluated with a view to providing a basis for the development of jujube wine adapted to market demand.

2. MATERIALS AND METHODS

2.1. Raw materials and chemicals

The fresh Muzao (*Zizyphus jujuba* cv. Muzao) was collected from Lvliang, Shanxi, China. The sampling dates corresponded to the white-ripe period jujube (WRJ, August 2020) and full-red period jujube (FRJ, October 2020). All fruit were packed and delivered to the laboratory and frozen at −20 °C before winemaking. Folin–Ciocalteu's phenol reagent, gallic acid, and catechin were purchased from Solarbio Science & Technology, Co., Ltd (Beijing, China). The HPLC grade of 2-octanol were obtained from Aladdin (Shanghai, China). All other reagents were of analytical grade and were used without further purification.

2.2. Jujube wine preparation

The frozen WRJ and FRJ were thawed, and then the jujube pit was separated from fresh jujube fruit. According to the ratio of jujube fruit to water, to the jujubes water was added, then were crushed and pumped into 10 L glass flasks. The ratio of the jujube fruit and water was 1:1, 1:3, and 1:5 (w/v). Sucrose was added into the fermentation broth to attain 15 °Bx, so that the initial sugar concentration was the same level for all samples, followed by the addition of sulphur dioxide (50 mg L^{-1}). After 2 h, 20 mg L^{-1} of pectolytic enzymes (Novozymes, Tianjin, China) were added. The ameliorate fermentation broth was inoculated with 0.03% (w/w) commercial active wine dry yeast of *S. cerevisiae* EXCELLENCE® XR (Lamothe Abiet; Canéjan, France), and static fermentation was implemented at 25 ± 1 °C. After 5 days of fermentation, 50 mg L^{-1} SO_2 was added to stop the fermentation. The jujube wine was filtered through filter plates to eliminate jujube flesh, and the resulting jujube wines were left for natural clarification for 7 days. After this, the wines were cold stabilised (−5 ± 1 °C) for 2 months prior to follow-up analysis. In summary, six different jujube wines were obtained from two mature stages of jujube, considering three different ratios of the jujube pulp and water.

2.3. Analytical techniques

Total soluble solids (TSS, °Brix), titratable acidity (TA), reducing sugars (RS), and ethanol content (EC, % vol) were determined using a WineScan instrument (WineScan™ FT120: Foss, Hillerød, Denmark). The content of total phenolics (TP) in the wine was established according to the Folin–Ciocalteu colorimetric method (Deng et al., 2011), and the total tannin (TT) (mg L^{-1} (+)-catechin) was measured as previously described (Rajković and Sredović, 2009).

The volatile compounds of jujube wines were determined by our previously described SPME GC-MS method (Wang et al., 2016a, 2016b) with slight modification. Equilibration time and absorption time was 10 and 30 min at 45 °C, respectively.

2.4. Sensory evaluation

Sensory analysis was conducted by 20 trained panellists (10 females and 10 males). Before the sensory evaluation, each wine was placed in a sample bottle and left undisturbed at room temperature for 1 h with the lid closed. Then, a quantitative sensory description was conducted using a graded 10-point scale (0 = weak, and 10 = strong) to measure the colour, clarity, taste, aroma, and typicality. Detailed sensory evaluation rules are listed in Supplementary Table 1.

2.5. Statistical analysis

Data processing was performed using the statistical package Statistica. The one-way analysis of variance (ANOVA) and least significant difference test (LSD) were used to measure variations between treatments at a probability level of $P < 0.05$. Principal component analysis (PCA) was performed with XLSTAT 2021.2.2. Cluster heat map analysis was performed using hiplot on-line (https://hiplot.com.cn) to display the correlation matrix.

3. RESULTS AND DISCUSSION

3.1. Conventional and polyphenolic compounds

As expected, increasing the addition of water significantly decreased the content of TSS, TA, TP, and TT in jujube wine (Table 1). The RS in the jujube wine (1:1) was significantly higher than that of other jujube wines (1:3 and 1:5) after 5 days of fermentation ($P < 0.05$), although the starting degrees Brix was adjusted consistently. A higher proportion of jujube fruit may result in

Table 1. Oenological parameters of jujube wines produced with different treatment after being cold stabilised for two months

Treatment	Ethanol (% vol)	Reducing sugar (g L^{-1})	Soluble solids (%)	Titratable acidity (g L^{-1})	Total phenolics (g L^{-1})	Total tannin (g L^{-1})
Full-red period (1:1)	9.8 ± 0.4^{a}	45.53 ± 6.42^{a}	5.3 ± 0.3^{b}	7.82 ± 0.46^{b}	2.92 ± 0.22^{b}	1.88 ± 0.12^{b}
Full-red period (1:3)	9.2 ± 0.5^{a}	22.26 ± 4.38^{c}	5.0 ± 0.5^{b}	7.55 ± 0.31^{b}	2.08 ± 0.16^{c}	1.16 ± 0.11^{d}
Full-red period (1:5)	9.5 ± 0.8^{a}	30.76 ± 4.91^{b}	4.2 ± 0.2^{c}	6.61 ± 0.42^{c}	1.42 ± 0.22^{d}	1.12 ± 0.08^{d}
White-ripe period (1:1)	8.3 ± 0.9^{ab}	52.86 ± 4.42^{a}	7.6 ± 0.4^{a}	9.96 ± 0.92^{a}	4.31 ± 0.32^{a}	2.31 ± 0.10^{a}
White-ripe period (1:3)	8.3 ± 0.3^{b}	27.61 ± 2.17^{bc}	5.2 ± 0.5^{b}	7.01 ± 0.31^{bc}	3.25 ± 0.11^{b}	1.52 ± 0.03^{c}
White-ripe period (1:5)	8.6 ± 0.6^{ab}	32.93 ± 5.33^{b}	5.0 ± 0.4^{b}	6.76 ± 0.37^{c}	2.34 ± 0.15^{c}	1.66 ± 0.07^{b}

Values are presented as mean ± SD ($n = 3$). Different letters in each column indicate significant differences according to LSD test ($P < 0.05$).

the presence of more TSS in jujube juice, such as polysaccharides, which can be converted to fermentable sugars during fermentation. Another explanation could be that Muzao has a higher fructose content than glucose (Gao et al., 2012), which is preferentially digested by *S. cerevisiae* (Tronchoni et al., 2009), resulting in more fructose being residual in the jujube wine (1:1) after the same fermentation time.

On the other hand, under the same ratio of jujube fruit to water, the EC of full-red period jujube wines (FRJW) was significantly higher than that of white-ripe period jujube wines (WRJW) ($P < 0.05$), while the contents of RS and TSS were the opposite, indicating that the fermentation rate of FRJW was faster than that of WRJW (Table 1). Furthermore, the contents of TA, TP, and TT in WRJW were significantly higher than those in FRJW ($P < 0.05$) at the same water additions. This is because the acidity and polyphenol contents of Muzao gradually decrease with the colour turning and ripening (Wu et al., 2012), leading to the extraction of more organic acids and polyphenols in WRJW. Therefore, making wine with WRJ as raw material not only improved the nutritional value of jujube wine, but also enhanced the wine body and made it more 'backbone' in sensory terms (Xu et al., 2019).

3.2. Volatile compounds

Esters are the main aroma compounds in jujube wine, which generally have pleasant fruity odours and are mainly produced in the alcoholic fermentation stage (Komes et al., 2015). With the increase of water addition, the total ester content of FRJW decreased, while the total ester content increased from 2261.56 µg L^{-1} to 3671.51 µg L^{-1} in WRJW (Fig. 1). The increase of esters in jujube wines can give the wine richer fruit and flower odours. Under esterase catalysis, an initial activation of the acid and ethanol can synthesise fatty acid ethyl ester, and this fatty acid is formed from the ethanolysis of fatty acyl-CoA (Borren and Tian, 2020; Tufariello et al., 2021). Among the 11 ethyl esters detected, increasing the amount of water addition decreased the contents of ethyl butyrate and ethyl isovalerate, but the contents of longer-chain ethyl esters gradually increased in all jujube wines and then decreased in FRJW (1:5) (Fig. 2). This explanation could be that the synthesis of ethyl esters started preferentially from the combination of short-chain fatty acids with ethanol, and the synthesis gradually progressed to the longer-chain fatty acids as the more water was added resulting in a low proportion of jujube fruit. Furthermore, because the macromolecules were less decomposed in unripe Muzao, the highest concentration of longer-chain ethyl esters remained in WRJW (1:5) and contributed to the aroma complexity. Except for the ester, benzeneacetaldehyde, noted as honey and floral flavour (Pino and Fajardo, 2011) content, which was significantly lower in jujube wine (1:1) were than the other jujube wines (Supplementary Table 2).

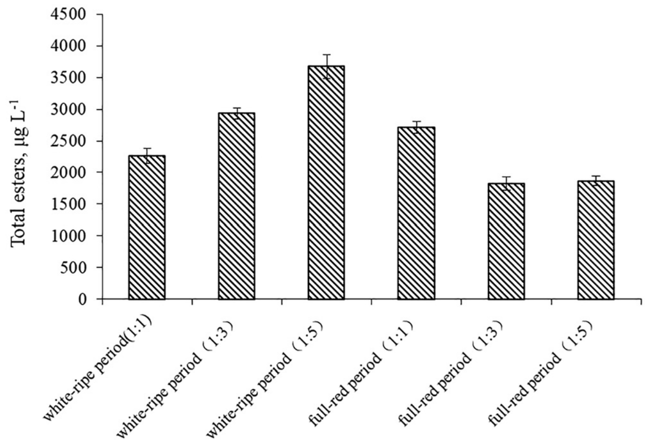

Fig. 1. The total esters contents of different jujube wines

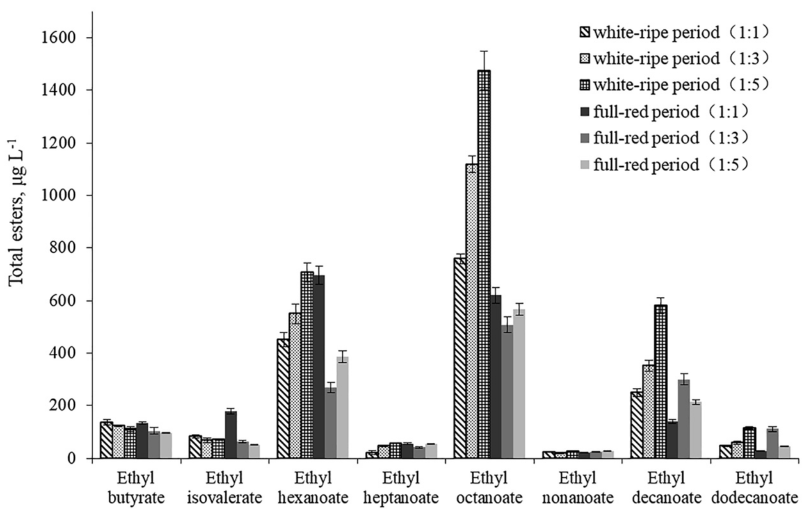

Fig. 2. The ethyl esters contents of different jujube wines

The synergistic superposition of aroma compounds with OAV (Odour activity value) higher than 0.1 may increase the complexity of wine aroma (Capone et al., 2013). The PCA of aromatic profiles (OAV > 0.1) was undertaken, and PC1 and PC2 accounted for 70.37% of the total variability. FRJW and WRJW were well distinguished on both sides of PC1 (Fig. 3A). In the loading plot shown in Fig. 2B, most aroma compounds were located on the right side, negatively linked with PC1, indicating that the content of aroma compounds in WRJW was higher, giving WRJW more complex flavour.

With the increase of water addition, the jujube wines gradually moved from the positive dimensions of PC2 to the negative (Fig. 3A). However, PC3, which explained 24.31% of the total variability, showed different trends between WRJW and FRJW with the increase of water

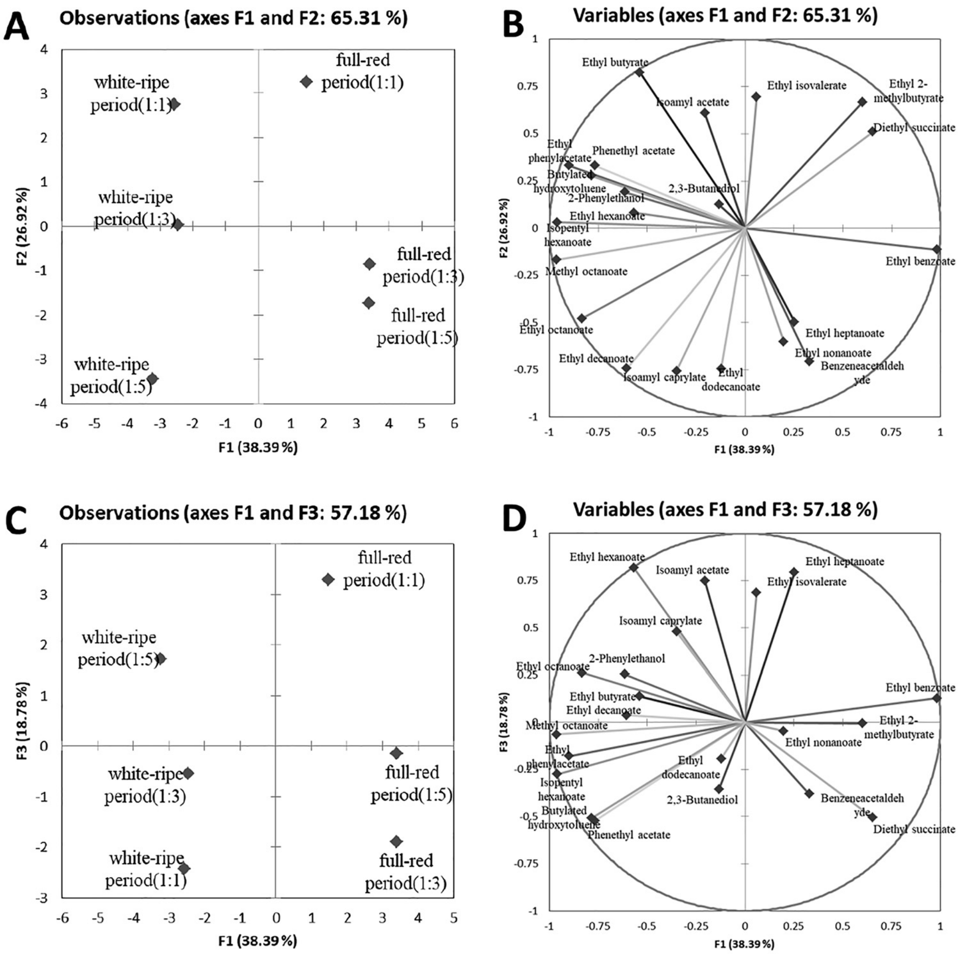

Fig. 3. Principal component analysis (PCA) with the results for aroma compounds in different jujube wines. (A and B) Graph of scores and loadings for PC1 (factor 1) and PC2 (factor 2), (C and D) Graph of scores and loadings for PC1 (factor 1) and PC3 (factor 3)

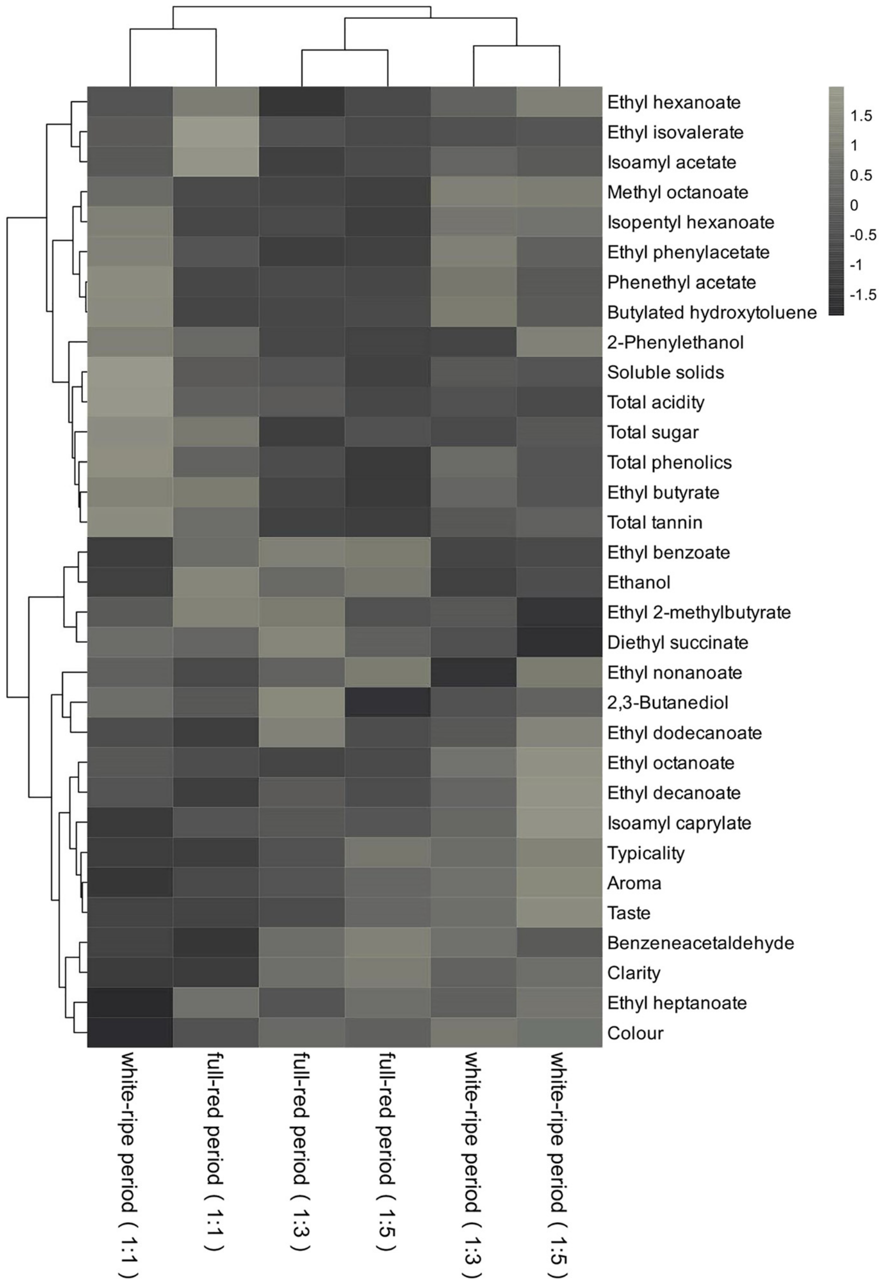

Fig. 4. Heat map analysis of jujube wines based on oenological parameters and volatile compounds

addition (Fig. 3C), indicating that the effect of increasing water addition on the different maturity of jujube wines was not consistent. In terms of the WRJW (1:5) with the highest total esters content, it can be found that the position sides of PC2 and PC3 in the loading plot are all loaded with esters of ethyl octanoate, ethyl decanoate, and isoamyl caprylate (Fig. 3B and Fig. 3D). These three esters not only have OAV higher than 0.1, but also contribute more complex odours of fruit, sweet, fat, cheese, and floral to jujube wines (Cai et al., 2014).

3.3. Sensory characteristic analysis and heat map analysis (HMA)

The spider web plot of jujube wine (Fig. 5) sensory profiles indicated that the total variance of all wine samples according to variance analysis had significant differences in each sensory parameter ($P < 0.05$). Pairwise multiple comparisons showed that jujube wines (1:1) in colour and

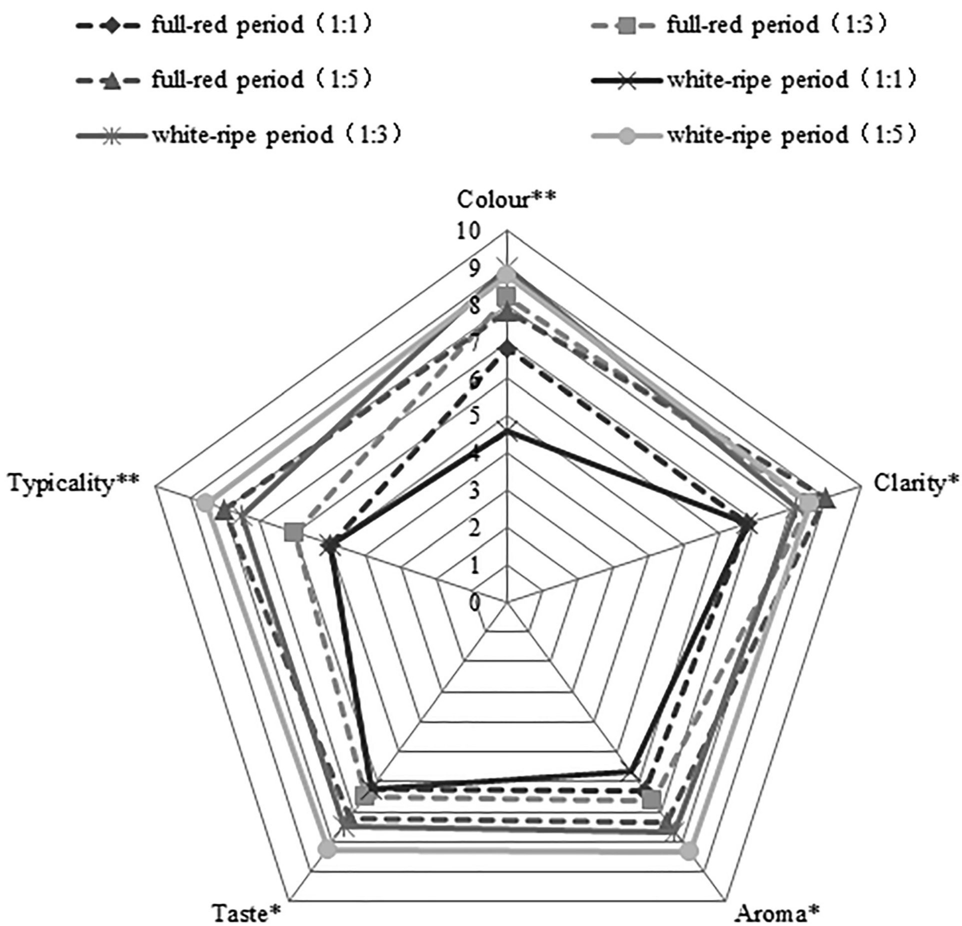

Fig. 5. Sensory evaluation of different jujube wines
Significance levels are analysed by LSD as follows: *: $P < 0.05$ and **: $P < 0.01$.

clarity notes were significantly different from other jujube wines (jujube fruits/water were 1:3 and 1:5) ($P < 0.05$). Additionally, WRJW (1:5) in taste and aroma was significantly different from other jujube wines ($P < 0.05$). WRJW (1:5) had the highest aroma, taste, and typicality scores.

HMA based on the relative abundance of physicochemical parameters and volatile compounds showed that the jujube wine samples were divided into two clusters (Cluster-I and Cluster-II) (Fig. 4). Cluster-I contained the samples of jujube wines (1:1), characterised by the low level of sensory profiles and volatile compounds, which probably led to the imbalance of jujube wines, even though the contents of non-volatile compounds in jujube wines (1:1) were higher. Cluster-II further segregated into WRJW (1:3 and 1:5) (IIA) and FRJW (1:3 and 1:5) (IIB). The clustering results showed that water had a major effect on the quality of jujube wine when the amount of water was low. However, with the increase of water content, the influence of maturity on the quality of jujube wine became a dominant factor. Cluster-IIB contained the samples of FRJW (1:3 and 1:5), characterised by the low concentrations of volatile and non-volatile compounds, which affect the sensory quality leading to a light style of jujube wine.

4. CONCLUSIONS

In the current study, WRJW had higher polyphenols contents than FRJW under the same operating conditions. The correlation analysis showed that the dominant influencing factor of jujube wine quality gradually changed from water additions to maturity when water amount was increased. With the increase of water content, the total esters content in WRJW increased, but it was opposite for FRJW. Especially, WRJW (1:5) had the highest contents of ethyl octanoate, ethyl decanoate, and isoamyl caprylate, and therefore had a more complex flavour. This wine was clear and transparent, light yellow, with pure elegant fruit and wine aroma, pleasant fragrance, harmonious wine body, fresh taste, sweet and sour, and typical characteristics of jujube wine. In conclusion, white-ripe period Muzao can be used to ferment excellent jujube wine under suitable conditions of adding water.

Conflict of interest: The authors declare no competing financial interest.

ACKNOWLEDGEMENTS

This project was supported by the Special Scientific Research Project of Agricultural Valley Construction in Shanxi Province (SXNGJSKYZX201905); the Science and Technology Innovation Project of Shanxi Agricultural University (2021BQ28).

SUPPLEMENTARY MATERIALS

Supplementary data to this article can be found online at https://doi.org/10.1556/066.2022.00187.

ABBREVIATIONS

WRJW white-ripe jujube wines
FRJW full-red jujube wines
WRJ white-ripe period jujube
FRJ full-red period jujube
TSS total soluble solids
TA titratable acidity
RS reducing sugars
EC ethanol content
TP total phenolics
TT total tannin
PCA principal component analysis
HMA heat map analysis

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