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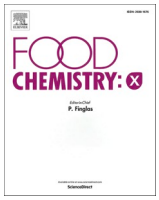

Chemometric analysis illuminates the relationship among browning, polyphenol degradation, Maillard reaction and flavor variation of 5 jujube fruits during air-impingement jet drying

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ABSTRACT

This study was designed to reveal the relationship among browning, polyphenol degradation, Maillard reaction (MR) and flavor variation in jujube fruit (JF) during air-impingement jet drying (AIJD). Five kinds of JFs were dried by AIJD at 60 °C and vacuum freeze drying. Colorimeter and chemometric analysis found that AIJD induced color changes of JF pulp and peel. AIJD also reduced the total polyphenols content and total flavonoids levels in JF. The Fe^{3+} reducing capacity and 2,2′-Azinobis-(3-ethylbenzothiazoline-6-sulphonate) cationic radical scavenging capacity of JF were reduced by 31.6% and 8.2%, respectively. Seven polyphenols were identified in JF, and epicatechin was found related to change of JF pulp color by sparse partial least square (sPLS). sPLS revealed that 3-deoxy glucosone, N-ε-carboxymethyl-L-lysine and 5-hydroxymethylfurfural associated with JF color. sPLS found that MR generated 3-methyl-butanoic acid and cyclobutanone during AIJD of JF. Chemometrics is an effective tool to disclose mechanism of color changes in food.

1. Introduction

Jujube (*Ziziphus Jujuba*) is native to China with a history of >4000 years, and distributes to over 30 countries around the world (Shen et al., 2022). Jujube fruits are highly sought after, by reason of their unique flavor, color, nutritional functions and health benefits about traditional medicine (Gao, Wu, & Wang, 2013). Although jujube fruit is resistant to decay during and after harvest, it only can be stored and protected for several years after well-dried process (Djilali, Nabiev, Gelicus, Benamara, & Allaf, 2016). In China, there are approximately 90% jujube fruits consumed in dried form (Song et al., 2020). Therefore, drying is the most common critical unit operation for jujube fruit. To date, air-impingement jet drying (AIJD), hot-air drying (HAD), vacuum freeze drying (VFD), swell drying, microwave drying, and combined drying are used in dehydration of jujube fruit (Djilali, Nabiev, Gelicus, Benamara, & Allaf, 2016; Wojdyło et al., 2016). Among these technologies, HAD had low efficiency and strong destruction on polyphenol; swell drying led to structural damage; VFD had very high energy consumption and low efficiency; microwave drying could cause local microwave aggregation and carbonization (Li et al., 2020; Djilali, Nabiev, Gelicus, Benamara, & Allaf, 2016). However, AIJD has >10 times higher heat and mass transfer rates than HAD (Deng et al., 2020), and AIJD-dried kiwifruit slices showed better color quality than HAD- and VFD-treated ones (Huang, Li, Shao, Gao, & Yang, 2017). Therefore, AIJD was a promising technology for jujube fruit process.

Drying not only extends storage period of jujube fruit, but also promotes formation of color and flavor quality of jujube fruit (Wojdyło, Figiel, et al., 2016). Particularly, color is the first significant factor that consumers consider when assessing the quality and acceptability of foodstuff before tasting (Aghdam et al., 2020; Lee, Lee, Lee, & Song, 2013). However, color of food changes easily during drying, baking, frying and other processes. Enzymatic oxidation of polyphenol plays an important role in color change of many fruits and vegetables (Huang et al., 2022; Segovia-Bravo, Jarén-Galan, García-García, & Garrido-Fernandez, 2007). Furthermore, non-enzymatic oxidation of polyphenol also influenced obviously on color (Liu et al., 2022). Whether the

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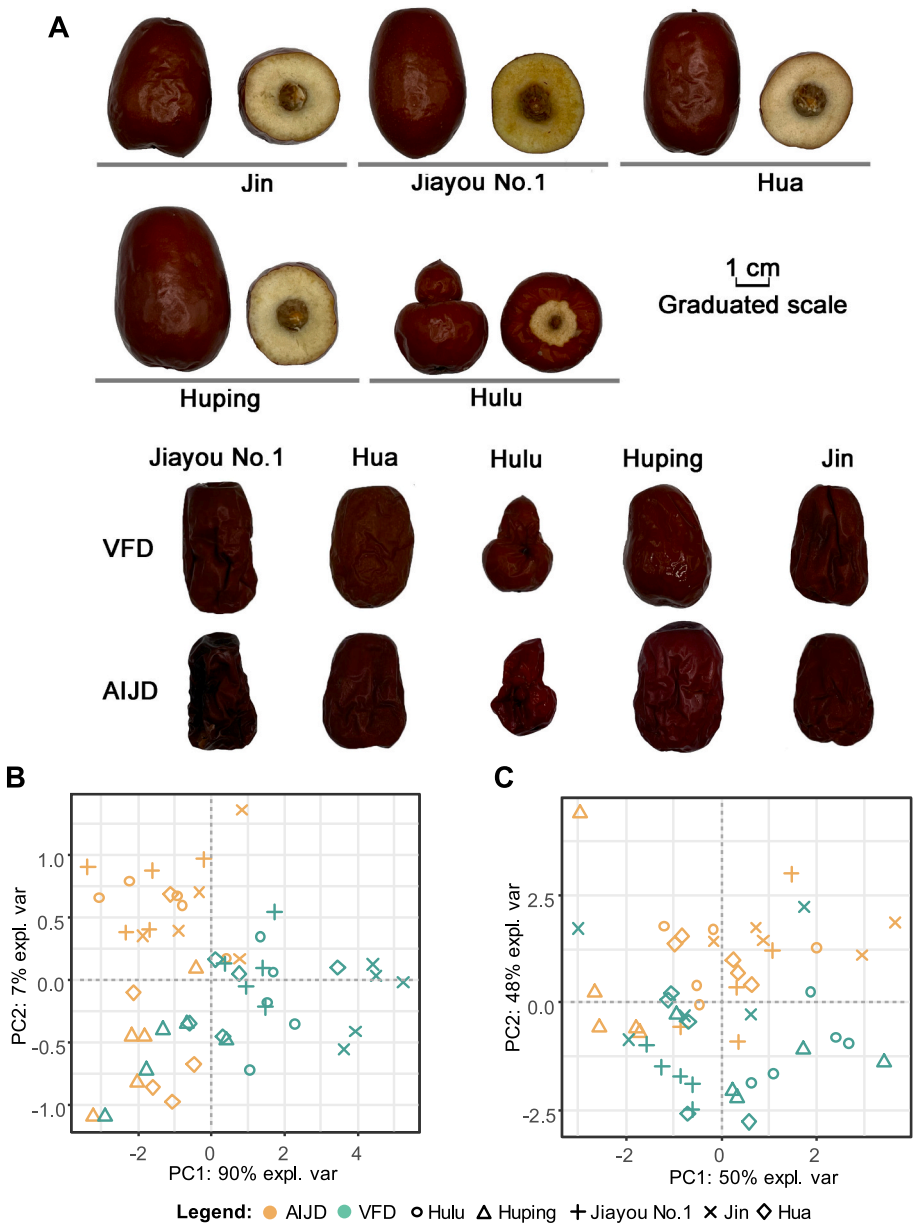

Fig. 1. Effects of drying on colors of 5 jujube fruits. A: the images of fresh, vacuum freeze dried and air-impingement jet dried jujube fruits. Jin, Jiayou No.1, Hua, Huping, and Hulu were the variety names of jujube fruits. B: the multilevel score plot of sparse principal component analysis (sPCA) for peel color of jujube fruits dried by vacuum freeze drying (VFD) and air-impingement jet drying (AIJD). C: the multilevel score plot of sPCA for pulp color of jujube fruits dried by VFD and AIJD.

non-enzymatic or enzymatic oxidation of polyphenol, leads to a decrease in the content of initial polyphenol molecule. Maillard is another famous reaction to cause food browning by condensing carbonyl and amino compound (Bork, Haase, Rohn, & Kanzler, 2022). Maillard reaction (MR) not only contributes to the desirable characteristics of the food color, but also ascribes taste, flavor, and antioxidant properties (Şen & Gökmen, 2022). However, we do not know that whether variations of color and flavor relate to polyphenol oxidation and/or MR of jujube fruit during AIJD or not. Investigating relevant mechanism contributed to accurately control of jujube fruit quality during AIJD.

We note that both polyphenol degradation and MR of food during drying are very complex (Moratalla-López, Lorenzo, Chaouqi, Sánchez, & Alonso, 2019). The related phenomenon is difficult to explain with some traditional and simple chemical analyses. Chemometrics provided multi-statistical tools to extract useful information from complex datasets (Granato et al., 2018). The principal component analysis and partial least squares analysis, two common multivariate tests, have been widely applied in food fraud detection and the effects of process variables on chemical composition of foods (Callao & Ruisánchez, 2018). Therefore, this work employed these statistical tools to illuminate the relationship among browning, polyphenol degradation, MR, and flavor variation of jujube fruit during air-impingement jet drying. This could provide a new strategy to elucidate the mechanisms underlying alterations in food quality during processing through applying chemometrics.

2. Materials and methods

2.1. Materials and reagents

Different varieties of jujube fruits, respectively named Jin, Jiayou No.1, Hua, Huping and Hulu, were collected from fruit market in Shaanxi, China (Fig. 1A). The standard compounds of gallic acid (GA,

pure >98%), rutin (pure >98%), epicatechin (pure >98%), vanillic acid (pure >97%), protocatechuic acid (pure >98%), ferulic acid (pure >98%), phloridzin (pure >98%) and hesperidin (pure >98%) were purchased from Shanghai Yuanye Biotechnology Co. Ltd. (Shanghai, China). Shanghai Yuanye Biotechnology Co. Ltd. (Shanghai, China) also provided the N-ε-carboxymethyl-L-lysine (CML, pure >97%), N-ε-carboxyethyl-L-lysine (CEL, pure >95%), 5-hydroxymethylfurfural (HMF, pure >98%), D(+)-glucose (pure >99.6%) and D-glucosone (G, pure >99.6%). Mixed solution of 17 amino acids (Table S1) and glyoxal solution (GO, 40%, *w*/w) was obtained from Beijing Solarbio Science & Technology Co. Ltd. (Beijing, China). 3-Deoxy glucosone (3DG, pure >75%) and 2,3-butanedione (DMG, pure >97%) were obtained from Merck KGaA (Darmstadt, Germany). HPLC-grade acetonitrile was acquired from EMD Millipore corporation (Burlington, USA). Other chemical reagents used in our work were analytical grade.

2.2. Drying treatments

Jujube fruit (250 g) was dried by air-impingement jet dryer at 60 °C, which has been used in previous study (Wang et al., 2022). The drying process was halted once the water content of the jujube fruits was below 25%, which is the general water content found in most commercially available jujubes. Vacuum freeze drying was applied as the control treatment. Briefly, 250 g of jujube fruit were vacuum freeze-dried for 96 h after freezing in the cold trap (−50 °C) of vacuum freeze dryer (Scientz-10 N, Ningbo Scientz Biotechnology Co., Ltd., Ningbo, China).

2.3. Color measurement

The L*, a* and b* values were measured by chromameter (NR60CP, Shenzhen Sanenchi Technology Co., Ltd., Shenzhen, China). We firstly tested the color parameters of the surface of jujube fruit. Subsequently, we proceeded to slice the jujube fruit using a knife and evaluated the color of the pulp within. All the measurements were repeated 5 times. The chroma (C*) and hue angle (H*) were calculated according to the equations (Pathare, Opara, & Al-Said, 2013) as follows:

$$C^* = \left(a^{*2} + b^{*2}\right)^{0.5} \quad (1)$$

$$H^* = tan^{-1}(b^*/a^*) \quad (2)$$

2.4. Determination of total polyphenols content (TPC) and total flavonoids content (TFC)

Sample (2 g) containing the pulp and peel was homogenized with 10 mL of ethanol and equal water by high-speed homogenizer (FSH-2 A, Changzhou Yuexin Instrument Manufacturing Co. Ltd., Changzhou, China). Then, homogenization was centrifuged at 3500 rpm for 10 min. The supernatant was used to test TPC and TFC according to our previous report with some modifications (Li et al., 2019).

Fifty microliters of sample were mixed with 10 μL of Folin-Ciocalteu reagent. Then, after incubation at 25 °C for 5 min, 100 μL of Na_2CO_3 and 80 μL of water were further added. After another incubation at 25 °C for 10 min, the absorbance was tested at 760 nm. The calibration curve was used to calculate the TPC, and the result was expressed as μg of equivalent GA per gram of dried matter (μg eq. GA/g d. m.).

Ten microliters of sample and equal volume of $NaNO_2$ (5%) were mixed in 96-well plates. The mixture was further mixed with 10 μL of $AlNO_3$ (10%), 100 μL of NaOH (4%), and 60 μL of 60% ethanol. After incubation at 25 °C for 15 min, the absorbance was obtained at 510 nm. The result was showed as μg of equivalent rutin per gram of dried matter (μg eq. rutin/g d. m.).

2.5. Assessing antioxidant activities

The antioxidant activity of our jujube fruit was assessed by reducing Fe^{3+} and scavenging $ABTS^{\cdot+}$, which have been used to assess antioxidant activity of fresh jujube fruit (Choi et al., 2012).

2.5.1. Ferric reducing antioxidant power (FRAP)

FRAP reagent was prepared by mixing 2.5 mL of TPTZ (10 mmol/L), 2.5 mL of $FeCl_3$ (20 mmol/L) and 2.5 mL of acetate buffer (10 mmol/L, pH = 3.6). Then, 100 μL of FRAP reagent was mixed with 50 μL of sample and 100 μL of water. After incubation at 37 °C for 10 min, the absorbance was measured at 593 nm. The FRAP value was expressed as μg equivalent ascorbic acid per gram of dried matter (μg eq. AA/g d. m.).

2.5.2. $ABTS^{\cdot+}$ scavenging activity

The 2,2′-Azinobis-(3-ethylbenzothiazoline-6-sulphonate) (ABTS) solution (7 mmol/L) was left in dark at room temperature for <12 h, after mixed with $K_2S_2O_8$ (2.45 mmol/L). The absorbance value of supernatant was adjusted to 0.70 ± 0.02 (@734 nm) by dilution. After reaction for 10 min, the absorbance of the mixture of sample (100 μL) and $ABTS^{\cdot+}$ reagent (100 μL) was measured at 734 nm. The $ABTS^{\cdot+}$ scavenging activity was calculated as μg equivalent ascorbic acid per gram of dried matter (μg eq. AA/g d. m.).

2.6. Analysis of individual polyphenol compound

The contents of individual polyphenols in jujube fruit were analyzed by our previous method (Li et al., 2019). Five microliters of sample were injected into a C18 column (100 × 2.1 mm i.d., 1.8 μL, Agilent, Waldbronn, Germany) for separation. The column was eluted by a mixed mobile phase that was composed by 0.1% formic acid-water (A) and 0.1% formic acid-methanol (B) at 0.2 mL/min. The gradient program followed that: 0–10 min, 5%–95%B; 10–11 min, 95%–95%B; 11–11.5 min, 95%–5%B; 11.5–15, 5%–5%B. The above procedures were operated on a UHPLC system (1290II, Agilent). Individual polyphenol in extractive was ionized by an electrospray ionization source (ESI) at the negative ion mode. The data were acquired at multiple reaction monitoring. Quantitative parameters of polyphenols were showed in Table S1. The drying gas (N_2) flow velocity and temperature respectively were 10 L/min and 350 °C.

2.7. Determination of glucose, MR products and free amino acids

The MR products were measured by UHPLC-MS/MS according to previous report (Chen et al., 2022). In addition, UHPLC-MS/MS was also used to simultaneously measure the contents of free amino acids. Serine, glycine, aspartic acid, glutamic acid, glucosone, glucose, 3DG and DMG were analyzed in negative ion mode. Alanine, lysine, arginine, histidine, proline, valine, methionine, tyrosine, leucin, threonine, phenylalanine, CEL, CML, GO and HMF were analyzed in positive ion mode. The analytical instrument, column and mobile phases were the same with the analysis of polyphenols in our work (part of 2.6). However, the gradient elution program was different from that. The gradient program both in ESI^+ and ESI^- analyses followed that: 0–8 min, 5%–30%B; 8–8.5 min, 30%–95%B; 8.5–9.5 min, 95%–95%B; 9.5–10, 95%–5%B; 10–15, 5%–5%B. The analysis in ESI^- mode was performed after the analysis in ESI^+ mode. Quantitative parameters of MR products and free amino acids were expressed in Table S1.

2.8. Analysis of volatile compounds

The volatile compounds of jujube fruits were analyzed by gas chromatography–mass spectrometry (8890-5977B, Agilent, California, USA). The analytic strategy was modified from Song and colleagues' report (Song et al., 2020). DB-wax chromatographic column (30 m × 250 μm × 0.25 μm) was used to separate compounds. The carrier gas was highly purified He, and the carrier gas flow rate was 1.0 mL/min without restrictor. The column temperature was kept at 40 °C for 3 min, and increased at 4 °C/min to 140 °C, and then improvedat 6 °C/min to

Table 1
The color parameters of jujube fruits dried by air-impingement jet drying (AIJD) and vacuum freeze drying (VFD).

Part	Drying	Varieties	Color parameters				
			L*	a*	b*	C*	H*
Peel	VFD	Hulu	$34.66^{ab} \pm 2.78$	$19.25^{a} \pm 3.11$	$11.57^{ab} \pm 2.61$	$22.48^{a} \pm 3.98$	$30.72^{b} \pm 2.20$
		Huping	$34.90^{a} \pm 2.49$	$16.64^{bc} \pm 2.10$	$12.14^{a} \pm 3.10$	$20.65^{ab} \pm 3.41$	$35.47^{a} \pm 4.77$
		Jiayou No.1	$31.72^{cde} \pm 1.48$	$15.19^{cd} \pm 2.64$	$9.72^{bc} \pm 2.08$	$18.06^{bc} \pm 3.18$	$32.58^{ab} \pm 3.49$
		Jin	$33.93^{abc} \pm 1.87$	$18.14^{ab} \pm 2.28$	$10.40^{ab} \pm 1.13$	$20.95^{ab} \pm 2.22$	$30.02^{bc} \pm 3.72$
		Hua	$33.08^{abcd} \pm 2.93$	$16.49^{bc} \pm 2.00$	$11.46^{ab} \pm 2.08$	$20.12^{ab} \pm 2.53$	$34.62^{a} \pm 4.04$
	AIJD	Hulu	$32.23^{bcde} \pm 2.52$	$18.75^{a} \pm 3.19$	$10.98^{ab} \pm 2.98$	$21.75^{a} \pm 4.23$	$29.90^{bc} \pm 2.91$
		Huping	$30.10^{e} \pm 1.41$	$14.00^{d} \pm 2.20$	$7.08^{d} \pm 1.23$	$15.69^{c} \pm 2.50$	$26.82^{cd} \pm 1.23$
		Jiayou No.1	$26.63^{f} \pm 1.54$	$9.36^{e} \pm 2.02$	$2.89^{e} \pm 0.98$	$9.80^{d} \pm 2.20$	$16.82^{e} \pm 2.49$
		Jin	$30.89^{de} \pm 2.37$	$14.77^{cd} \pm 2.51$	$7.54^{cd} \pm 2.01$	$16.60^{c} \pm 3.11$	$26.57^{d} \pm 3.16$
		Hua	$30.54^{de} \pm 2.24$	$13.67^{d} \pm 1.65$	$6.09^{d} \pm 1.10$	$14.98^{c} \pm 1.86$	$23.95^{d} \pm 2.59$
Pulp	VFD	Hulu	$58.73^{bcd} \pm 5.60$	$8.33^{d} \pm 1.02$	$20.29^{de} \pm 2.64$	$21.95^{d} \pm 2.60$	$67.47^{d} \pm 3.02$
		Huping	$69.00^{a} \pm 1.43$	$8.59^{d} \pm 0.62$	$24.41^{bc} \pm 0.75$	$25.88^{bc} \pm 0.91$	$70.67^{bc} \pm 0.78$
		Jiayou No.1	$61.21^{bcd} \pm 2.04$	$11.27^{bc} \pm 1.29$	$25.33^{ab} \pm 2.18$	$27.82^{bc} \pm 2.19$	$65.79^{bc} \pm 2.66$
		Jin	$65.37^{ab} \pm 2.82$	$9.60^{cd} \pm 1.48$	$23.98^{bc} \pm 1.62$	$25.85^{bc} \pm 2.04$	$68.41^{bc} \pm 1.88$
		Hua	$55.31^{cde} \pm 4.74$	$8.48^{d} \pm 0.95$	$19.36^{e} \pm 0.97$	$21.16^{d} \pm 0.73$	$66.29^{d} \pm 3.04$
	AIJD	Hulu	$62.31^{abc} \pm 8.50$	$13.48^{a} \pm 2.73$	$27.57^{a} \pm 1.22$	$30.82^{a} \pm 1.67$	$64.35^{a} \pm 4.65$
		Huping	$54.62^{de} \pm 2.88$	$11.18^{bc} \pm 0.88$	$22.89^{bcd} \pm 1.17$	$25.58^{bc} \pm 0.62$	$64.06^{bc} \pm 2.72$
		Jiayou No.1	$48.71^{e} \pm 4.64$	$12.29^{ab} \pm 0.22$	$21.74^{cde} \pm 2.18$	$25.00^{c} \pm 1.93$	$60.20^{c} \pm 2.51$
		Jin	$55.48^{cde} \pm 0.98$	$12.65^{ab} \pm 0.80$	$25.15^{ab} \pm 1.10$	$28.15^{ab} \pm 1.34$	$63.32^{ab} \pm 0.48$
		Hua	$55.93^{cde} \pm 3.94$	$11.38^{abc} \pm 0.86$	$23.80^{bc} \pm 1.30$	$26.41^{bc} \pm 1.34$	$64.38^{bc} \pm 1.74$

C*: chroma. H*: hue angle. $^{a-e}$ Different letters meant statistic difference ($P < 0.05$).

260 °C for 12 min. EI ion source was used in the mass spectrometry analysis. The ion source, inlet and quadrupole temperature were 230, 240 and 150 °C, respectively. The ionization voltage is 70 eV, and the scanning range is 30 ~ 350u. The NIST 17 database was applied to identify compounds.

2.9. Statistical analysis

The results of TPC, TFC and antioxidant activities were expressed as mean ± standard deviation (SD, $n = 3$). Multilevel principal component analysis (PCA) was analyzed by 'mixOmics' package. This R package was also used in the association analysis based on sparse partial least squares (sPLS). In addition, 'corrplot' package was employed to showed the correlation among polyphenols, free amino acids and MR products. The PCA, partial least squares discriminant analysis (PLS-DA) and orthometric PLS-DA (OPLS-DA) were employed to expose the flavors markers changed during AIJD of jujube fruits. Significant difference was assessed by Student *t*-test for two groups and one-way ANOVA Tukey's test for multigroup. *P*-value <0.05 was considered that the difference was significant.

3. Results and discussion

3.1. AIJD induced obvious changes in colors of jujube fruits

Upon the first visual assessment of product quality, color is a critical indicator prior to tasting and to predict its taste before making a decision about whether to purchase and eat (Lee et al., 2013). Both VFD and AIJD resulted in an evident change of the color of jujube fruit peel (Fig. 1A). Moreover, the dried jujube fruits showed a darker maroon color than the fresh ones. This might be that the dryness-induced surface folds were not conducive to light reflection. It is more important that the L* values of AIJD-treated jujube fruit peels were lower than that of VFD-treated ones (Table 1). The L-value of red radish treated with AIJD was found to be 9.56% lower compared to the VFD-treated sample in our previous study (Wang et al., 2022). AIJD-treated Jiayou No.1 peel showed the lowest L* value than the other varieties of jujube fruits. Concurrently, we also found that the peel of AIJD-treated Jiayou No.1 exhibited numerous dark furrows. This result confirmed what we had already suspected. The a* took positive values for reddish colors and negative values for the greenish ones, whereas b* took positive values for yellowish colors and negative values for the bluish ones (Pathare et al., 2013). Although all the a* and b* values of jujube fruits peels were >0, AIJD caused a noteworthy reduction in a* and b* values (Table 1). All the a* values of jujube pulp in our work (8.33–12.65) were close to the a* values of jujube pigment (5.76–8.53, Shen et al., 2021). This also resulted in a decrease of C* and H* values of peels because they were calculated according to a* and b* values. C* represented the quantitative attribute of colorfulness, and could be applied to assess the degree of difference of a hue in comparison to a grey color with the same lightness (Pathare et al., 2013). Thus, the colorfulness of jujube fruit peel was reduced by AIJD. Furthermore, AIJD also weakened the red hue of jujube fruit peel. However, AIJD did not result in a deterioration of the color quality in the pulp of all jujube fruits (Table 1). The AIJD-treated Hulu and Hua pulps showed higher a*, b* and C* values than the VFD-treated ones. However, AIJD induced decreases in H* values of VFD-treated Hulu and Hua pulps.

Although the above analysis found some influences of AIJD on the color of jujube peel and pulp, it could not reveal the change of color at the aggregate level. Therefore, the L*, a*, b*, C*, and H* values of jujube fruit peel and pulp were further subjected to sPCA analysis in order to explore any underlying similarities and patterns among samples when groupings were not clear (Granato et al., 2018). As shown in Fig. 1B, the principal component (PC) 1 explained 89% of variates, and distinguished the peel colors between AIJD-treated and VFD-treated jujube fruits. However, the number of AIJD-treated Huping were close to that of VFD-treated ones, suggesting that there was no obvious difference between their peel colors (Fig. 1B). Moreover, a more evident distinction was found in the sPCA score plot for pulp colors of jujube fruit dried by AIJD and VFD (Fig. 1C). This suggested that AIJD caused a stronger change in the pulp color than in the peel color of jujube fruits. The PC1 showed 50% of explained variance, and availably distinguished the AIJD- and VFD-treated Huping, Jiayou No.1 and Hulu (Fig. 1C). In general, there was a difference in the effects of the same drying method on the color of different jujube varieties.

3.2. Colors changes were related to AIJD-caused degradations of polyphenols

The polyphenol compounds presented in jujube fruits also served as crucial pigments, exerting an influence on the coloration of them (Shen et al., 2021). Therefore, we hypothesized that the AIJD-induced alteration in the color of jujube fruit was associated with the degradation of polyphenol compounds. In line with previous study (0.37–59.50 mg GA

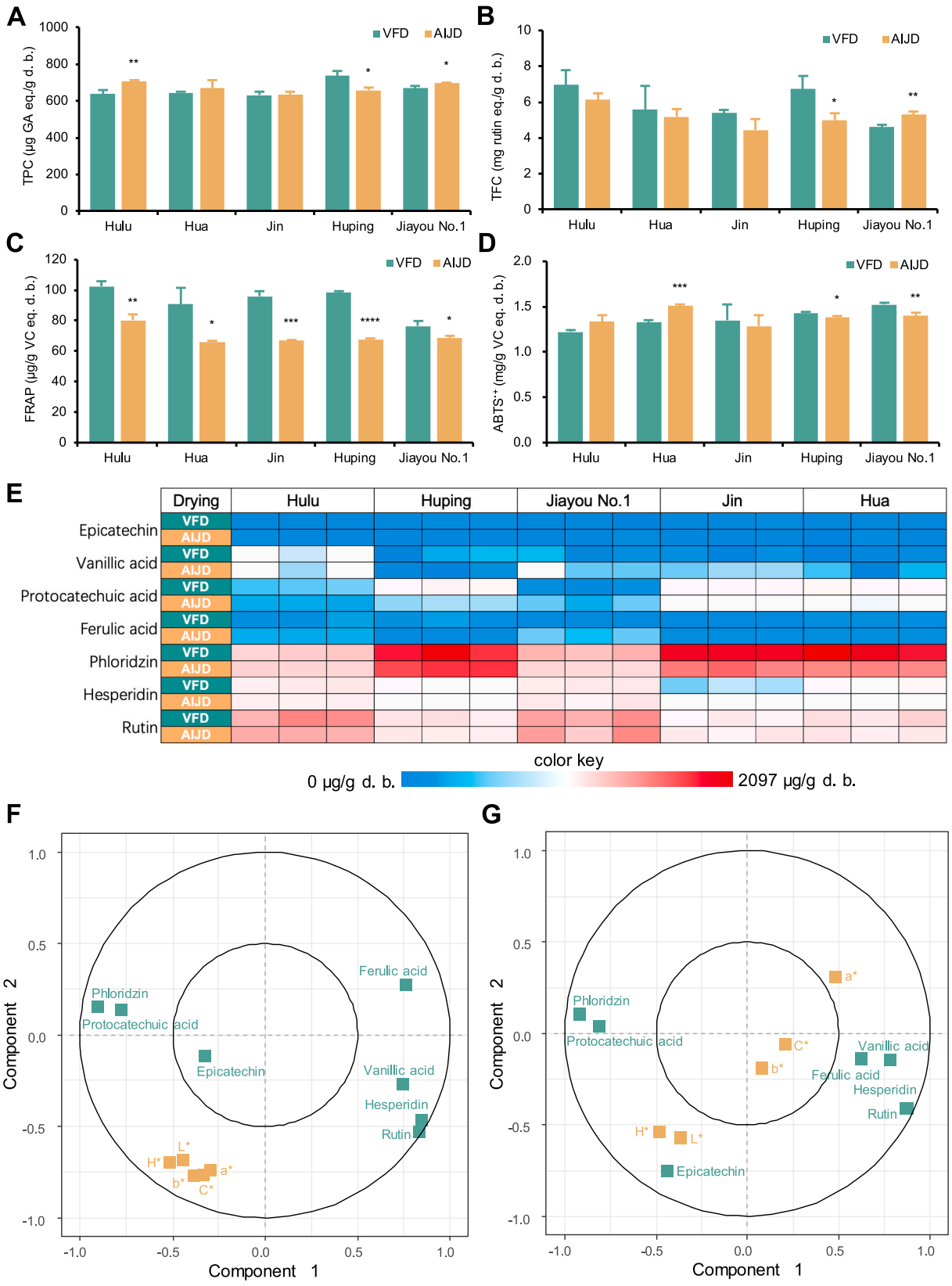

Fig. 2. Contents of polyphenols and antioxidant activities in 5 jujube fruits dried by VFD and AIJD. A: total polyphenol content (TPC). B: total flavonoid content (TFC). C: ferric ion reducing antioxidant power (FRAP). D: scavenging activity of 2,2′-Azinobis-(3-ethylbenzothiazoline-6-sulphonate) cationic radical ($ABTS^{\cdot+}$). E: contents of individual polyphenol compounds. F: correlation circle plot (CCP) from sparse partial least square (sPLS) analysis for peel color and polyphenol contents. G: CCP from sPLS analysis for pulp color and polyphenol contents. $^{*}P < 0.05$, $^{**}P < 0.01$, $^{***}P < 0.001$ and $^{***}P < 0.0001$.

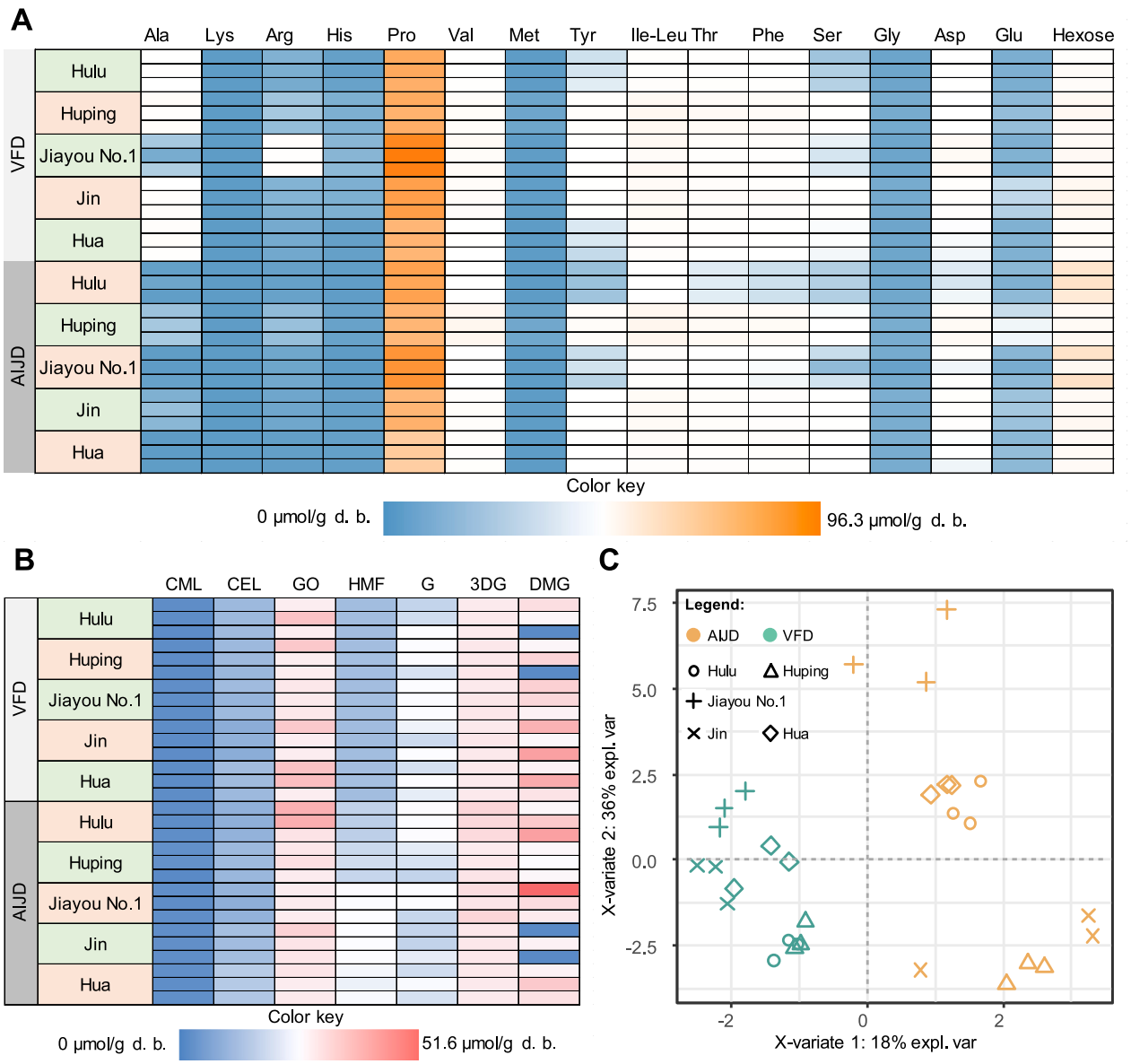

Fig. 3. AIJD caused changes in free amino acids (AA), hexose and Maillard reaction products (MRP) of jujube fruits. A: the contents of AA and hexose. B: the MRP contents of jujube fruits. C: the score plot of sPLS-DA for AA, hexose and MRP of jujube fruits dried by VFD and AIJD. Aspartic acid: Asp. Threonine: Thr. Serine: Ser. Glutamic acid: Glu. Proline: Pro. Glycine: Gly. Alanine: Ala. Cystine: Cys2. Valine: Val. Methionine: Met. Isoleucine: Ile. Leucin: Leu. Tyrosine: Tyr. Phenylalanine: Phe. Lysine: Lys. Histidine: His. Arginine: Arg. D-glucosone: G. Glyoxal solution: GO. 5-Hydroxymethylfurfural: HMF. 3-Deoxy glucosone: 3DG. N-ε-carboxymethyl-L-lysine: CML. N-ε-carboxyethyl-L-lysine: CEL. 2,3-butanedione: DMG.

eq./g d. b., Djilali, Nabiev, Gelicus, Benamara, & Allaf, 2017), we found that the TPC in jujube fruits were 637.39–736.79 μg GA eq./g d. b. (Fig. 2A). Additionally, AIJD significantly reduced the TPC and TFC of Huping, while increased their levels in Jiayou No.1 (Fig. 2A, B). Despite increasing the TPC of Hulu, AIJD caused a slight decrease in the TFC of it. The TPC and TFC in AIJD-treated Hua and Jin showed no significant difference compared to those in VFD-treated Hua and Jin, respectively. Our previous work also found that the different dry methods caused different changes of TPC and TFC in *Psidium guajava* fruits (Tan et al., 2020). The thermal degradation of polyphenols, particularly the flavonoid glucosides, was identified as the main factor contributing to the reduction in TPC and TFC during the drying process. (Karaaslan et al., 2014). However, the heating-based drying does not always cause polyphenol degradation either (Nayak, Liu, & Tang, 2017). The contents of hesperetin, rutin, ferulic acid and p-coumaric acid in the peel of *Citrus reticulata* Blanco, CV. Hongjv were elevated by hot air drying (Li et al., 2020). This may be because drying induced releases of extractable polyphenols with a low molecular weight from non-extractable polyphenol components and polymeric polyphenols (Domínguez-Rodriguze, Marina, & Plaza, 2017). Previous studies found that jujube fruits polyphenols showed strong antioxidant activities to scavenge $ABTS^{\cdot+}$ and reduce Fe^{3+} (Wojdyło, Carbonell-Barrachina, Legua, & Hernández, 2016). Moreover, AIJD also significantly decreased the FRAP of all jujube fruits by 10.6%–31.6% (Fig. 2C). Although the AIJD resulted in 3.1% and 8.2% decreases in $ABTS^{\cdot+}$ scavenging activities of Huping and Jiayou No.1 respectively, it also increased 13.6% of $ABTS^{\cdot+}$ scavenging activity in Hua (Fig. 2D). The alteration patterns among TPC, TFC, FRAP and $ABTS^{\cdot+}$ induced by AIJD do not align precisely. This may be attributed to the presence of different individual polyphenols in these fruits (Tan, Lan, Chen, Zhong, & Li, 2023). Thus, we tested the contents of individual polyphenols in jujube fruits dried by AIJD and VFD. We found epicatechin, vanillic acid, protocatechuic acid, ferulic acid, phloridzin, hesperidin and rutin in jujube fruits (Fig. 2E). Shen and colleagues also found the protocatechuic acid, epicatechin, rutin,

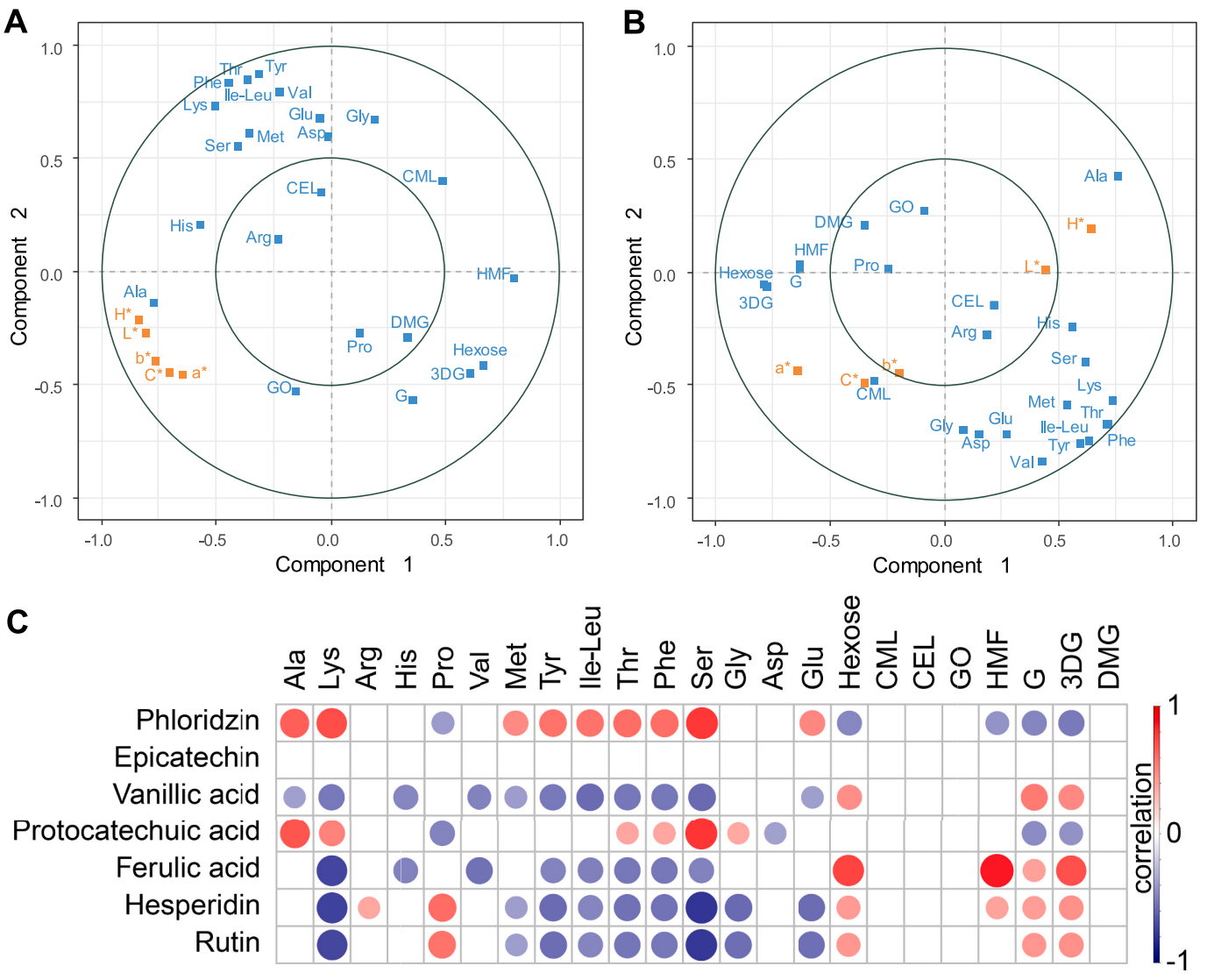

Fig. 4. Correlations between peel color, AA, hexose and MRP of jujube fruits (A). Correlations between pulp color, AA, hexose and MRP of jujube fruits (B). Correlations between polyphenols, AA, hexose and MRP of jujube fruits (C).

vanillin, and ferulic acid in jujube peel (Shen et al., 2022). Additionally, we also observed a similarity between the individual polyphenol levels in the dried jujube fruits (0–0.21 g/100 g d. b., Fig. 2E) and the fresh samples (0–22.67 g/100 g d. b., Choi et al., 2012). AIJD mainly decreased the contents of epicatechin, protocatechuic acid and phloridzin. However, the contents of vanillic acid and ferulic acid exhibited primary increases in jujube fruits during AIJD.

To investigate the relationship between AIJD-induced color change and variation of polyphenol content in jujube fruits, we conducted the sPLS analysis, a correlation-based method that provides variable selection in two-type data sets (Lê Cao, Martin, Robert-Granié, & Besse, 2009). sPLS distinguished the AIJD- and VFD-dried jujube fruits based on the colors or polyphenol contents or their combination, except the Hulu (Fig. 1S). Correlation circle plot highlighted the contributing variables that *co*-explain the covariance between the colors and polyphenol contents (Fig. 2F, G). The L*, a*, b*, C* and H* values of the jujube fruit peels were closely clustered together in the lower-left quadrant of the correlation circle with a coefficient of '1' (the excircle), while being nearly orthogonal to most polyphenols (Fig. 2F). This result indicated that there were strong inter-correlations among L*, a*, b*, C* and H* value of peel, while the associations with polyphenols were weaker. Different from the peel, the H* of pulp presented strong positive correlation with L* and negative correlation with a* (Fig. 2G). Relevantly, the epicatechin exhibited a significant positive correlation with the L* value of pulp, and a negative correlation with the a* value of pulp. These results suggested that the alteration of jujube fruit pulp color induced by AIJD stronglyrelated to the decrease of epicatechin content within the jujube fruits. Previous reports by Su et al. (2022) clearly showed that enzymatic oxidation of catechin, an isomer of epicatechin, led to the enhancement of yellow and red tones. This phenomenon was observed in close proximity to the alteration of jujube fruit pulp color induced by AIJD. Therefore, we speculated that AIJD-induced reduction of epicatechin content in jujube fruit was mainly due to enzymatic oxidation rather than thermal degradation.

3.3. *Colors changes associated with AIJD-caused Maillard reaction*

MR, a complex series of sequential and parallel nonenzymatic reactions, could lead to the browning of food through the production of pigmented compounds (Vistoli et al., 2013). The MR readily occurs when reducing sugars react with compounds containing a free amino group, such as amino acids (Şen & Gökmen, 2022). Therefore, this study investigated the contents of free amino acids and hexose, as depicted in Fig. 3A. Proline in VFD-dried Jiayou No.1 showed the highest content

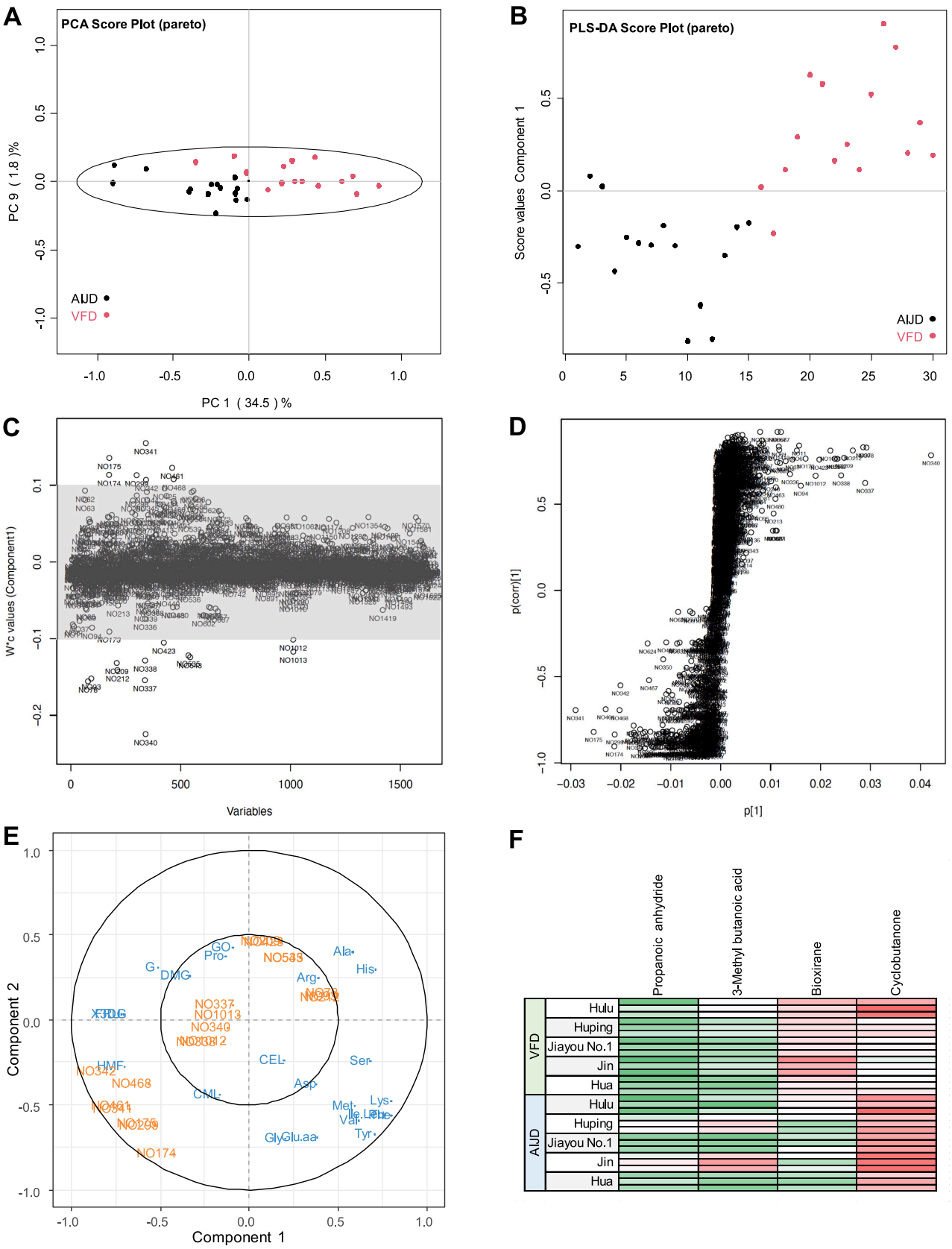

Fig. 5. Multivariate analysis for volatile compounds in jujube fruits. A: score plot of PCA. B: score plot of PLS-DA. C: loading plot of PLS-DA. D: s-plot of orthorhombic PLS-DA (OPLS-DA). E: CCP of sPLS for MRP and potential markers (volatile compounds). F: contents of the key volatile compounds.

(96.3 μmol/g d. b.) than other amino acids (Fig. 3A). Choi and colleagues also found that proline (138.5 μmol/g d. b.) was the maximum amino acid in mature Korean Boen-daechu jujube fruit (Choi et al., 2012). However, proline, alanine, arginine, histidine and serine in most of jujube fruits were decreased by AIJD. Additionally, AIJD also caused the reduction of hexose contents in Huping and Jin (Fig. 3A). Conversely, the MR products, CML, HMF, CEL and 3DG, presented higher levels in jujube fruits dried by AIJD than that in control (Fig. 3B). Furthermore, AIJD-treated most jujube fruits have lower G, GO and DMG contents than the VFD-treated ones (Fig. 3B). It is well known that MR could be divided into early, advance, and final stages (Şen & Gökmen, 2022). The 3DG, GO, G and DMG were formed in the advance stage by depredating Amadori product (AP) and Heyns product (HP), which were produced in the early stage (Hollnagel & Kroh, 1998). CML and CEL, two markers of formation of advanced glycation end products (AGEs), were produced by oxidating AP and HP, and reacting GO with amino group (Srey et al., 2010). HMF also formed in the advanced stage through dehydrating 3DG, or directly degrading from sucrose in dry systems (Perez Locas & Yaylayan, 2008). Accordingly, our results (Fig. 3A, B) indicated that AIJD caused strong MR in jujube fruits.

Although sPCA did not effectively distinguish the AIJD- and VFD-treated jujube fruits according to amine acids and MR products (Fig. S2A), sPLS-DA clearly distinguished them by the first PC explaining 18% of variate (Fig. 3C). sPLS-DA as the most popular discriminant analysis can understand which variables carry the class separating information, in other words, it confirms whether, or not, an object can be associated with a targeted class of interest (Granato et al., 2018). Alanine (Ala), histidine (His), glycine (Gly), 3DG, CML, hexose, arginine (Arg), HMF, proline (Pro) and G in jujube fruits showed the top 10 of loading values on PC 1 of the sPLS-DA, suggesting these compounds might mainly change during AIJD (Fig. S2B). These aforesaid intermediates formed during the advanced stage of MR underwent further condensation and polymerization in the final stage to formed melanoidins which are brown nitrogenous compounds (Namiki, 1988). Next, we utilized sPLS to further reveal the relationship between colors and MR products (Fig. S2C—F). The color parameters of jujube fruit peel presented positive correlation with GO, Ala and His, and negative correlation with CML, HMF and Gly (Fig. 4A). Additionally, positive correlations were also observed between the H* value of pulp and Ala, as well as the a*, C*, and b* values of pulp with CML (Fig. 4B). The H* value of pulp showed strong negative correlation with 3DG, hexose, G and HMF, but a* value of pulp demonstrated an association with Ala (Fig. 4B). These results demonstrated that MR had a more significant impact on pulp color than peel color of jujube fruits. It could be also found that hexose possessed robust positive correlation with MR products, including 3DG, G and HMF (Fig. 4A, B). This was in line with previous findings (Bork et al., 2022). Moreover, sugars can easily be converted to intermediates such as hydroxymethyl furfural (HMF), and then reacted with phenolic compounds to form colored substances in acidic environment (Liu, Chang, & Wu, 2003). The sPLS analysis revealed a significant positive correlation between HMF and ferulic acid as well as hesperidin, while a significant negative correlation was observed with phloridzin (Fig. S3, Fig. 4C). In addition, ferulic acid and hesperidin also positively related with HMF. According to these findings, it could be postulated that the formation of HMF in jujube fruits might potentially arise from intricate reactions involving ferulic acid, hesperidin, and hexose, consequently leading to alterations in fruit coloration.

3.4. Relationship between Maillard reaction and flavors

The Maillard reaction plays a pivotal role in the formation of flavor (Sun et al., 2023). We obtained the matrix contained 1625 × 30 volatile compounds from GC–MS data through peak alignment and integration. This matrix was subsequently subjected to PCA analysis, which dominant patterns present within samples and variables (Granato et al., 2018). As expected, unsupervised PCA distinguished the VFD-treated and AIJD-treated jujube fruits, indicating that there were significantly difference in their flavors (Fig. 5A). However, the supervised analyses were more widely applied to reveal the variables that contribute to the class separating information (Callao & Ruisánchez, 2018). Thus, PLS-DA and OPLS-DA were used to further monitor the characteristics that could discriminate the VFD- and AIJD-dried jujube fruits. As can be seen in Fig. 5B, PLS-DA successfully discriminated the flavor components in VFD- and AIJD-dried jujube using only one PC. A variate might be as a marker for the discrimination that should far away from the origin (0,0) in the loading plot of PLS-DA and s-plot of OPLS-DA (Li et al., 2019). The cross-validation identified 19 potential variates (Fig. 5C, D). The variates were further selected by exhibiting robust positive correlation with Maillard reaction products by sPLS analysis (Fig. 5E). Subsequently, these variates were further characterized by NIST database (Fig. 5E). As a results, propanoic anhydride, 3-methyl butanoic acid, bioxirane, and cyclobutanone were related to the MR induced by AIJD in jujube fruits (Fig. 5F). Among these components, 3-methyl butanoic acid accounts for 32.8% of the total volatile compounds in Hawaiian green coffee beans (*Coffea arabica* L.) (Lee & Shibamoto, 2002). Cyclobutanone had been identified as a main flavor compound in *Enteromorpha prolifera* (Xu, Miao, Zhao, Cao, & Su, 2013). AIJD caused decrease of 3-methyl butanoic acid in jujube fruits (Fig. 5F). Although the cyclobutanone level in Jiayou No.1, Jin and Hua were also reduced by AIJD treatment, the content of Hulu and Huping were elevated after AIJD treatment (Fig. 5F). This information could be utilized to improve the drying process of jujube fruits. Although propanoic anhydride and bioxirane are not considered as flavors in food, our results could also indicate that chemometrics is a tool to reveal the relationship between the Maillard reaction and flavor formation.

4. Conclusion

Color change could be attributed to polyphenols degradations and Maillard reaction during air-impingement jet drying of jujube fruit. Maillard reaction caused by AIJD in jujube fruit also promoted the generation of 3-methyl-butanoic acid and cyclobutanone, which were two potential flavor substances. These new findings suggested that chemometrics was an effective tool to expose mechanism of color change in food during processing. However, our study mainly utilized correlation analysis. It was well known that strong association was not causality. Therefore, new experiment should be designed to further verify the chemometric result.

CRediT authorship contribution statement

Wenfeng Li: Writing – original draft, Software, Methodology, Investigation, Formal analysis, Data curation. **Chan Liang:** Investigation. **Fangtian Bao:** Investigation. **Tingting Zhang:** Investigation. **Yanru Cheng:** Resources. **Wanjie Zhang:** Writing – review & editing. **Yalong Lu:** Writing – review & editing, Supervision, Project administration, Funding acquisition, Conceptualization.

Declaration of competing interest

The authors declare that they have no competing financial interests.

Data availability

Data will be made available on request.

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Kong) for proof reading of the manuscript.

Abbreviations

amino acids	AA
2,2′-Azinobis-(3-ethylbenzothiazoline-6-sulphonate) cationic radical	$ABTS^{\cdot+}$
advanced glycation end products	AGEs
air-impingement jet drying	AIJD
alanine	Ala
Amadori product	AP
arginine	Arg
aspartic acid	Asp
chroma	C*
N-ε-carboxymethyl-L-lysine	CML
N-ε-carboxyethyl-L-lysine	CEL
correlation circle plot	CCP
cystine	Cys2
3-deoxy glucosone	3DG
2,3-butanedione	DMG
electrospray ionization	ESI
ferric ion reducing antioxidant power	FRAP
D-glucosone	G
glyoxal solution	GO
glycine	Gly
glutamic acid	Glu
hue angle	H*
Heyns product	HP
histidine	His
5-hydroxymethylfurfural	HMF
isoleucine	Ile
leucin	Leu
lysine	Lys
Maillard reaction	MR
Maillard reaction products	MRP
methionine	Met
orthorhombic partial least squares discriminant analysis	OPLS-DA
principal component	PC
phenylalanine	Phe
partial least squares discriminant analysis	PLS-DA
proline	Pro
serine	Ser
sparse partial least square	sPLS
sparse principal component analysis	sPCA
total polyphenol content	TPC
total flavonoid content	TFC
threonine	Thr
tyrosine	Tyr
ultra-high performance liquid chromatography in tandem with triple quadrupole mass spectrometry	UHPLC-MS/MS
valine	Val
vacuum freeze drying	VFD

Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.fochx.2024.101425.

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