

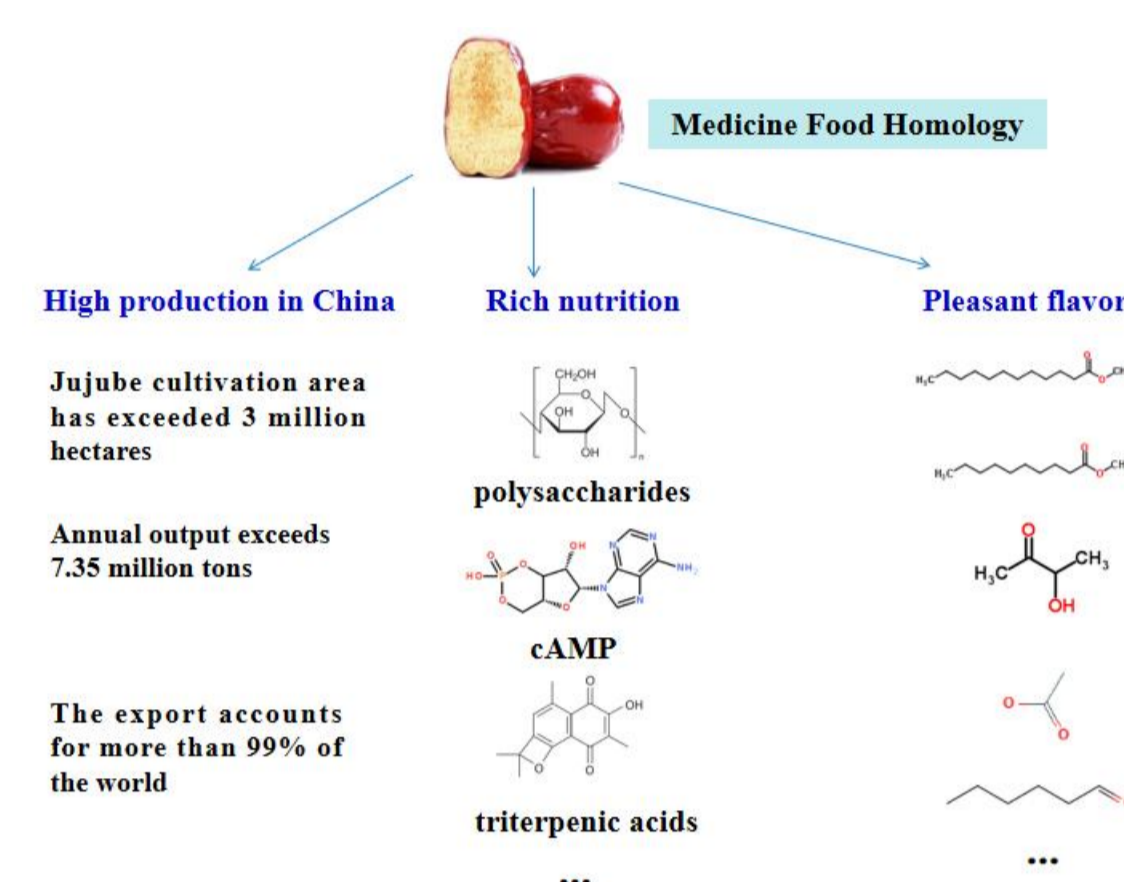
Novel insight into the evolution of volatile compounds during dynamic freeze-drying of *Ziziphus jujuba* cv. Huizao based on GC-MS combined with multivariate data analysis

Min Gou^{1,2}, Qinqin Chen¹, Xinye Wu¹, Gege Liu¹, Marie-Laure Fauconnier², Jinfeng Bi¹

(1. Institute of Food Science and Technology, Chinese Academy of Agricultural Sciences, Beijing, China; 2. Laboratory of Chemistry of Natural Molecules, Gembloux Agro-Bio Tech, University of Liège, Gembloux, Belgium)

INTRODUCTION

1. Jujube (*Zizyphus jujuba* Mill.) is a plant of the family Rhamnaceae and originated in China with a long history of more than 4000 years.



2. With the development of freeze drying (FD), freeze-dried red jujube has become a popular product, with better nutrition, appearance, color and aroma.

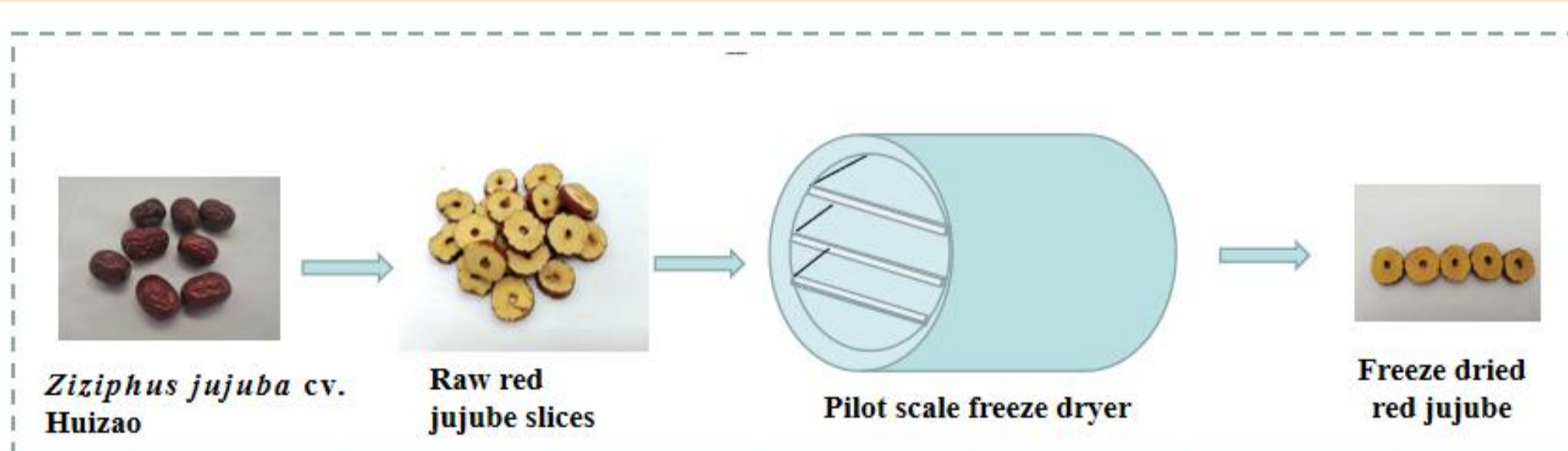
3. However, the causes of aroma differences between raw and freeze-dried jujube and the aroma formation pathway during FD are still unclear.

OBJECTIVES

- The changes of aroma, reducing sugars, fatty acid and free amino acids, and related enzyme activities in the pilot scale freeze drying process of red jujube will be investigated.
- To explore the correlation between aroma and aroma precursors and enzyme activities through the Mantel test and network analysis.
- To provide novel insights into the aroma evolution in dynamic FD of red jujube, as well as guidance for future research including optimization of the freeze dried process to improve the aroma profile of red jujube.

METHODS

Sample preparation



Freeze drying (FD) treatment

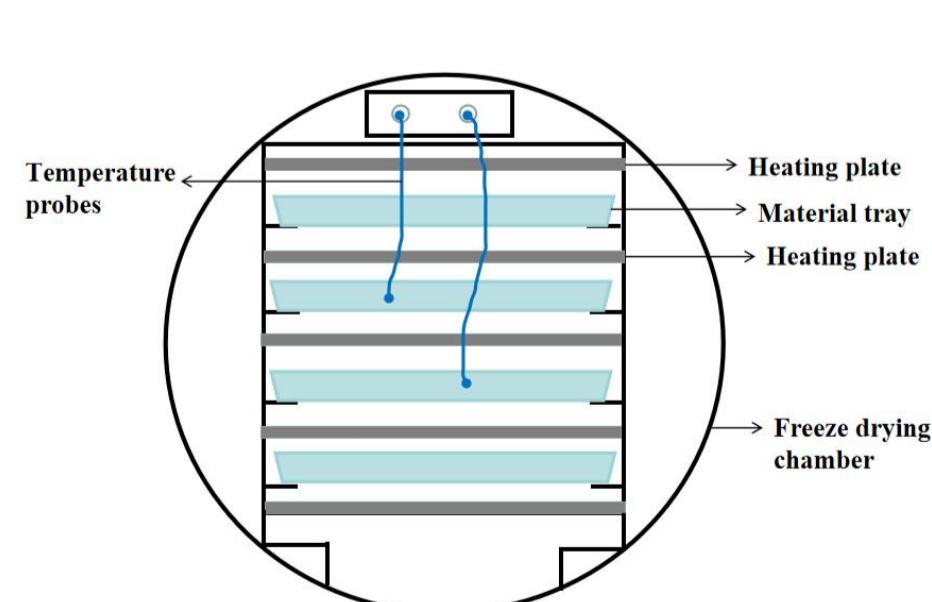


Fig.1 The diagram of drying chamber in pilot scale freeze dryer.

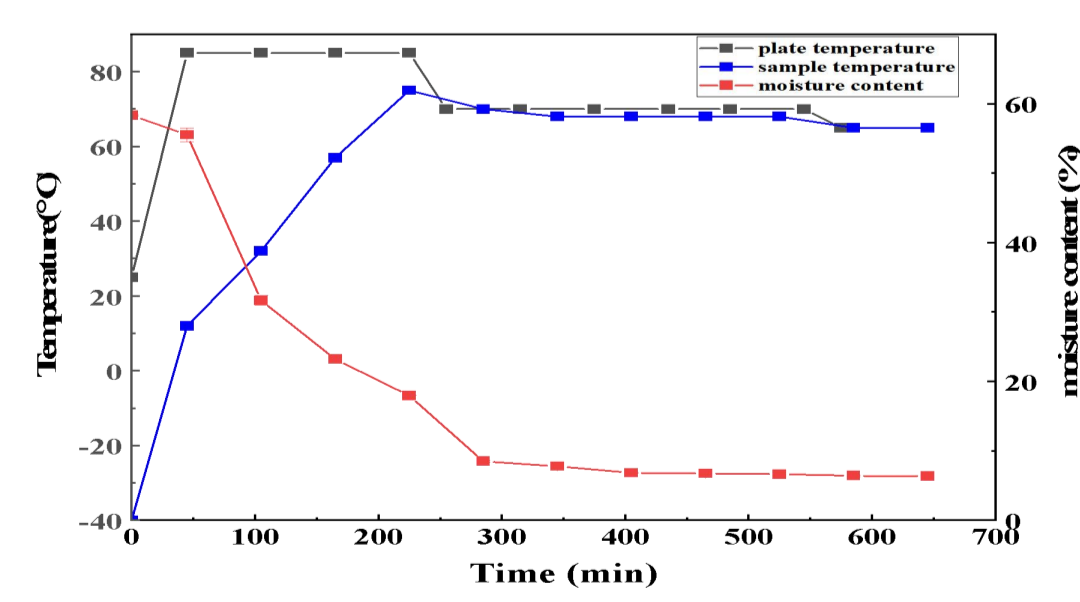


Fig.2 The changes of plate temperature, sample temperature and moisture content of red jujube during freeze drying processing.

RESULTS

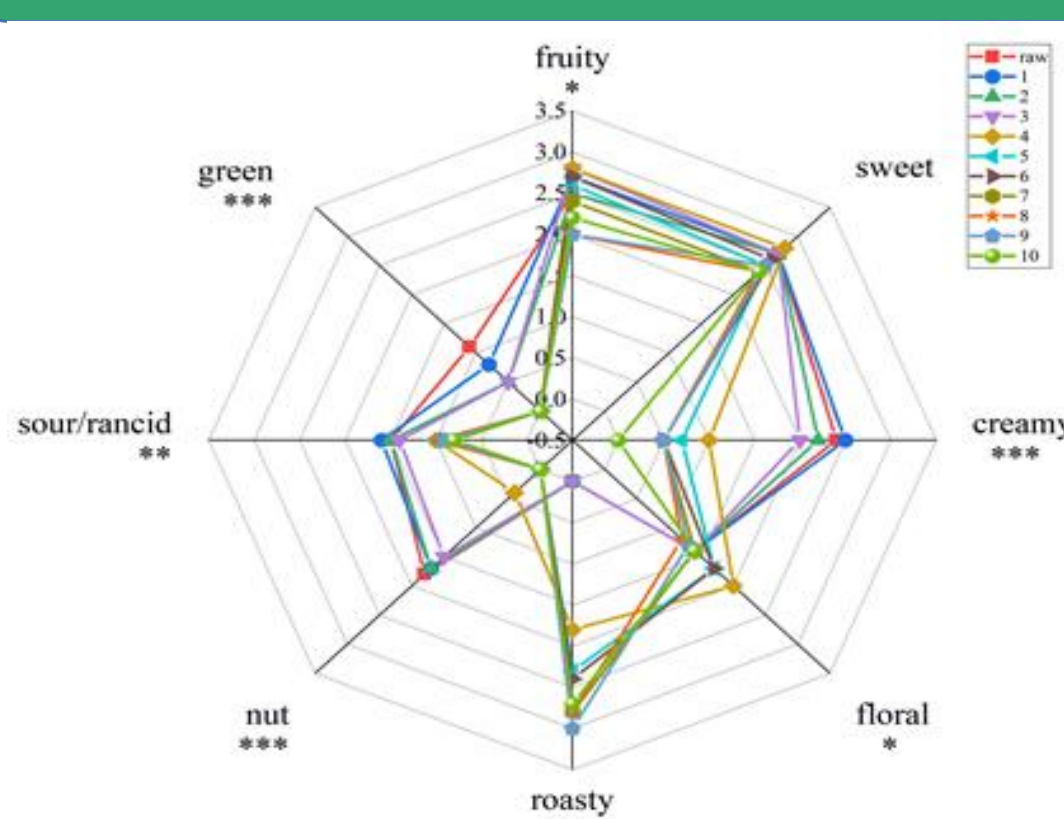


Fig.3 The changes in aroma profiles (a) (***, very highly significant ($p \leq 0.001$); **, highly significant ($p \leq 0.01$); and *, significant ($p \leq 0.05$))

RESULTS

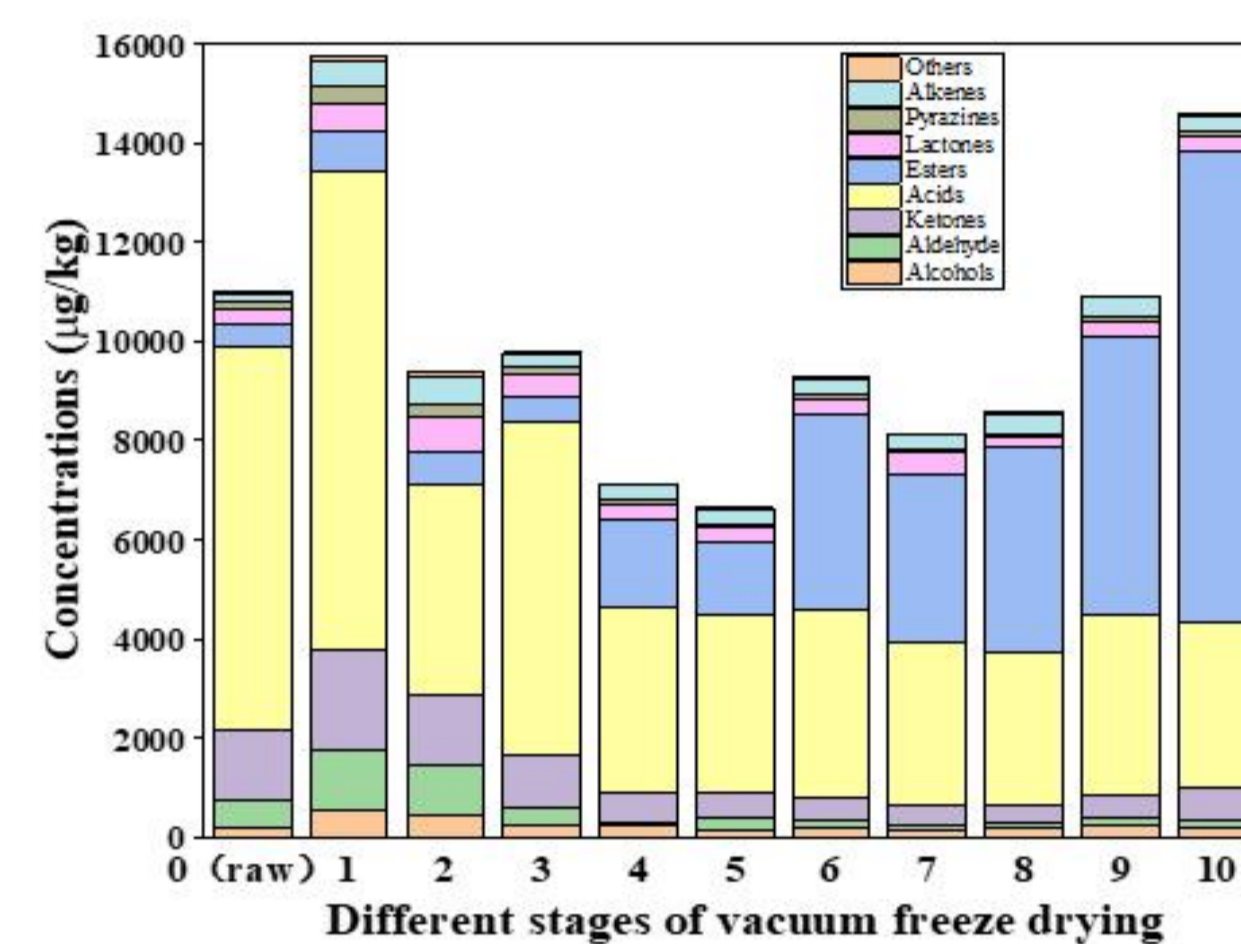


Fig.4 The changes in contents of different group of aroma compounds

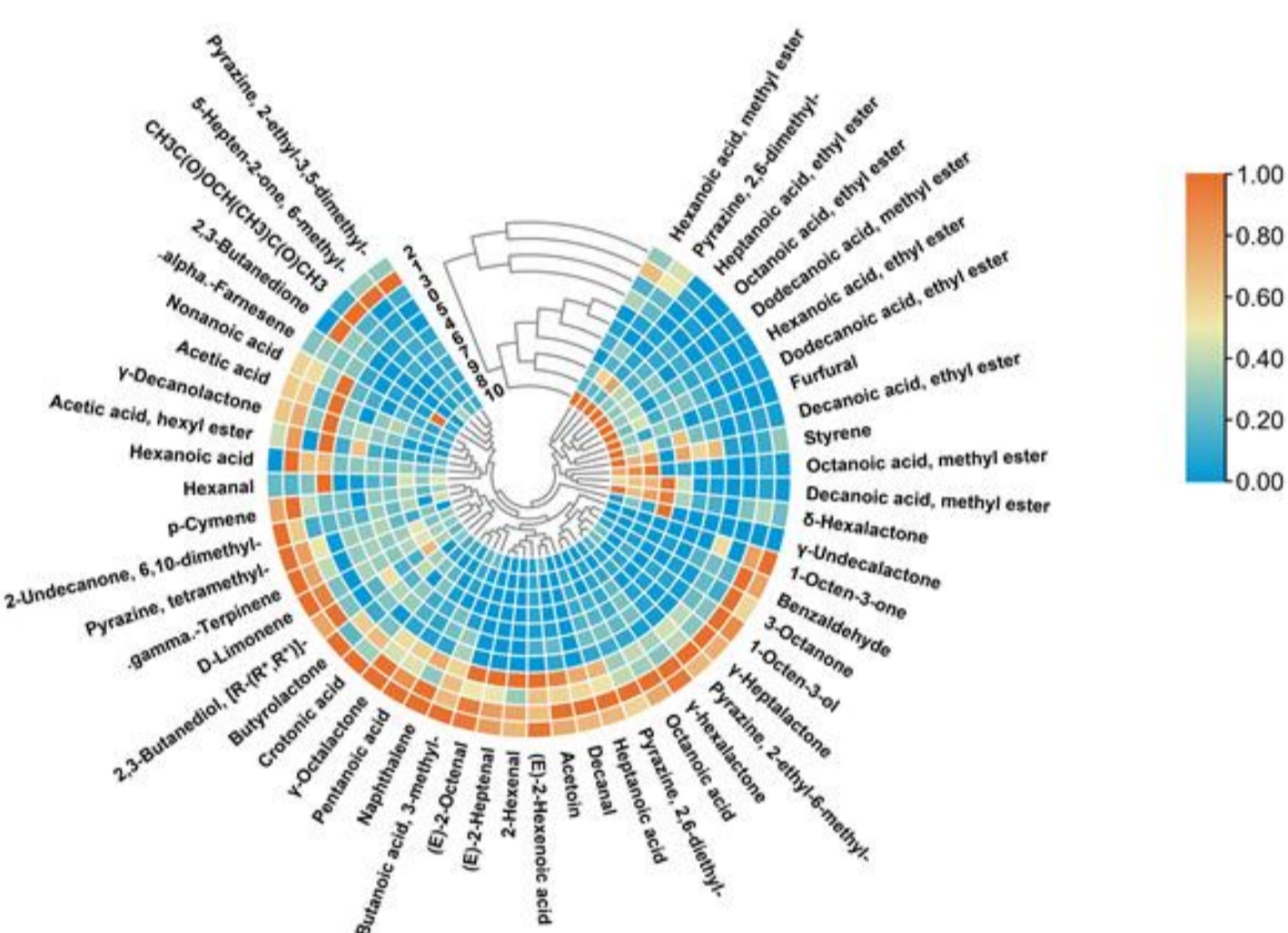


Fig.5 The clustering aroma compounds content heatmap (c) in red jujube during different freeze drying stages

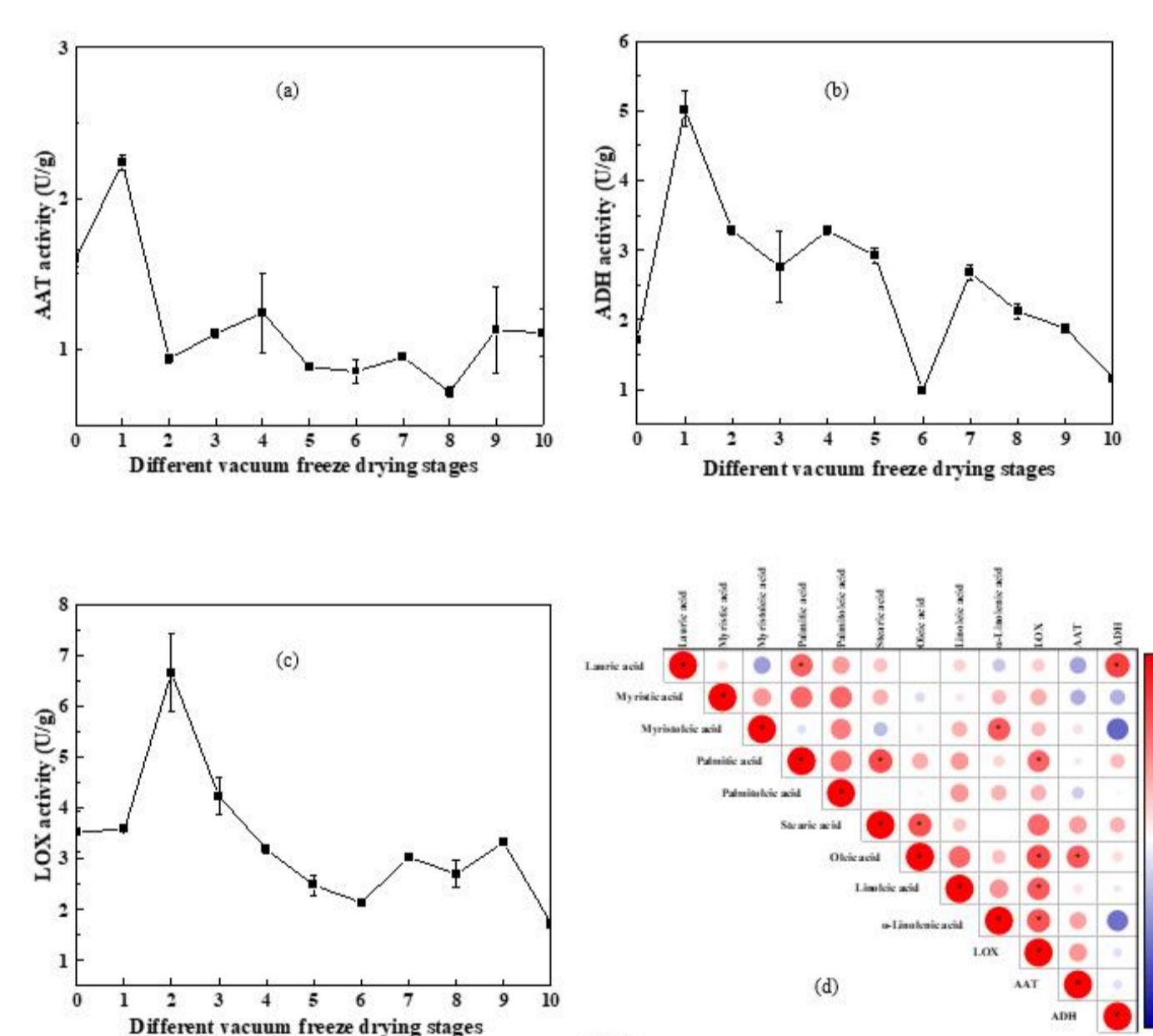


Fig.6 The enzyme activity changes of lipoxygenase (LOX) (a), alcohol dehydrogenase (ADH) (b) and alcohol acyltransferase (AAT) (c) during the different freeze drying stages, and the correlation between enzyme activities and fatty acids (d)

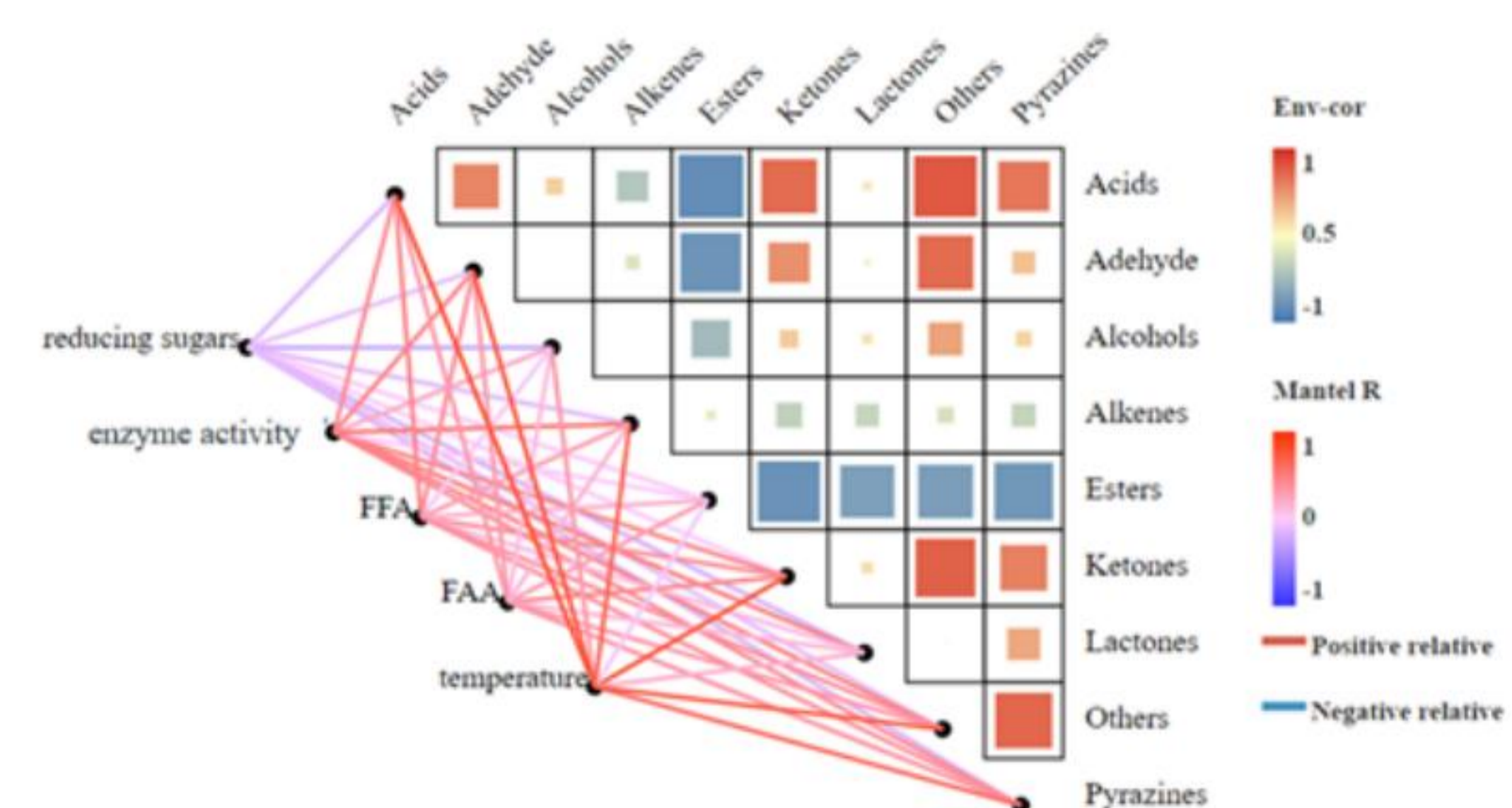


Fig.7 Correlation analysis between classes of volatile compounds and precursors, enzyme activities and temperature by Mantel test. The upper right diagram showing the Spearman correlation of different classes of aroma compounds. A color gradient denotes the Spearman's correlation coefficients. The bottom left graph shows the Mantel test between effect parameters (reducing sugar, enzyme activities, FFA, FAA, and temperature) and different classes of aroma compounds mentioned above. FFA, free fatty acids; FAA, free amino acids

RESULTS

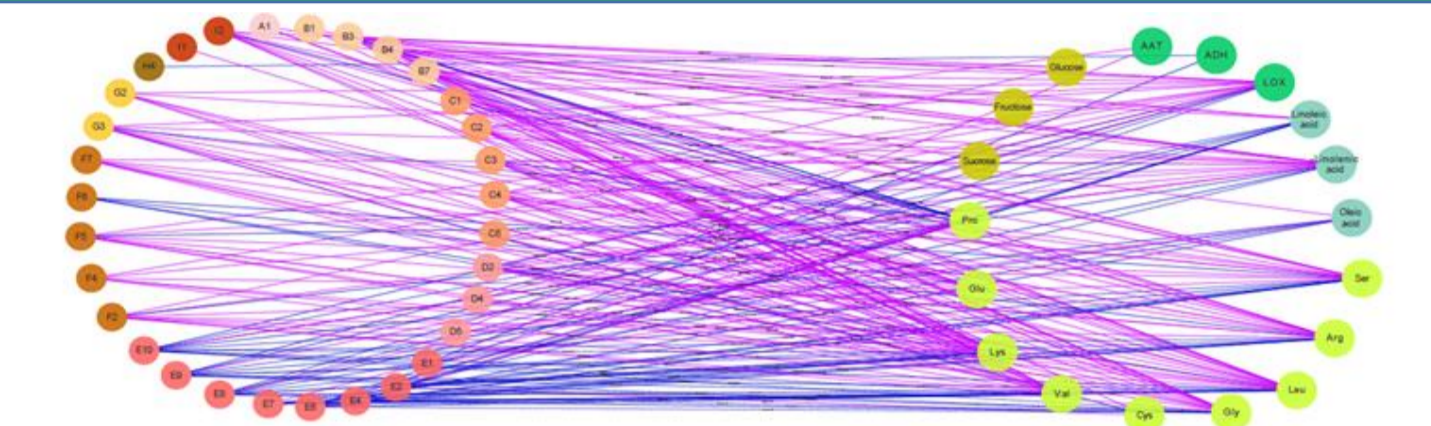


Fig.8 Spearman correlation networks showing relationships between aroma-active compounds (OAV>1) and flavor precursors, enzyme activities in red jujube during freeze drying stages. The left-hand circle represents the aroma-active compounds, and the right-hand circle represents the main flavor precursors and enzyme activities in the red jujube during freeze drying. The purple and blue lines respectively represent the positive and negative correlation between the aroma compounds and flavor precursors, enzyme activities. And correlation coefficients between them were calculated using values from all samples. Only significant correlations ($|r| > 0.6$, $p < 0.05$) are indicated, and line thickness represents the correlation coefficients of interactions.

CONCLUSIONS

- A total of 30 aroma-active compounds of 53 aroma compounds were detected in all red jujube samples during FD processing, and the aroma content increased 32.7% after FD, ketones content was significantly decreased by 54.11%, resulted in the loss of creamy note in freeze-dried jujube (FDJ).
- Through the network analysis, serine, glycine, proline, valine, cysteine, arginine, glutamic acid, lysine and leucine had the significant correlation with pyrazines, dominated the roasty note of FDJ. Linoleic acid, α -linolenic acid and oleic acid with lipoxygenase had important effects on the increase of esters, contributed fruity and sweet notes of FDJ.
- Besides, through the Mantel test, the influence degree of factors on the formation of FDJ aroma was ranked as temperature > enzyme activity > fatty acids > amino acids.
- The multi-stage and variable-temperature procedure of FD enhanced lipid pyrolysis reaction and non-enzymatic reaction efficiency, which significantly improved the aroma of red jujube.

REFERENCES

- Amanpour, A., Vandamme, J., Polat, S., Kelebek, H., Van Durme, J., & Selli, S. (2019). Non-thermal plasma effects on the lipoxygenase enzyme activity, aroma and phenolic profiles of olive oil. *Innovative Food Science and Emerging Technologies*, 54(April), 123-131. <https://doi.org/10.1016/j.ifset.2019.04.004>
- Deng, S., Cui, H., Hayat, K., Zhai, Y., Zhang, Q., Zhang, X., & Ho, C. T. (2022). Comparison of pyrazines formation in methionine/glucose and corresponding Amadori rearrangement product model. *Food Chemistry*, 382(February), 132500. <https://doi.org/10.1016/j.foodchem.2022.132500>
- Gou, M., Chen, Q., Qiao, Y., Li, J., Long, J., Wu, X., Zhang, J., Fauconnier, M., Jin, X., Lyu, J., & Bi, J. (2022). Comprehensive investigation on free and glycosidically bound volatile compounds in *Ziziphus jujuba* cv. Huizao. *Journal of Food Composition and Analysis*, 112(March), 104665. <https://doi.org/10.1016/j.jfca.2022.104665>
- Li, W., Li, R., Chen, W., Feng, J., Wu, D., Zhang, Z., Zhang, J., & Yang, Y. (2022). The anabolism of sulphur aroma volatiles responds to enzymatic and non-enzymatic reactions during the drying process of shitake mushrooms. *Food Chemistry*, 371(July 2021), 131123. <https://doi.org/10.1016/j.foodchem.2021.131123>
- Schwab, W., Davidovich-Rikanati, R., & Lewinsohn, E. (2008). Biosynthesis of plant-derived flavor compounds. In *Plant Journal* (Vol. 54, Issue 4, pp. 712-732). <https://doi.org/10.1111/j.1365-313X.2008.03446.x>

ACKNOWLEDGEMENTS

The funding support of the Financial Fund of Agricultural Science and Technology Innovation Program, Institute of Food Science and Technology, Chinese Academy of Agricultural Sciences (CAAS-ASTIP-2022-IFST).