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Detection of biosynthetic citric acid and sugar addition in Italian tomato passata using liquid chromatography-chemical oxidation-isotope ratio mass spectrometry

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Summary: A novel LC-co-IRMS method was developed to detect adulteration in Italian tomato passata. By measuring compound-specific $\delta^{13}\text{C}\%$ values of citric acid, glucose and fructose, the diagnostic ratios RCA/G ($\delta^{13}\text{C}$ Citric acid / $\delta^{13}\text{C}$ Glucose), and RCA/F ($\delta^{13}\text{C}$ Citric acid / $\delta^{13}\text{C}$ Fructose) successfully identified exogenous sugars and biosynthetic citric acid, offering a robust tool against food fraud.

Keywords: Liquid chromatograph-chemical oxidation-isotope ratio mass spectrometry; Tomato passata; Compound specific analysis.

Introduction

According to EU legislation, the addition of citric acid (E330) to tomato sauces is allowed and must be declared in the label, while the addition of exogenous sugars is forbidden. Since tomato is a C3 plant, the addition of C4-derived organic compounds can be detected through isotopic analysis. To the best of our knowledge, no isotopic references for the citric acid naturally present in tomatoes are currently available.

Experimental

A comprehensive dataset including 100 authentic Italian tomato sauces sampled by the ICQRF (Central Inspectorate for Fraud Repression and Quality Protection of Agri-Food Products and Foodstuffs) and 23 commercial market samples was analysed using LC-co-IRMS to simultaneously measure the $\delta^{13}\text{C}\%$ of citric acid, glucose, and fructose. The analytical method was validated by determining the within-day and between-days repeatability on a reference sample. To assess the minimum detectable addition of exogenous citric acid, a selected authentic sample was spiked with increasing amounts of C4-derived citric acid (ranging from 0% to 100%). Furthermore, isotopic difference ratios RCA/G ($\delta^{13}\text{C}$ Citric acid / $\delta^{13}\text{C}$ Glucose), RCA/F ($\delta^{13}\text{C}$ Citric acid / $\delta^{13}\text{C}$ Fructose) and RG/F ($\delta^{13}\text{C}$ Glucose / $\delta^{13}\text{C}$ Fructose) were calculated as diagnostic parameters.

Results

The LC-co-IRMS method allowed for the successful establishment of the official $\delta^{13}\text{C}$ (‰) reference ranges for authentic Italian tomato sauces based on the ICQRF reference dataset. Method validation confirmed high analytical precision across repetitive within-day and between-days measurements. The spiking experiments with C4-derived citric acid (0– 100%) demonstrated high sensitivity, enabling the identification of minimal exogenous additions. The calculated isotopic ratios RCA/G , RCA/F and RG/F provided robust supportive criteria, allowing clear discrimination between authentic products and commercial samples subjected to fraudulent additions of exogenous sugars or industrial citric acid.

Conclusions

In this study, a new approach through LC-co-IRMS was adopted, and the $\delta^{13}\text{C}$ of citric acid, glucose and fructose were measured to detect additions of biosynthetic citric acid or C4 exogenous sugars in Italian tomato passata. The method also demonstrated good repeatability, with intra-day and inter-days standard deviations below 1.1 ‰ for citric acid, glucose and fructose.