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Gas chromatography and hyphenated techniques in assessment of Baijiu spirits authenticity and quality

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Abstract

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In this study, different analytical methods were developed to assess the authenticity and quality of Baijiu - traditional Chinese spirit which is characterized by unique technology - saccharification and spontaneous fermentation under the solid state, consortia of microorganisms, the absence of rectification which results in the richness of volatile compounds. Gas chromatography equipped with a flame ionization detector (GC-FID) and two different polarity columns (CPWax-57CB and DB-624) was used for developing a method to quantify 62 compounds at one time by using direct injection. The limit of detection (LOD) was < 1 mg/L for all compounds and regression coefficient of determination (R^2) ≥ 0.999 for 61 compounds. However, data obtained by this method was insufficient to classify the regional origin and aroma type of the total of 38 Baijiu samples. Headspace solid-phase microextraction coupled with comprehensive two-dimensional gas chromatography and time-of-flight mass spectrometry (HS-SPME-GC×GC-TOFMS) equipped with different column setups was further investigated for 65 Baijiu samples aroma types and regional origins assessment. The orthogonality and repeatability were compared for the conventional and reversed column setups. The same 65 sample set was used to develop methods using headspace-solid phase microextraction–mass spectrometry without chromatographic separation (HS-SPME-MS) and gas chromatography based electronic nose (GC-E-Nose) to elaborate the feasibility of rapid analysis in Baijiu authenticity determination. The HS-SPME-GC×GC-TOFMS equipped with conventional setup achieved best accuracy in the Baijiu aroma type assignment by using orthogonal partial least square discriminant analysis (OPLS-DA) model compared with HS-SPME-MS and GC-E-Nose. Both HS-SPME-GC×GC-TOFMS and HS-SPME-MS obtained 100% accuracy for classification of Baijiu from Sichuan, Heilongjiang and Jiangsu regions within Strong aroma type. To trace the botanical origin of spirits, 5 Baijiu samples labelled with single ingredient (sorghum) and 40 raw spirits produced from corn, potato, wheat and rye were included in the study of the comparison between HS-SPME-MS and isotope ratio mass spectrometry (IRMS). IRMS can differentiate the C3 plants spirits from C4 plants

while cannot clearly classify the botanical origin within C3 plants. The successful botanical origin assessment for all spirits was obtained by HS-SPME-MS coupled with OPLS-DA.

Keywords: Baijiu, aroma, origin, classification, GC, GC×GC, SPME-MS

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