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Original Article

Assessment of the key aroma compounds in rose-based products

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ABSTRACT

In this study, headspace solid phase microextraction–gas chromatography-mass spectrometry and GC-olfactometry were used to analyze the key aroma compounds in three types of rose-based products, including low-temperature extracts (LTEs), high-temperature extracts (HTEs), and rose drinks (RDs). In combination with the Guadagni theory, it was confirmed that the key aroma components of LTE were β-phenyl ethyl alcohol, citronellol, geraniol, and eugenol. The main aroma compounds in HTE were β-phenyl ethyl alcohol, citronellol, geraniol, eugenol, linalool, and rose oxide. The four key aroma compounds in RDs were β-phenyl ethyl alcohol, eugenol, geraniol, and linalool.

1. Introduction

Rose is a precious traditional Chinese medicinal material, which is also an important raw material in the perfume and food industries [9]. The demand for rose and its products has diversified in the international and domestic markets, resulting in it becoming a research focal point to determine the key compounds of the rose aroma found in rose-based products [4–6]. Currently, using headspace solid phase microextraction–gas chromatography-mass spectrometry (HS-SPME–GC-MS) technology, research into the aroma compounds of rose and its products is mainly focused on quantifying the aroma constituents of the rose flower bud, petal, the rose flower recovered at different periods, and rose essential oil. In flower buds, γ-muurolene, α-himachalene, and α-pinene are the major constituents. Meanwhile, β-citronellol, citronellol acetate, phenethyl alcohol, geraniol are the major constituents in fresh flower at the early opening stage, and at the full opening stage, β-citronellol citronellol acetate, phenethyl alcohol, geranyl acetate, geraniol, phenethyl acetate, nerol, *n*-hexyl acetate and α-myrcene, and alcohols are the major constituents [4,10,11]. In rose oil, there are 20 kinds of compounds whose content exceeds 1%, including 22.606% phenethyl alcohol, 12.015% citronellol, 6.772% geraniol, 6.194% eugenol, and 2.329% neroli alcohol

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[5,6,12]. As the thresholds of the compounds differ, the impact of the aroma at the highest content may not be the most significant; hence, determining the main aroma of rose-based products is crucial.

GC-olfactometry is an instrument used to detect odors online and simultaneously record the results. HS-SPME–GC-MS combined with GC-olfactometry technology has been widely used in determining the aromas of balsamic vinegar, distillate spirits, apple, cheese, and other foods [3,7,8,13]. However, analyzing the aroma of rose and its products using GC-MS combined with GC-olfactometry has not been previously reported.

HS-SPME–GC-MS combined with GC-olfactometry was used in this study to assess the odors of a serially diluted low-temperature extract (LTE), high-temperature extract (HTE), and rose drinks (RDs), to determine the main aroma substances of the three rose-based products. Meanwhile, according to the theory that a higher content and lower threshold confers a larger contribution to the sample, the key aromas of the samples were determined, providing a more reliable scientific basis for the quality control of rose-based products.

2. Methods

2.1. Materials

LTE refers to a distilled extract produced during the rose drying process, a colorless and clear liquid, produced on June 10, 2013. HTE is a distilled extract produced during the rose add-water distillation process, a colorless and clear liquid produced on May 3, 2013. RD refers to the rose drink named *jiuduomeigui*, a red and clear liquid, which is commercially available; its production process is not clear, but it was produced on June 21, 2013. These experimental materials were provided by NI'S International Rose Industry Co. Ltd. (Zaoyang, Hubei Province, China).

All aroma standards were purchased from Sigma–Aldrich (St. Louis, MO, USA) and were of the highest purity available: rose oxide, linalool, α-terpineol, citronellol, nerol, geraniol, β-phenyl ethyl alcohol, and eugenol. Sodium chloride AR was obtained from Beijing Chemical Plant (Beijing, China).

2.2. Instruments and equipment

GC-MS (Agilent Technologies, 7890A, 5975C), SPME handle (Supelco, USA), PDMS fibers (Supelco), and Olfactometry (ODP3; Gerstel, Germany) were used during the tests. Helium was used as the carrier gas, with a constant flow rate of 1.0 mL/min. The GC system was equipped with a split–splitless injection port at 230°C. The splitless mode was used to inject the fiber and used a 5-minute desorption time. An Elite Wax Etr column [30 m, 0.25 mm (ID), 0.25 μmdf], supplied by Perkin-Elmer, was used to separate the volatiles. After injection, the column temperature was held at 50°C for 2 minutes and increased to 80°C at 10°C/min. Subsequently, the temperature was increased to 115°C at 10°C/min, followed by an increase to 240°C at 3°C/min, with a final holding time of 5 minutes. The temperature of the transfer line to the mass spectrometer was set at 240°C. The mass spectrometer was operated using the electron impact ionization mode (70 eV) at 230°C.

2.3. Experimental

2.3.1. Sample preparation

Depending on which of the rose-based products was to be tested, the samples were diluted to varying ratios. First, 2 mL of the LTE stock solution, as well as two-fold diluted, fold-fold diluted, and 10-fold diluted solutions were taken to perform the HS-SPME. Then, 2 mL of each of the HTE stock solutions, as well as four-fold diluted and 10-fold diluted solutions were taken to perform the HS-SPME. Next, 2 mL of each of the RD, two-fold diluted, and four-fold diluted solutions were used perform the HS-SPME, and the aroma components were extracted. The HS-SPME procedure was carried out as described by Su et al [9].

2.3.2. Sniffing and detection

The method for sniffing the odors using GC-olfactometry involved a four-way flow divider at the end of the GC column, which split the sample (split ratio, 2:1) to the mass detector and olfactometer. While sampling, five trained personnel sat at the outlet of the olfactometer, and recorded and described the odors they smelled. According to the aroma intensity, the aroma compounds were divided into four grades—1, 2, 3, and 4—from weak to strong. A sniffing aromagram could then be formed according to the grades. Each sample was assessed three times by each judge. The sniffing time for each run was 50 minutes.

Identification was achieved from comparisons of mass spectra obtained from the samples with those present in the NIST05 MS Library database. Retention index was calculated using the software system named AMDIS. Identification was considered tentative when it was based only on the mass spectra data. Compounds that were identified by confirming their mass spectrogram to that of authentic chemicals, purchased from commercial sources, were considered to be positively identified.

The method for determining the key compounds involved increasing the dilution ratio, resulting in a gradual reduction of the aroma intensity and species. The detected aroma compounds were noted; the compounds of a higher frequency, detected using GC-olfactometry, were regarded as the key aromas. At the same time, the content of the aroma compounds was detected by GC-MS, and each compound was given an odor activity value (OAV), which is the ratio of the concentration and the threshold. Hence, the key aroma compounds in rose-based products were finally determined by HS-SPME–GC-MS and GC-olfactometry.

2.3.3. Quantification

The standard addition method was used in this analysis. The working solutions of aroma standards at five different levels of concentration were added to the samples prior to the HS-SPME. Quantitative analysis was finished by the calibration curve acquired. Stock standard solutions of the analytes were prepared in ethanol and stored at 4°C under refrigeration. Furthermore, working solutions were prepared by dilution of the stock solutions with ethanol in the appropriate quantities.