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A comprehensive analysis of aroma compounds and microstructure changes in brown rice during roasting process



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ABSTRACT

Roasting of brown rice not only forms the aroma compounds but also leads to a complete alteration of the microstructure, but the change mechanism during the roasting process is still unclear. Sensory evaluation, electronic nose, gas chromatography-tandem mass spectrometry and scanning electron microscope were combined to investigate the changes of aroma compounds and microstructure during roasting process. Eleven categories of volatile compounds were detected, and the roasting process made an increase in the content of heterocycle compounds and a decrease in the types and content of hydrocarbons and benzene derivatives. Furans and pyrazines made a great contribution to the aroma quality. Furfural, 5-methyl furfural, 2,5-dimethylpyrazine, 2-methylpyrazine, 2-ethyl-6-methylpyrazine and 2-ethyl-3,5-dimethylpyrazine were the main flavor compounds in the roasted brown rice. The microstructure change of brown rice during roasting was found to have a major impact on the volatilization of aroma compounds.

1. Introduction

Brown rice contains high amounts of water-soluble bioactive components such as dietary fiber, γ -aminobutyric-acid, polyphenols, trace minerals and vitamins, thus labeling as a kind of popular whole grain (Cho & Lim, 2016). Consumption of diets rich in whole grains has been associated with lower risk of chronic diseases including cardiovascular disease (Kelly et al., 2017). However, the taste of brown rice hardly meet requirements of consumers farthest when treat as a staple food. Therefore, it is urgent to use processing techniques to develop brown rice products. Germination, fermentation, baking and roasting has been increasingly employed to enhance and change desired food qualities, such as color, texture, appearance, taste and flavor.

Aroma is one of the most important sensory qualities of cereals, stimulating the nasal olfactory receptors by the volatile aroma components directly affect the evaluation of quality and consumer acceptability (Rawat et al., 2007). Generally, roasting is a powerful processing technology to increase amounts of aroma compounds in cereals, which recently has been widely used in whole grain rice products (Youn & Chung, 2012). Under roasting, brown rice could form a unique flavor and increase the favorite degree of consumers. Identification of volatile

compounds present in different kinds of rice has been the theme of many researches (Jezussek, Juliano, & Schieberle, 2002). Up to now, over 300 volatiles compounds have been identified in all kinds of rice, including ketone, aldehyde, ester, alcohol, heterocycle compounds (D. S. Yang, Lee, Jeong, Kim, & Kays, 2007). Moreover, studies have shown that aroma consists of diverse volatile compounds at different concentrations essentially (Zhu et al., 2016). Roasting with different time and temperature could greatly influence the chemical compositions (Ramli, Hassan, Said, Samsudin, & Idris, 2006). In addition, roastinginduced changes in microstructure have a major impact on the final product quality. Studies showed that the loss of aroma compounds and the subsequent change in flavor profile are probably related to the extent of exposure of inner surface (Schenker, Handschin, Frey, Perren, & Escher, 2010). Soaking such as the brewing process of tea could accelerate the dissolution of water-soluble nutrients, the release of aroma and the changes in microstructure (Kraujalytė, Pelvan, & Alasalvar, 2016). Therefore, in this study, roasting was chosen as the processing method on brown rice and brew into tea to investigate the changes of aroma compounds and microstructures.

Sensory evaluation is a common technology used to differentiate flavor. However, it could be easily swayed by physiological,

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psychological and environmental factors (Bhattacharyya et al., 2007). Electronic nose (E-nose) consists of a series of electronic chemical sensors with partial specificity (Cheng, Qin, Guo, Hu, & Wu, 2013) and gas chromatography-mass spectrometry (GC-MS) had combined in the determination of the volatile aroma components (Abdulra'uf & Tan, 2015). GC-MS/MS has higher accuracy and sensitivity than GC-MS and could perform quantitative analysis (Mayr et al., 2015). Pyrazines, furans and aldehydes were found as the main compounds in roasted tartary buckwheat tea (P. Qin, Ma, Wu, Shan, & Ren, 2011). Heterocyclic compounds have been identified as the major volatile compounds in Houjicha which roasted from 170 to 200 °C (Mizukami, Sawai, & Yamaguchi, 2008). Although there were some researches on the determination of aroma compounds, few studies were focused on the change regulation during roasting. Simultaneously, although we know that roasting would change the microstructure of brown rice which in turn influence the release of the aroma, the relationship between the aroma compounds, microstructure and flavor quality was not clear. Therefore, it is necessary to understand the influence of roasting on aroma compounds and microstructures in brown rice through a comprehensive analysis.

In present study, sensory evaluation, E-nose and GC–MS/MS analysis were employed to investigate the effect of roasting on aroma compounds and characteristic compounds, in order to statistically obtain the optimal condition of brown rice roasting, including temperature and time. Moreover, scanning electron microscope (SEM) was performed to observe the microstructure change of brown rice, and thus further clarify the potential relationship between the structural changes, the formation and the release of aroma compounds under roasting.

2. Materials and methods

2.1. Preparation of roasted brown rice

Brown rice (Japonica rice) was purchased from Yanzhifang Food Company Limited (Anhui, China), and washed with clean water for three times to remove impurities, including dust and chaff. Then the brown rice was dried through a drying oven at 40 °C. After drying, the moisture content of brown rice was about 12%. Approximately 50 g of brown rice in whole grain was then roasted in an electric roaster (Meidi WK2102, China) with constant stirring up at different temperature (60 °C, 80 °C, 100 °C, 120 °C and 140 °C). The roasting time was set at 15 min, 25 min, 35 min, 45 min and 60 min. The roasted brown rice was sealed after cooling to room temperature.

2.2. Sensory analysis of aroma

Sensory analysis was performed according to the green tea sensory analysis method, with a slight modification (Han et al., 2016). Brown rice (approximately 5 g of whole grain) with different roasting conditions was infused with 30 mL of boiled water for 5 min. The infused roasted brown rice was removed and tea infusions were transferred to glasses. Six trained panelists were required to evaluate the sensory characteristics of aroma of roasted brown rice. The aroma quality of samples with a score less than 100 point was evaluated. The training sessions of panelists were carried on the national standard GB/T 23776–2018. The odor characteristics of roasted brown rice samples were described according to the national standard GB/T 10221–2012. Between sensory evaluation, water was provided to each panelist to cleanse their palate.

2.3. Electronic nose analysis

E-nose analysis of odors was performed according to Lin et al. (Lin et al., 2013). Approximately 2 g of whole brown rice was added in 20 mL headspace vials, together with 5 mL of boiling water and 1.8 g of

NaCl. After 10 min of heating at 85 °C, an E-nose system (FOX300, Alpha MOS, Heracles, France) equipped with 12 metal oxide gas sensors (MOS sensors) based on different sensing materials was used. The aroma emitted samples were sensed by these sensors and formed a characteristic signal pattern (Jiang, Li, Zheng, Lin, & Hui, 2015). The operation conditions were as follows: flow rate, 150 mL/min; head-space generation time, 60 s; producing temperature, 50 °C; stirring speed, 500 r/min; headspace injection volume, 3 mL; injection velocity, 0.5 mL/s; acquisition time, 360 s; delay time, 120 s.

2.4. Headspace solid phase microextraction of volatiles

HS–SPME of aroma components was carried out followed by Qin et al. (Z. Qin et al., 2013). About 5 g of brown rice was placed in 25 mL headspace vials, and 10 mL of boiling water together with 3.5 g of NaCl were added. Subsequently, 2,4,6-trimethylpyridine (0.2 g/kg, 2 μ L) was subsequently placed into the vial as internal standard (IS). The vials were sealed immediately with PTFE-silicone septa (Supelco, Bellefonte, PA, USA) and equilibrated in a water bath at 60 °C for 10 min. The aging DVB/CAR/PDMS composite extraction fiber (50/30 μ m, Supelco, USA) was inserted into samples vial for 45 min at 60 °C. Then the fiber was removed from the vial and inserted into the injection port of the GC–MS/MS apparatus for analysis of volatile compounds. The peaks of 2,4,6-trimethylpyridine in GC–MS/MS total ion chromatogram of all samples were kept uniformity to ensure the instruments and the protocol.

2.5. Gas chromatography-tandem mass spectrometer

GC-MS/MS analysis was carried out as described by Yang (W. Yang et al., 2016). The analysis of volatile aroma components was analyzed on an Agilent 7890 GC combined with a 7000C triple quadrupole MS operated with an EI ion source (7890B/7000C, Agilent Technologies, Santa Clara, CA, USA). Volatiles were separated using a 5% phenyl-type polarity (HP-5MS) capillary column (Agilent Technologies, Santa Clara, CA, USA; $33 \text{ m} \times 250 \,\mu\text{m} \times 0.25 \,\mu\text{m}$ film thickness). The injector temperature was maintained at 250 °C. High purity helium gas was the carrier gas at a flow rate of 1.0 mL/min. The oven temperature program was set at 30 °C for 1 min, raised to 210 °C at a rate of 10 °C/min, kept at 210 °C for 2 min, finally increased to 250 °C at a rate of 15 °C/min and maintained isothermally for 3 min. The electron ionization source temperature was maintained at 230 °C and mass spectra were obtained by electronic impact at 70 eV. Temperature of interface and quadrupole were 280 °C and 150 °C, respectively. The data were collected at full scan mode with a range of 25-450 amu.

2.6. Scanning electron microscope (SEM)

SEM was followed the method of Zhou et al. with slight modification (Zhou et al., 2009). Brown rice (20–25 g) and soaked brown rice (soaked for 55 min) pre-fixed in 2.5% (w/w) glutaraldehyde with 0.1 M phosphate buffer (pH 7.8) for 2 h and rinsed in distilled water 3 times (15 min each time). These samples were further dehydrated in absolute ethanol (at concentrations of 30%, 50%, 60%, 70%, 80%, 90% and 100% sequentially) and subsequently in tert-butanol (at concentrations of 50%, 70% and 100% sequentially, 10 min each time). Then these samples were dried with the mixture of tert-butanol and acetonitrile. They were coated with a layer of gold by a sputter coater (BAL-TEC AG, Balzers, Liechtenstein) and then photographed using JEOLJSM-6390LV (Japan Electron Optics Laboratory Corporation) scanning electron microscope.

2.7. Statistical analysis

All the tests were conducted in triplicate. Data were expressed as means \pm standard errors and analysis of variance (ANOVA). Duncan's

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