CHANGES IN VOLATILE COMPOUNDS OF COOKED BEEF DURING FREEZE DRYING

R. M. Luo[1,2] G.H. Zhou[1*] C.B. Li[1]

[1] *Key Lab of Meat Processing and Quality Control, College of Food Science and Technology, Nanjing Agricultural University, 210095, PR. China*
[2] *College of Agriculture, Ningxia University, 750021, PR.China*

Key Words: Cooked beef slice, freezing, freeze-drying, rehydration, microstructure

Introduction

Freeze-drying is a dehydration process in which water is removed by sublimation of ice from frozen materials. As ice sublimes, the sublimation interface, which starts at the outside surface of the material recedes, and a porous shell of dried material remains. Vapourized water is transported through the porous layer of the dried material. Freeze-drying, although expensive, is the best method for drying foods with regards to its quality. Preservation of most of the initial raw material properties such as shape, appearance, taste, colour, flavour, texture, biological activity etc. makes it one of the best drying methods. The product also maintains its initial shape and dimensions (James P. George, et al, 2002)

Flavor was one of the most important sensory attributes of cooked meat. It has been investigated intensively and over one thousand volatile compounds have been identified in meat (Mottram 1998). However, few reports were on rehydration products of freeze dried cooked meat. The objectives of the present work were as follows: (1) to investigate the volatile compounds of fresh cooked beef; and (2) to study the changes of volatile compounds of cooked beef during freeze drying.

Materials and Methods

The cooked beef loaf were purchased locally (lanzhou, Gansu, China) and cut into 8mm thickness slice. The samples were frozen at -30 ℃ refrigerator (fareast-II-185L, China) for 5h. Freeze-drying was carried out in freeze dryer (JDG-0.2, CAS, China). Rehydration experiments were carried out at ambient temperature (22±1℃) in deionized water for 30 min.

Twenty grams of fresh cooked beef (FCB) and rehydration sample of dried beef (RDCB) were cut into small thin pieces, frozen by liquid nitrogen and ground into powder, which then was placed in a 15 mL headspace vials (Supelco, Bellefonte, PA, USA), sealed with PTFE/silicone septum and equilibrated for 15 min at 45 °C. For extraction, a 75 μm carboxen-polydimethylsiloxane (CAR-PDMS) SPME fiber (Supelco, Bellefonte, PA, USA) was exposed for 40 min at 45 °C to the vapor phase above 7 g sample (Liu and others 2004). After extraction, the SPME device was removed from the sample vials and immediately inserted into the injection ports (splitless mode) of the GC-MS system. Prior to analysis, the SPME fiber 75 μm CAR/PDMS was preconditioned at 280 °C for 30 min in the gas chromatography.

All analyses were performed using a Finnigan Trace GC-MS (Finnigan, USA). The SPME fibers were thermally desorbed at 250 °C for 120 s in the injector port in the splitless mode. During desorption, the oven was held at 40 °C. A 5% phenyl-methylsilicone (DB-5MS) bonded phase fused-silica capillary column (60 m × 0.32 mm i.d., 1 μm film thickenss, J&W, USA) was used to separate the volatile components from FBC and RDBC. After desorption, the oven was held at 40 °C for 2 min, then heated to 100 °C at 6 °C/min, followed up to 180 °C at 4 °C/min, and finally to 250 °C at 8 °C/min and held at this temperature for 12 min. Helium was used as carrier gas with a constant flow of 1.2 mL/min in the splitless mode. The mass spectrometer operated in electron impact ionisation mode with an emission current of 200 μA and electron energy of 70 eV. The GC-MS transfer line temperature was 250 °C. The mass spectrometer scanned masses from m/z 33 to 450.

Kováts indices (KI) were calculated by using an n-alkanes series (C_5-C_{24}) under the same chromatographic conditions as samples according to Van and Kratz (1963). Compound identification was based on comparison of (1) KIs with those of standards or reported in the literatures (García-Esteban and others 2004; Acree and Arn 1997; Insausti and others 2002); (2) mass spectra with those in the databases of NIST98 (National Institute of Standards and Technology, Gaithersburg, MD, USA) and the Wiley 6.0

---

* Corresponding author. Tel.: +86-25-84395376; Fax: +86-25-84395939. E-mail address: ghZhou@njau.edu.cn (G. H. Zhou).

(Wiley, New York, NY, USA).

Results and Discussion

The volatiles of fresh cooked beef (FCB) and rehydration sample of the freeze dried cooked beef (RDCB) were given in Table 1. Total 32 volatile compounds were tentatively identified in FCB. Twenty two compounds were found in RDCB. The thirty-two volatile compounds from fresh sample were mainly Alkyls (17 compounds), aldehydes (4 compounds), acids (2 compounds), benzene (3 compounds) and alcohols (2 compounds). During freeze drying, most compounds had no remarkable changes. Among the 32 volatiles of cooked beef, 6 compounds were not found in RDCB. They were p-xylene, 2,2-dimethyl-4-pentenal, l-limonene, isobutyl oxalic acid, 4-ethyl-1-octyn-3-benzene, 6-methyl-octadecane. Alkyls were the most numerous compounds in FBC, accounting for 17 of the total 32, followed by 4 hydrocarbons and 2 alcohols, respectively. Hexanal derived from ω-6 unsaturated fatty acids is the most abundant aldehyde identified in FBC and RDBC. The flavor and the aroma note of hexanal had been described as intense grass-like, faint scent (Shahidi and Pegg 1993; Elmore and others 1999).

Table 1 Comparison of volatiles between fresh (a) and rehydration (b) samples

Compound name	Content (a) (%)	Content (b) (%)	Compound names	Content (a) (%)	Content (b) (%)
1,5-dideuteriopentane	1.59	1.77	3-methyl-3-eicosane	2.16	3.17
pentanal	0.09	0.13	nonanal	1.91	3.02
hexanal	61.47	60.01	2-ethyl-1-hexanol	1.10	2.13
1-chloro-hexane	2.99	0.15	dimethyldecane	0.52	0.69
p-xylene	0.55	—	2,6-dimethyl-undecane	0.29	0.17
heptanal	2.31	3.01	isobutyl ester oxalic acid	0.18	—
2,2-dimethyl-4-pentenal	0.11	—	3-methyl-undecane	0.74	0.96
benzaldehyde	2.17	2.09	dodecane	2.20	1.03
1-octen-3- benzene	0.92	0.43	pentadecane	0.07	1.35
2,3-octanedione	1.68	0.79	4-ethyl-1-octyn-3-benzene	0.03	—
decane	3.50	5.63	2-ethyl-1-hexanol	0.21	2.07
l-limonene	0.49	—	tetradecane	1.11	2.01
2,4,6-trimethyl-decane	0.19	0.27	pentadecane	0.07	0.04
2-methyl-octane	0.53	1.23	6-methyl-octadecane	0.02	—
2,6,7-trimethyl-decane	0.63	1.53	1-hexadecanol	0.03	0.12
2-methyl-1-pentanol	0.61	0.22	hexadecanal	0.12	0.15

Conclusions

Freeze drying is based on the dehydration by sublimation of a frozen product. Due to the absence of liquid water and the low temperatures required for the process, most of deterioration and microbiological reaction are stopped which gives a final product of excellent flavor. The research indicated that volatiles of cooked beef were not seriously affected by freeze drying.

References

1. Van den Dool H, Kratz PD. 1963. A generalization of the retention index system including linear temperature programmed gas liquid partition chromatography. J Chromatogr 11:463-471.
2. García-Esteban M, Ansorena D, Astiasarán I, Ruiz J. 2004. Study of the effect of different fiber coatings and extraction conditions on dry cured ham volatile compounds.
3. Acree T, Arn H. 1997. Flavornet: Gas chromatography-olfatography (GCO) of Natural Products, Cornell University. Available form: www.nysaes.cornell.edu/flavornet/. Accessed Dec 12, 2004
4. Insausti K, Berlain MJ, Gorraiz C, Purroy A. 2002. Volatile compounds of raw beef from 5 local Spanish cattle breeds stored under modified atmosphere. J Food Sci 67:1580-1589.
5. Shahidi F, Pegg RB. 1993. Hexanal as an indicator of meat flavor deterioration. J Food Lipids 1:177-186.
6. Elmore JS, Mottram DS, Eva H. 2000. Two-fibre solid-phase microextraction combined with gas chromatography–mass spectrometry for the analysis of volatile aroma compounds in cooked pork. J Chromatogr A 905:233–240.
7. James P. George, A.K. Datta. 2002.Development and validation of heat and mass transfer models for freeze-drying of vegetable slices. Journal of Food Engineering 52: 89-93.