Vietnam J. Agri. Sci. 2016, Vol. 14, No. 10: 1485 - 1490 Tạp chí KH Nông nghiệp Việt Nam 2016, tập 14, số 10: 1485 - 1490
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EFFECT OF DRYING TEMPERATURE ON THE VOLATILE COMPOSITION OF ORTHODOX BLACK TEA

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Received date: 20.01.2016 Accepted date: 31.08.2016

ABSTRACT

The effects of drying temperature on the profile of volatile compounds produced by black tea were evaluated at 80, 90, 100, 110, 120, 130, and 140^{o}C. Aroma concentrate was prepared by the Brewed Extraction Method (BEM) method and analyzed by GC/MS. The volatile compounds content increased as the drying temperature increased from low to high temperatures. However, the relative content of group II volatile compounds, which are the degradation products of carotenoids and amino acids, rapidly increased more than the group I volatile compounds which are mainly the products of lipid breakdown, but when the drying temperature was higher than 120^{o}C, the relative content of some volatile compounds belonging to group II rapidly decreased more than the volatile compounds belonging to group I. The highest flavour indice, which is defined as the ratio between desirable to undesirable volatile compounds, was obtained in samples dried at 120, followed by 110^{o}C. Given the above results, in the present study, the optimal temperature condition to dry black tea was 120^{o}C or 110^{o}C.

Keywords. Aroma compounds, drying, Vietnam OTD black tea.

Ảnh hưởng của nhiệt độ sấy lên thành phần bay hơi của chè đen OTD

TÓM TẮT

Ảnh hưởng của nhiệt độ sấy đến thành phần bay hơi của chè đen OTD được tiến hành ở các nhiệt độ lần lượt là 80, 90, 100, 110, 120, 130 và 140°C. Thành phần bay hơi được thu nhận bằng phương pháp chiết nước-dung môi và phân tích bằng sắc ký khí khối phổ (GC/MS). Thành phần tương đối của các chất bay hơi nhìn chung tăng lên khi nhiệt độ sấy tăng. Tuy nhiên, thành phần tương đối của nhóm các chất bay hơi là sản phẩm phân hủy từ nhóm tiền chất carotenoid và axít amin có xu hướng tăng nhanh hơn so với nhóm các chất bay hơi có nguồn gốc từ quá trình oxi hóa chất béo, nhưng khi nhiệt độ sấy cao hơn 120°C, thành phần tương đối của một số chất bay hơi thuộc nhóm II bị giảm đi nhanh chóng so với một số thành phần bay hơi thuộc nhóm I. Chỉ số chất thơm (FI), được định nghĩa là tỉ lệ giữa nhóm chất bay hơi II trên nhóm chất bay hơi I, đạt giá trị cao nhất ở nhiệt độ sấy 120°C và tiếp theo là ở nhiệt độ sấy 110°C. Theo các kết quả nghiên cứu cho thấy, điều kiện nhiệt độ tối ưu cho quá trình sấy chè đen OTD là ở 120^{o}C hoặc 110°C.

Từ khóa: Chè đen OTD Việt Nam, hợp chất thơm, sấy.

1. INTRODUCTION

Black tea is a fermented tea that is consumed around the world (Senthil Kumar, 2013). The quality of black tea is due to many factors, one of the most contributory factors being its aroma. The volatile compounds of black tea have been identified by many studies, and more than 600 compounds have been reported (Yang *et al.,* 2013). Vietnam is one of the countries that has high black tea production in both types of black tea, OTD and CTC. During the manufacturing process of black tea, the black tea volatile compounds change depending on technical parameters. The purpose of drying is to arrest fermentation and stop enzyme activities. Further, the aroma compounds of black tea are balanced during drying because some of the undesirable compounds are removed, thus accentuating the presence of the more useful compounds. Another purpose of drying is to remove the moisture content up to 95 - 97% to maximize the shelf life (Temple and Boxtel, 1999). The volatile compounds of black tea were investigated in previous studies by gas chromatography (GC) and gas chromatography-mass spectrometry (GC-MS) (Rawat, 2007; Sereshti *et al.*, 2013). However, a comparison of the effect of drying temperatures on volatile composition of black tea during the drying processing is not mentioned in any previous research.

In tea, volatile organic components (VOCs) are present in very low quantities, i.e. 0.01% of the total dry weight, but these have a high impact on the flavour of the products due to their low threshold value and result in high odour units. These VOCs can be divided into two groups. Group I compounds are mainly the products of lipid breakdown, which imparts an undesirable grassy odour. However, group II compounds, which impart a sweet flavoured aroma to black tea, are mainly derived from terpenoids, carotenoids, and amino acids. The aroma quality of black tea depends on the ratio of the sum of group II VOCs to that of group I VOCs, which is the flavour index or volatile flavour compounds (VFC) index (Ravichandran, 2002).

Therefore, in the present paper, we report that the change in the volatile composition during the drying processing of OTD black tea in terms of group I and group II VOCs as well as their ratios at different drying temperatures was extracted by the brewed extraction method and identified using GC-MS.

2. MATERIALS AND METHODS

2.1. Materials and Experimental

Tea leaves of cultivar PH11, representing the genetically diverse Northern Vietnam cultivars, were harvested from Phu Tho province, Vietnam and were used for manufacturing.

Ten kilograms of young shoots, comprised of about 70% with two leaves and a bud, plus minor amounts of three leaves and a bud, and loose leaves, were plucked. The plucked leaves were allowed to wither under ambient conditions for 16 h and then formed into miniature rolling–dhools. The dhool was fermented for 180 min at 30 - 35°C. The fermentation was terminated by drying the dhool to a moisture content of about 3% using a miniature dryer set at different the temperatures of 80, 90, 100, 110, 120, 130, and 140°C inlet (Senthil, 2013). All dried tea samples were collected and kept in polymer bags (200 g/bag) and stored in the dark at room temperature before analysis.

2.2. Volatile compounds analysis

Brewed Extraction Method: Twenty grams of a black tea sample was brewed in 140 ml of deionized boiling water for 10 min. After filtration, the filtrate was saturated with sodium chloride and was extracted using 100 ml of dichloromethane. The extract was dried over anhydrous sodium sulfate for 1h. After the sodium sulfate was filtrated out, the solvent was removed carefully using an evaporative concentrator. The extraction was carried out in duplicate for each sample (Kawakami, 1995). The experiments were carried out in duplicate.

***GC-MS analysis*:** The Thermo trace GC Ultra gas chromatograph coupled with the DSQ II mass spectrometer was used to perform the aroma analysis. An HP-5 capillary column (30 m × 0.25

mm × 0.25 μm) was equipped, with purified helium as the carrier gas, at a constant flow rate of 1 ml min^{-1}. The oven temperature was held at 50°C for 3 min and then increased to 190°C at a rate of 5°C min^{-1} and held at 190ºC for 1 min, and then increased to 240ºC at a rate 20ºC min^{-1} and held at this temp for 3 min. The ion source temperature was set at 200°C and spectra was produced in the electron impact (EI) mode at 70Ev (Lin, 2013). Volatile compounds were identified by electron impact mass spectrum and similarly match index. The flavour index was calculated for each compound expressed as ratio of group II to group I VOCs.

2.3. Statistical analysis

Principal component analysis (PCA) was conducted by Multibase_2015, an add-in tool of Excel version 2010.

3. RESULTS AND DISCUSSION

3.1. Changes in the volatile compounds of OTD black tea by different drying temperatures

Aroma constituents of various black tea products are interesting research topics with potential commercial applications and have been continually investigated by many researchers (Pripdeevech and Wongpornchai, 2013). The brewed extraction was employed to extract volatile flavour components in order to characterize dried black tea flavour. The GC-MS profile of the extracted flavours shows the presence of a wide range of compounds, including terpenoids, alcohols, acids, aldehydes and ketones. Table 1 shows the list of volatile compounds that belong to group I and group II, which were identified in the dried black tea obtained from the various drying temperatures. Most of the compounds have previously been reported from black tea either on polar or non-polar GC columns by different extraction methods such as SDE (simultaneous distillation extraction), hydro-distillation, and Clevenger (Rawat, 2007).

In dried black tea, volatile compounds in both groups increased as drying temperature increased from 80°C to 120°C and decreased when the drying temperature was higher than 120°C. The results showed, however, that the volatile compounds of group II increased more rapidly than those of group I. This result could be explained by the flavour index, which increased from samples dried at 80°C to 120°C. Many volatile compounds were produced during drying and their content increased as a function of drying temperature, especially the by-products of Maillard reactions, such as 2-acetyl-1-pyrroline and N-ethyl-succinimide, as well as the degradation products of fatty acids and carotenoids. The flavour index of samples at drying temperatures of 130 and 140°C decreased due to evaporation, and group II lost more than group I.

3.2. Principal component analysis (PCA)

Principal component analysis (PCA) was used to determine the effect of drying temperature on the composition of volatile compounds in black tea (Fig. 1 and 2). The principal components (PC) were chosen according to the highest significance of drying temperature as well as those with the highest explanation of the variation. The first principal component (PC1) explained 60.9% of the total variation of the volatile compounds listed in Table 1, and PC2 accounted for 23.5%. The PC1 on the negative axis was highly influenced by the following compounds: trans-geraniol, 3-hexen-1-ol, β-ionol, acetaldehyde, *trans*-linaloloxide, salicylic acid, benzyl alcohol, 2-hexen-1-ol, (*E*)-epoxylinalol, benzenethanol, and beta-ionol. Some of them were reported as the degradation products of fatty acids and carotenoids by drying temperature, such as 3-hexen-1-ol and beta-ionol (Ho *et al.*, 2015). We observed that all these compounds were related to samples dried at temperatures 80, 90, 100, 110, and 120°C.

Table 1. Volatile compounds commonly detected in Orthodox black tea samples by brewed extraction/GC-MS

No	Volatile compounds	Peak area percentage (%)						
		Drying temperature (°C)						
		80	90	100	110	120	130	140
	Group I*							
1	3-hexen-1-ol	2.14	2.34	2.35	2.50	2.61	1.98	1.70
2	hexanal	2.64	2.20	2.24	2.22	2.01	1.51	1.01
3	(E)-2-hexen-1-ol	2.59	2.43	1.79	1.89	3.07	0.76	0.66
4	(E)-2-hexenal	1.88	2.28	1.46	0.70	1.61	0.94	0.37
5	hexanol	1.19	1.22	1.27	1.30	2.77	0.80	0.72
6	nonanal	0.13	0.31	0.84	1.67	1.95	3.25	3.53
7	2-nonanol	0.68	0.61	0.82	0.83	0.74	2.68	3.76
	Group II*							
8	acetaldehyde	0.19	0.22	0.29	0.37	0.17	nd	nd
9	benzaldehyde	nd	0.08	0.29	0.29	nd	nd	nd
10	*trans*-linaloloxide	0.34	0.70	0.58	0.92	0.18	nd	nd
11	β-linalool	0.46	0.51	0.95	1.27	1.74	0.82	0.68
12	benzyl alcohol	3.09	3.01	2.28	2.50	3.53	1.04	0.71
13	benzeneacetaldehyde	1.69	1.73	1.56	1.74	2.80	1.08	0.79
14	phenylethyl alcohol	1.69	1.66	1.54	1.53	2.69	0.89	0.65
15	epoxylinalol	1.35	1.47	1.15	1.10	1.00	0.47	0.38
16	*cis*-linaloloxide	0.46	0.54	0.76	1.18	1.20	1.04	0.91
17	2-acetyl-1-pyrroline	1.32	1.99	1.43	1.57	1.78	6.36	7.86
18	methyl salicylate	0.14	0.30	0.20	0.23	0.74	0.61	0.52
19	succinimide, N-ethyl-	nd	1.03	1.02	1.01	1.08	1.61	1.90
20	*trans*-geraniol	0.05	0.08	0.10	0.17	0.15	0.05	0.04
21	salicylic acid	0.60	0.63	0.72	0.79	0.86	nd	nd
22	β-damascenone	0.29	0.69	0.78	0.87	1.00	0.39	nd
23	benzaldehyde, 4-hydroxy-3-methoxy-	0.29	0.49	0.57	0.71	0.94	0.41	0.03
24	benzeneethanol, 4-hydroxy-	1.36	1.17	1.07	0.84	0.65	nd	nd
25	ethyl linalool	1.19	1.22	1.45	0.83	0.77	0.49	0.29
26	3-hydroxy-.beta.-damascone	0.34	0.13	0.29	0.29	0.46	nd	nd
27	β-ionone	1.19	1.27	1.41	1.47	2.82	1.08	0.87
28	α-ionone	1.01	1.05	1.27	1.07	2.79	0.73	0.37
29	beta. Ionol	0.55	0.48	0.65	0.78	0.58	0.05	0.02
	Group I	11.24	11.40	10.78	11.11	14.76	11.92	11.75
	Group II	14.98	17.92	17.49	18.82	25.03	16.52	15.50
	Flavour index (Group II/Group I)	1.33	1.57	1.62	1.68	1.70	1.39	1.32

*Note: * Volatile compounds belong to Group I and Group II was mentioned by Ramaswamy R., 2002 .*

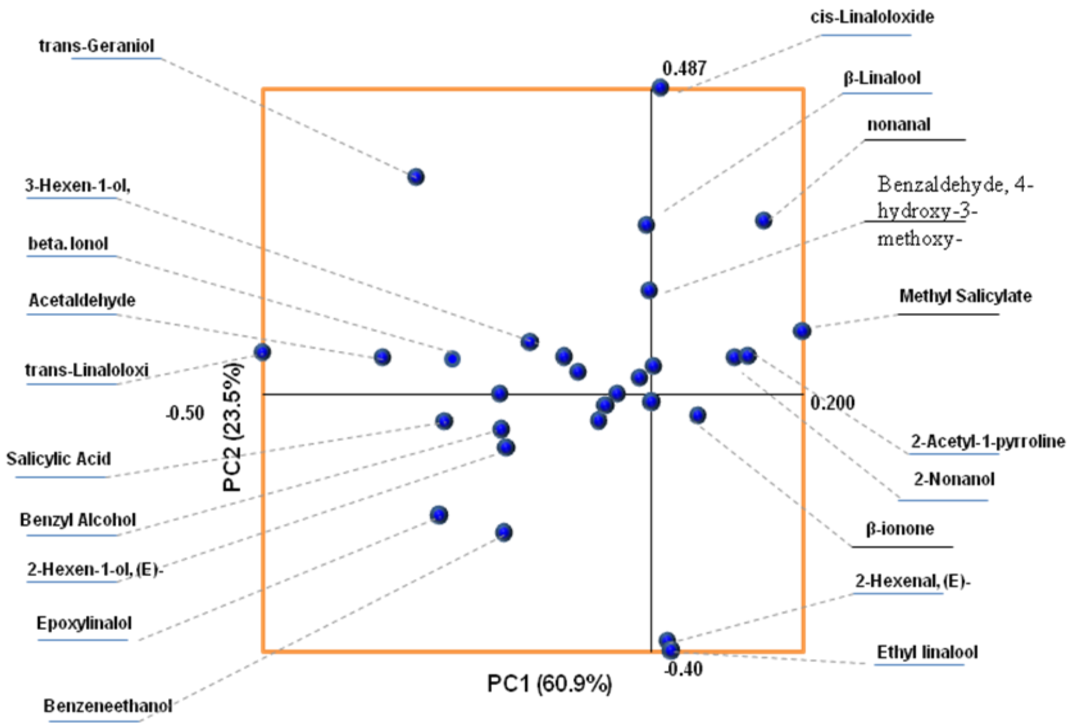

Figure 1. Variables plot between the first 2 PCs

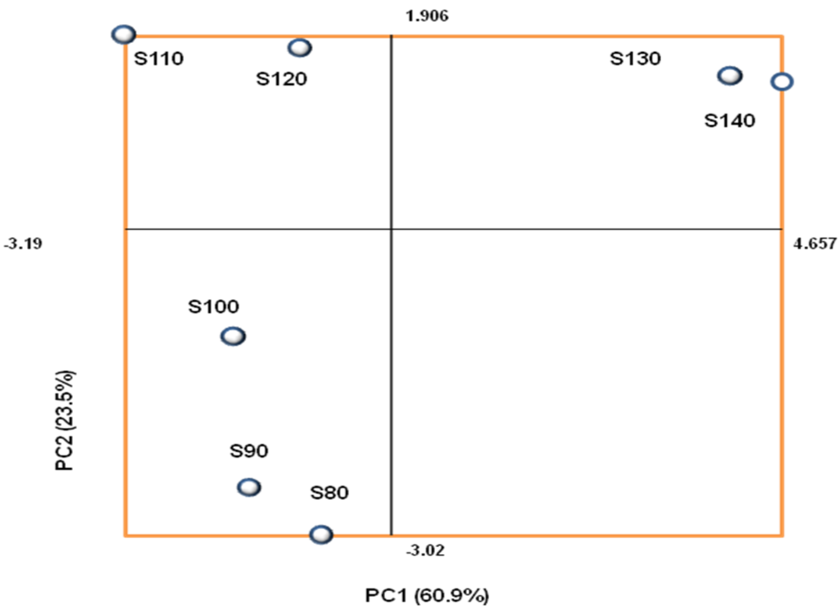

Figure 2. Score plot between the first 2 PCs

The PC1 in the positive axis grouped the compounds that were observed related to higher drying temperatures i.e 130 and 140°C, such as nonanal, benzaldehyde, 4-hydroxy-3-methoxy-, 2-acetyl-1-pyrroline, 2-nonanol, β-ionone, 2-hexenal, and (*E*)- ethyl linalool. Of these, 2-acetyl-1-pyrroline, a product of Maillard reactions, and 2-nonanol and nonanal, products of lipid oxidation, were found at a significantly higher relative content (Ho *et al.*,, 2015). Regarding PC2, compounds with the highest weight were 4-hydroxy-3-methoxy-benzaldehyde, nonanal, 3-hexen-1-ol, β-ionol, acetaldehyde, benzaldehyde, 4-hydroxy-3-methoxy, and 2-acetyl-1-pyrroline. All these compounds were grouped on the positive side and related to samples dried at 110, 120, 130, and 140°C. While the compounds salicylic acid, (*E*)-2-hexen-1-ol, and (*E*)-2-hexenal were found on the negative axis and related to lower drying temperatures. The score plot from PC1 and PC2 (Fig. 2) helped to discriminate the treatments by drying temperature. The treatments at 80, 90, and 100°C drying temperatures were located on the negative side both of PC1 and PC2, while the treatments at 130 and 140°C drying temperatures were found on the positive side of both PC1 and PC2, and samples dried at 110 and 120°C were located on negative side of PC1.

4. CONCLUSION

The drying temperature process had an effect on the profile of volatile compounds. In dried black tea, volatile compounds in both groups increased as drying temperature increased from 80°C to 120°C and decreased when drying temperatures were higher than 120°C. The results, however, showed that the volatile compounds of group II increased more rapidly than those of group I. This result would explain the flavour index which increased in dried samples at 80°C to 120°C. The flavour index of samples at drying temperatures of 130 and 140°C decreased due to evaporation and group II lost more than group I. In the case of aroma quality, we recommend an optimal drying temperature should be arranged from 110 to 120°C with flavour indices from 1.68 to 1.70.

Acknowledgments: The authors would like to thank the Ministry of Education & Training of Vietnam for providing financial support.

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