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EFFECT OF PACKAGING ON VOLATILE PROFILES OF MULBERRY TEA

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ABSTRACT

This research aimed to investigate the effect of packaging materials and storage time on volatile profiles of mulberry tea. The mulberry tea samples were packaged in linear low-density polyethylene laminated aluminum (AL) bag and polypropylene (PP) bag and stored at $30\pm1°C$ for 18 months. The volatile profiles were monitored using a headspace solid phase microextraction/gas chromatography-mass spectrometry. The results showed that storage time had no significant impact on the key volatile compounds of the AL packaged samples. However, some volatiles associated with lipid oxidation like hexanal and 4-oxo-2-nonenal were observed to significantly ($p<0.05$) increase in the PP packaged samples with storage time.

1. INTRODUCTION

The consumption of mulberry (*Morus alba* L.) leaf tea products is popularly increasing worldwide especially in Asian countries increasing due to its special flavors and health benefits (SÁNCHEZ-SALCEDO *et al.*, 2015; SHOAIB ZAFAR *et al.*, 2013).

It has been recognized that the sensory quality of herbal tea especially flavor profile is a key factor for quality classification and content. Since the changes in flavor and aroma profile of herbal tea during storage result in the greatly decline in its quality and consumer acceptance, appropriate packaging is crucial for preserving the product's flavors and nutritional quality throughout the storage. HARNNURAK and RIEBROY (2014) reported that the alumimium foil bag could protect the quality attributes of mulberry leaf tea during storage better than paper bag and sachet, respectively. LEE and CHAMBERS (2010) reported that packaging materials significantly ($p<0.05$) affected changes in volatile components of green tea during storage.

To date, several researches have undertaken on the quality attributes and health benefit of mulberry tea. However, scientific information on aroma constituents and quality attributes of herbal tea from mulberry leaves during storage are still limited. Information on the volatile compounds of mulberry tea is beneficial to the herbal tea industry for quality control of products and shelf life evaluation. The purpose of this study was to determine the effect of packaging and storage time on volatile profiles of mulberry leaf tea using headspace solid phase microextraction-gas chromatography/mass spectrometry (SPME-GC/MS).

2. MATERIAL AND METHODS

2.1. Tea preparation and storage condition

Herbal mulberry tea (Thai Silk Products Co., Ltd., Thailand) was prepared using the process of The Queen Sirikit Department of Sericulture as described by RUENGDECH and SIRIPATRAWAN (2019). One hundred grams of mulberry tea were packed into linear low-density polyethylene laminated aluminium (AL) bag (10×15 cm²; 0.0924 mm in thickness) and polypropylene (PP) bag (10×15 cm²; 0.0774 mm in thickness), sealed under atmospheric condition, and stored at 30±1°C in a controlled humidity chamber at 75% RH. The samples were randomly retrieved at three-month interval to monitor volatile profiles using SPME-GC/MS.

2.2. Determination of volatile compounds

The tea infusion was prepared by adding 2 g of mulberry leaf tea in 100 ml of hot water at 95±2°C. The samples were infused for 5 min and were then filtered through a Whatman No.4 filter papers.

Volatile compounds of mulberry tea infusion were collected by SPME technique. Briefly, 10 ml of tea infusion were pipetted into a 20 ml glass vial (Chromselection, Brescia, Italy) and 3 g of sodium chloride was added. The vial was then tightly closed with aluminum crimp cap attached with a PTFE silicone septum (Chromselection, Brescia, Italy). After 10 min of sample equilibration at 40°C in an auto-incubator, the 50/30 μm Divinylbenzene/Carboxen/ Polydimethylsiloxane (DVB/CAR/PDMS) SPME fiber (Stableflex; Supelco. Inc., Bellefonte, PA, USA) was exposed to the headspace of the sample vial for 30 min and then immediately inserted into the gas chromatograph (GC)

(AutoSystem XL GC, PerkinElmer, Waltham, MA, USA) injection port with splitless mode at 250°C for 5 min.

The GC column was Stabilwax®-MS capillary column (30 m × 0.25 mm ID, 0.25 μm film thickness; Restek, Bellefonte, PA, USA) was employed for GC-MS analysis. Helium was used as carrier gas with flow rate at 1 ml/min. The GC oven temperature gradient was 50°C (held for 5 min) initially, increased to 125°C (held for 3 min) at 3°C min⁻¹, then ramped to 180°C (held for 3 min) at 2°C min⁻¹, and finally increased to 230°C (held for 5 min) at 6°C min⁻¹. The transfer line temperature was 280°C. Mass spectrometer (MS) (Turbomass MS, PerkinElmer, Waltham, MA, USA) was coupled to GC. The ion source temperature was 230°C and the MS was scanned at 70 eV over 40 to 500 mass range. Compounds were tentatively identified by using the National Institute of Standards and Technology (NIST) database with the reverse match factor more than 800.

Principal components analysis (PCA) was carried out to analyzed volatile compound data. PCA has been used to visualize the hidden trends in a data matrix composed of n (13) samples defined by m (12) selected volatile compounds, which changed in intensity during storage.

3. RESULTS AND DISCUSSION

The effects of packaging materials and storage time on volatile components of mulberry leaf tea stored at 30±1°C were determined using SPME-GC/MS. GC chromatograms of volatile profiles of mulberry leaf tea in PP stored for 0, 6, 12, and 18 months are shown in Fig. 1. Volatile compounds were then identified by comparison of their mass spectra of authentic reference compound with those of the NIST (version 11.0) database. Fifty-three volatile components in mulberry leaf tea include aldehyde, alcohol, acid, ester, and others. Volatile compounds including benzaldehyde; 1-octen-3-ol; 6-methyl-5-hepten-2-one; (E, E)-2,4-heptadienal; nonanal; decanal; 2,6,6-trimethyl-1-cyclohexene-1-carboxaldehyde; and 6,10-dimethyl-5,9-undecadien-2-one found in our sample were also found in mulberry tea produced in China as report by LIU et al. (2012). Hexanal and 2-hexenal, which have been reported to be responsible for green and grassy odors in mulberry leaves (RUENGDECH and SIRIPATRAWAN, 2019; TANAKA *et al.*, 2009), also present in the mulberry leaf tea in our study. 2(4H)-benzofuranone,5,6,7,7a-tetrahydro-4,4,7a-trimethyl contributed to sweet, herbaceous, and tea-like odor has been reported as an important aroma constituent of tea and herbal tea (CHEN *et al.*, 2013).

The GC results showed that storage time did not affect volatile components of mulberry the leaf tea. The average concentration of all volatile components of the samples in AL did not significantly change over the whole storage period. However, intensity of volatiles of the PP packaged samples including 2-methyl-butanal; hexanal; 5-hepten-2-one,6-methyl; 6-methyl-3,5-heptadiene-2-one; 3,5-octadien-2-one; heptan-2-one; 3,4-dimethyl-benzaldehyde; 4-oxo-2-nonenal; 3-buten-2-one-4(2,6,6-trimethyl-1-cyclohexen-1-yl); 2(4H)-benzofuranone,5,6,7, 7a-tetrahydro-4,4,7a-trimethyl; 5,9-undecadien-2-one,6,10-dimethyl; and 1,6-octadien-3-ol- 3,7-dimethyl,2-aminobenzoate changed with storage time. The intensities of 2-methyl-butanal and 3,4-dimethyl-benzaldehyde found in mulberry leaf tea in PP bag gradually decreased with increasing storage time, whereas the remaining 10 compounds gradually increased during 12 months of storage and rapidly increased from 15 to 18 months of storage (Fig. 2).
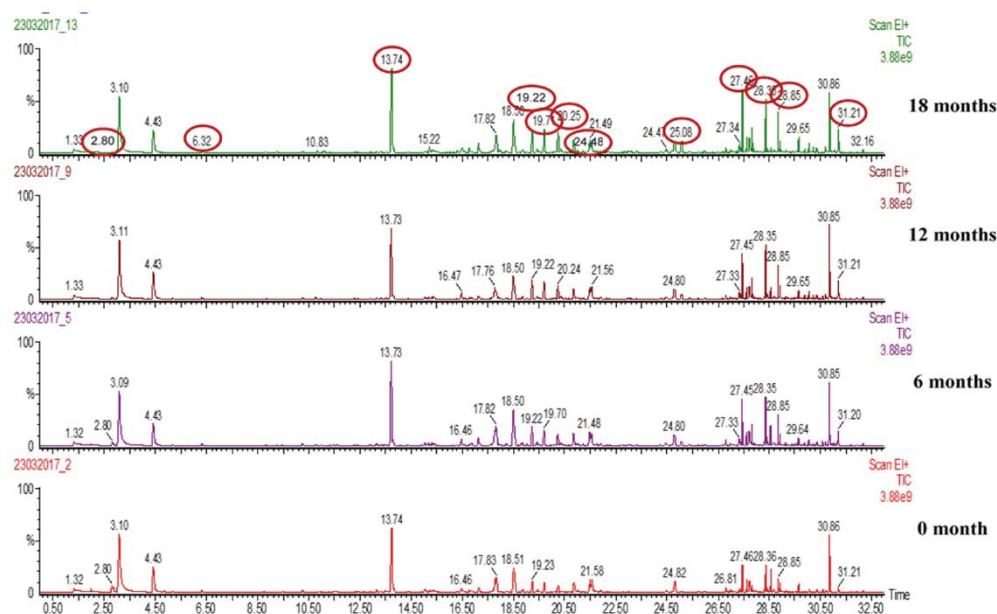
Figure 1. GC chromatograms of volatile compounds of mulberry leaf tea in PP bag during storage at 30±1°C, 75% RH for 0, 6, 12, and 18 months. The peaks of twelve volatile compounds with color circle indicate significant ($p<0.05$) changed in intensity during storage.
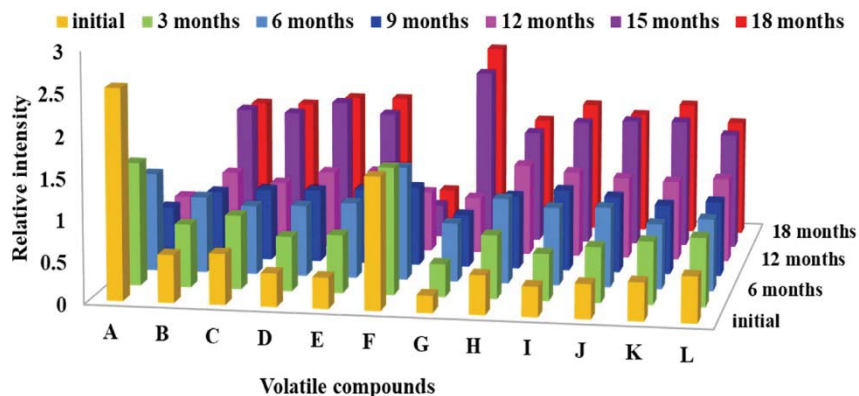

Figure 2. Changing in intensity of twelve volatile compounds found in the mulberry leaf tea packaged in PP during storage at 30±1°C, 75% RH for 18 months.
A: 2-methyl-butanal; B: hexanal; C: 5-hepten-2-one,6-methyl; D: 6-methyl-3,5-heptadiene-2-one; E: 3,5-octadien-2-one; F: 3,4-dimethyl-benzaldehyde; G: 4-oxo-2-noenal; H: 3-buten-2-one-4(2,6,6-trimethyl-1-cyclohexen-1-yl); I: 2(4H)-benzofuranone,5,6,7,7a-tetra- hydro-4,4,7a-trimethyl; J: heptan-2-one; K: 5,9-undecadien-2-one,6,10-dimethyl ; L: 1,6-octadien-3-ol-3,7-dimethyl,2-aminobenzoate.

Changes in the intensity of some volatiles associated with oxidation reaction of components in mulberry leaf tea. Hexanal generated from lipid oxidation contributes to off-flavors such as stale, cardboard, and fishy in foods. Hexanal content is directly related to the deterioration of food quality during storage (MAARSE, 2017). A 4-oxo-2-nonenal is a lipid peroxidation product derived from oxidized omega-6 polyunsaturated fatty acids such as arachidonic acid and linoleic acid. It has been widely used as a marker of lipid

peroxidation (LEE and BLAIR, 2000). 2-methyl-Butanal, which is a branched-chain aldehyde, is generally perceived as malty or chocolate-like odor in food products. A decrease of 2-methyl-butanal of mulberry leaf tea in PP during storage may be attribute to the conversion of branched-chain aldehyde compounds to alcohol or acid compounds via reduction or oxidation, respectively (SMIT et al., 2009).

SHOAIB ZAFAR et al. (2013) reported that the fresh mulberry leaves contain fat in a range of 0.64 to 1.51 g/100 g dried sample. While in dried mulberry leaves, fat is in a range of 2.09 to 4.93 g/100g dried sample. During storage, UV-light, oxygen, and moisture can all induce lipid oxidation causing off-flavor development in mulberry leaf tea. Since AL has better UV-light, oxygen, and moisture barrier properties than PP, it can protect the product from lipid oxidation better than PP bag. This result is in agreement with the studied of KAACK and CHRISTENSEN (2008), who reported that AL bag had more efficiency to protect the quality of tea processed from flowers of black elder (*Sambucus nigra* L.) than plastic film.

The PCA used to facilitate the data interpretation of various volatile compounds of mulberry tea during storage at different time points. For PCA, data were made up of 78 samples from 13 groups and each group had 6 replicates. Each sample was analyzed using 12 selected volatile compounds, which changed in intensity during storage. The selected volatile compounds of all samples were arranged in a matrix (78 samples×12 volatiles variables). Fig. 3 shows PCA score plot and loading plot of mulberry leaf tea stored at 30±1°C for 18 months using the selected volatile profiles data set. The PCA results allowed for good discrimination among samples, with the PC1 and PC2 can explain 99% of the total variance. The results showed that initial sample and all samples in AL located on the negative region of PC1. The result indicated that volatile compounds of the sample in AL were not significantly different during storage. The samples in PP could divide into 2 groups; one group is the samples store at 3 to 12 months and the other is the samples stored from 15 to 18 months. The PCA results are in accordance with the GC/MS results, which showed that volatile profiles of mulberry leaf tea in AL were not significantly different throughout the storage. Whereas storage time affected volatile intensity of the sample in PP. Twelve volatiles found in mulberry leaf tea in PP of which their intensities gradually changed during 12 months of storage and greatly changed at 15 and 18 months of storage. Thus, the samples could be classified into three groups according to their volatile patterns.
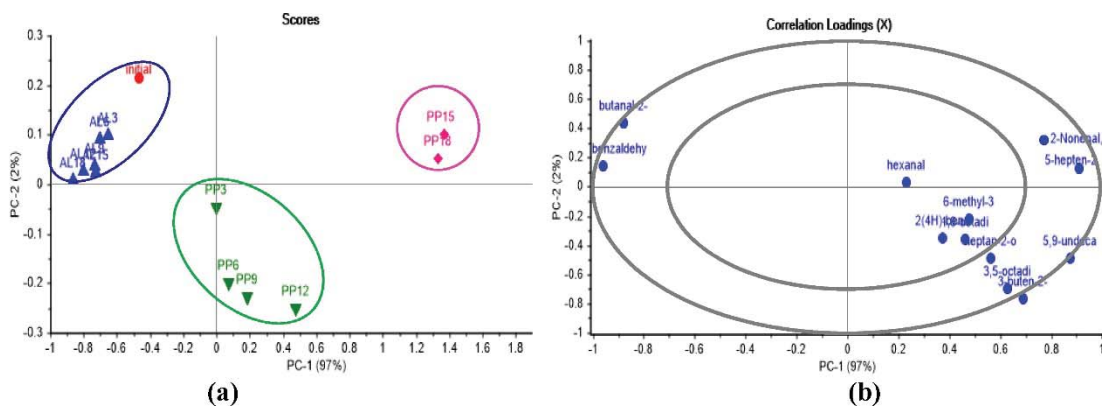

(a) **(b)**

Figure 3. PCA score plot (a) and loading plot (b) on PC1 and PC2 of mulberry leaf tea in AL and PP bags and stored at 30±1°C, 75% RH for 18 months using twelve selected volatile compounds data set.

The loading plot established the relative importance of each selected volatile compounds, and the relationships between the volatiles and the samples. Most of the selected volatile compounds positively correlated to PC1 whereas 2-methyl-butanal and 3,4-dimethyl-benzaldehyde negatively correlated to PC1. From the loading plot, volatiles positively correlated to PC1 were associated with the samples in PP whereas volatiles negatively correlated to PC1 were associated with the samples in AL. The analysis of volatile profiles of mulberry tea suggested that packaging materials used affected its volatile profiles and possibly the odor and flavor of the products.

4. CONCLUSIONS

The results of this study demonstrated that packaging material and storage time affected volatile profiles of mulberry tea. AL bag showed high-effective barrier properties with the quality mulberry leaf tea were not significantly different throughout the storage. Due to a low effective barrier property of PP, the qualities of mulberry leaf tea significantly ($p<0.05$) difference during storage. To protect the product qualities during storage, AL bag is suggested as the packaging of mulberry leaf tea. The information obtained from this study is useful for the selection of suitable packaging material and storage time for mulberry or herbal tea.

ACKNOWLEDGEMENTS

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