Araştırma Makalesi / Research Paper
Bayburt Üniversitesi Fen Bilimleri Dergisi
Bayburt University Journal of Science

dergipark.gov.tr/bufbd
ISSN: 2667-579X

Cilt 7, Sayı 2, Yıl 2024, Sayfa 79-90
Vol 7, Issue 2, Year 2024, Pages 79-90
doi.org/10.55117/bufbd.1547999

Geliş Tarihi / Date of Recieve: 10.09.2024
Revizyon Tarihi / Date of Revision: 12.09.2024
Kabul Tarihi / Date of Acceptance: 16.09.2024

Volatiles and Sensory Characteristics of Turkish Black Tea Drink Produced by Rapid Solid-Liquid Dynamic Extraction

Kübra DOĞRU[1*], Ahmet Levent İNANÇ[1]

[1]*Kahramanmaraş Sütçü İmam University, Department of Food Engineering, Kahramanmaraş,Türkiye*

Keywords:

Brewed black tea, Extraction, Oxygenated water

Abstract

Black tea occupies the majority of the world market among tea varieties. Turkish black tea is one of tea varieties, and it is the most popular drinks in Turkey, too. But its brewing time is longer than black teas such as Ceylon tea. The rapid extraction system in the laboratory scale was established to reduce brewing time of Turkish black tea. The effects of oxygenated pure water as well as pure water in the system were investigated to approach the properties of classical Turkish tea. The properties of the teas were determined and compared by sensory and volatile component analysis. 14 aldehydes, 11 alcohols, 7 ketones, 2 aliphatic hydrocarbons and 1 acid compound were identified in the teas. The major and minor alcohols in the samples were linalool (56.18 µg/g) and geraniol (2.22 µg/g). Three compounds with the highest concentration in ketone group were 6-methyl-5-hepten-2-one (59.68 µg/g), β-ionone (51.93 µg/g) and α-ionone (44.63 µg/g) detected in the classic tea. Two compounds in aliphatic hydrocarbon group were decane and dodecane. Methyl salicylate acid is the only compound in the acid group. Its highest concentration (25.93 µg/g) was detected in the classic tea. The significant differences among volatile compounds in the teas extracted by two methods were evaluate at the level of $p < 0.05$. Ranking and scoring difference-from-control tests were applied in the sensory analysis. It was determined that the best times of the teas produced using the oxygenated and the non-oxygenated waters (each separately) with the dynamic liquid extraction method were 120s. The color and brightness values of the samples were different from each other and from reference sample. The aroma and odor values of the teas obtained from the dynamic liquid extraction were similar to each other but different from reference sample ($p<0.05$).

*e-mail: sariyeyrl80@hotmail.com

Bu makaleye atıf yapmak için:
Kübra DOĞRU; Ahmet Levent İNANÇ, "Fatigue Analysis of an Aerospace Elastoplastic Structural Cylindrical Component with Hole under Cyclic Mechanical Load using COMSOL Multiphysics and Taguchi Method Optimization", Bayburt Üniversitesi Fen Bilimleri Dergisi, C. 7, s 2, ss. 79-90

How to cite this article:
Kübra DOĞRU; Ahmet Levent İNANÇ, "Volatiles and Sensory Characteristics of Turkish Black Tea Drink Produced by Rapid Solid-Liquid Dynamic Extraction", Bayburt University Journal of Science, vol. 7, no 2, pp. 79-90

1 INTRODUCTION

Tea is a healthy drink [1]. There are six most basic types of tea: green tea, black tea, white tea, yellow tea, pu-erh tea and oolong tea. Black tea has become the most popular tea in the world, it accounts for over 90% of all tea sold in the western countries and today, there are 13 types of black tea and 794 related products in the Chinese tea market [2]. But Turkey was the most tea-consuming country in the world with approximately 6.96 kilos in 2016 although that figure is 1.25 kilos per person per year in China [3,4]. Moreover, Turkey ranks 7th in the world in terms of the size of tea cultivation land, 5th in dry tea production and 1st in per capita tea consumption in 2019 [5]. World black tea production is projected to grow (at 2.9 percent annually to reach 4.17 million tonnes) at a slightly higher rate compared to the previous decade by 2023 because it may be a positive effect on health. The major chemical compounds of tea; which has the effects on the health benefits, are catechins, alkaloids (caffeine, theobromine and theophylline), amino acids, volatile compounds, carbohydrates, lipids, vitamins, inorganic elements and organic acids [4, 6,7]. Two of the important factors that determine the quality of black tea are aroma and the brewing conditions. Aroma is formed by the comprehensive action of volatile aroma compounds contained in black tea. It has also an important factor on the flavor characteristics of black tea and determines the value of black tea [8, 9]. Generally, tea as a beverage is made as a liquid prepared by mixing hot water with dried tea leaves or extracts obtained from tea leaves [10]. Brewing temperature, time, vessel, the water-to-leaf ratio, and the water composition affect the taste of the brewed [11-13].

It is reported that information on the importance of water content in brewing tea dates back to 758 AD and it was felt that tea made from mountains streams was ideal, river water was sufficient, and well water was inferior [14, 15]. MKT (2016) posted that the water source has a direct correlation to how much oxygen is available in the water [16]. Oxygen generally makes water taste good and brew better tea. The source of water that contains the highest levels of oxygen are often found in high mountain creeks and fast-moving water. But based on Henry's Law it is expected the solubility of oxygen in liquid and solid excipients to similarly decrease with increasing temperature [17].

Turkish-style tea is brewed with ground roasted black tea in a teapot or samovar over continuous boiling water [18]. Although there are different opinions like 4, 15, 20, 25 or 30 minutes about the brewing time for the traditional Turkish black Tea, the ideal brewing time is approximately 15 minutes. Also, Brewing time may vary depending on the type of tea and the taste of the drinker [19-22]. On the website of a tea company, according to the results of its own research, "Black teas produced in the tropical climate zone (Sri Lanka, India, Kenya, Indonesia, China) are prepared by steeping in leaves form for 5-7 minutes, and teas in bagged tea form for 2-3 minutes. It is stated that Turkish teas achieve the optimum taste profile in the 18th minute" [23].

The time for the beverage to reach the customer could be problem at offices where every minute is valuable. it could be resolved by tea vending machines [24]. Tegeltija et al., (2020) reported that vending machines such as tea making machine are mostly installed in busy big places such as shopping malls, bus and train stations, airports, schools, university campuses, companies [25]. There are two distinct types of tea and coffee vending machines (Household Tea and Coffee Machine and Tea and Coffee Vending Machine). The operation of Household Tea and Coffee Machine is quite simple. It consists of a vessel containing tea or coffee premix powder and water vessel which is connected to the heater. The water is heated and added into the container thereby providing the required beverage. In the past years, for example, in 2011, one study (tea-matic project) was performed in Arçelik Inc. Research and Development Centre, as a tea maker concept, which has a movable infuser, was studied to brew black tea by Çoban (2011) [26]. Based on that point, it was designed a food operation process that provides the desired Turkish black tea of the quality in a shorter time. Tea brewed by the traditional method was accepted as the desired quality tea drink and it was investigated how much the quality of tea drinks obtained from the designed process approached to the quality of tea brewed by the traditional method.

2 MATERIALS AND METHODS

2.1 Material

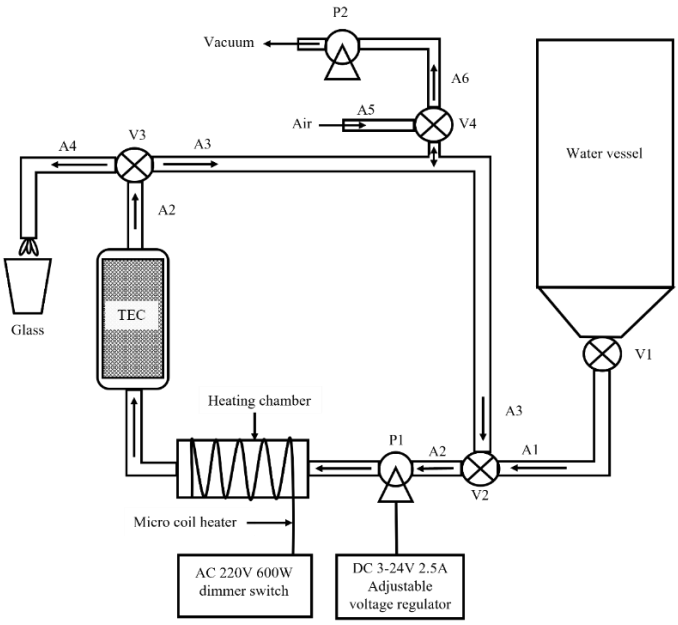
Altınbaş Black tea of Çaykur brand (Rize, Turkey), purchasing from a local market was used as black tea material. The standard reagents and methanol (Merck, İstanbul, Turkey) were chromatographic grade, and the others were all analytical grade chemicals.

2.2 Method

2.2.1 Dynamic tea extraction process

Dynamic tea extraction process for tea brewing was carried out in a laboratory-built apparatus. Schematic diagram of it is illustrated in Fig. 1. It consists of a water vessel (2.5L PET bottle with cap fitted with two way mini ball valve), a mini water pump (Pandoraplanet; model 385, DC 6V-12V, high temperature resistance 100°C), a 316 stainless steel cylindrical heating chamber (L:130, ID: 8 and OD: 18; mm) installed a coiled heater (500W, 220V AC, L:82 and ID: 18; mm) on, a rotary dimmer switch (3-600W, 230V AC), a 316 stainless steel cylindrical tea extraction chamber (TEC); having L:100, ID: 27 and OD: 50; mm, high temperature silicone tubing hose-food grade (9x14; mm), an Universal DC 3-24V adjustable voltage regulator (LED Display screen, Input:100-240VAC 50-60Hz, output: 2.5A 60W), three stainless steel 304 high pressure high temperature ¾ inch L port three-way mini ball female valves (port size: 9mm), a stainless steel 304 ¾ inch two-way mini ball female valves (port size: 12mm), a mini vacuum pump (Boeco, R-300), ¾ inch BSPT male x 10mm hose barbed 304 stainless steel pipe fitting hose tail connectors and hose clamps.

Both ends of the main body of the extraction chamber were sealed with nipples fitted with quick couplings (3/8-inch male thread socket; 506 EGB 17 and 10mm hose tail socket; 506 H 10). Disc filters (Stainless Steel Woven Wire 100 mesh) were placed between the nipples and the body to retain solid tea particles. The chambers, valves and hoses were entirely surrounded by thermal insulation materials. The operation principle of the process consisted of bleeding the air from the system, adjusting the temperature of the system to 85±3°C by circulating water without being present tea in TEC chamber, and water cycle for tea extraction. The total internal volume of the hoses between valves V1 and V2 was 200 ml. When A2-A3 ports of V2 and V3 valves were open (A1, A4 ports and V4 valve were closed), the total internal volume of the system (including valves, hoses, pumps and hoses) was 200mL, too. The circulation time of 200 ml water, which left from V2 valve (A2 direction) and entered to V2 valve (A3 direction) again, was adjusted to 30 seconds and the temperature to 85±3°C by the voltages of the water pump and heater. That is one cycle of water passing through the tea chamber. Putting 5 g of tea into TEC chamber, 1-5 cycles were performed. A2-A4 port of the V3 valve (A3 was closed) and A3-A5 port of V4 valve (A6 was closed) were opened and the brewed tea sample was taken from the A4 line after the desired number of cycles. All controls (opening and closing of the valves, adjusting the pumps) were manually done. The volume of brewed tea obtained from the process was 180±3 ml. Two kinds of pure water were used in the process; the oxygenated and non-oxygenated water. The HI-98193 portable dissolved oxygen meter (Hanna, UK) was used to been determined the dissolved oxygen amounts in the waters. Non-oxygenated water had 7.5±0.02 mg/L of dissolved oxygen concentration. Oxygenated water which had 35.7±0.04 mg/L of dissolved oxygen concentration was obtained as described below. Half of the PET bottle volume was filled with oxygen-free water at room temperature. After cap with valve was closed, the other half was filled by pressing oxygen from the pure oxygen tube. It was kept for 24 hours at room temperature to bring to equilibrium it by shaking the bottle for 15 minutes.



A, flow direction; P1, water pump; P2, vacuum pump; TEC, tea extraction chamber; V, valve

Figure 1. Schematic diagram of the tea extraction system

2.2.2 Traditional black tea brewing process

Hot oxygen-free water of 200mL at 85±3°C was poured into a thermal isolated vessel then black tea of 5 g was added on it and it was left to brew for 20 minutes (the time that the tea manufacturer recommended). The brewed tea in the vessel was passed through filter (Stainless Steel Woven Wire 100 Mesh) and a 180±3 ml volume of brewed tea sample was obtained. That tea was accepted as the reference sample (R). The samples coded as O and NO refer black teas prepared using oxygenated and non-oxygenated water by the dynamic tea extraction process.

2.3 Analysis

2.3.1 Determinations of volatiles by gas chromatography-mass spectrometry

Static solid-phase microextraction (SPME; DVB/CAR/PDMS; Divinylbenzene/Carboxen/Polydimethylsiloxane; 50/30 µm coating thickness; Supelco, Bellefonte, PA, ABD) was used for extraction of volatile compounds in black tea samples and the identification of the volatiles were determined by a gas chromatography mass spectrometry (GC-MS) system (Shimadzu GC-2010/QP-2010, Kyoto, Japan). Extraction and identification methods were given the following.

2.3.1.1 Extraction of the volatiles

Black tea sample (3±0.001 g) was poured into a sealed vial (15 ml) with an accuracy of ±0.001 g and it was added 10 µL internal standard ((2-metil-3-heptanon pentanoic acid) to it. The mixture allowed to equilibrate at 40 °C for 30 min. and then SPME fiber was inserted for adsorption into the vial for 30 min at 40 °C.

2.3.1.2 The operating conditions of GC-MS and identification of the volatiles

Gas chromatography-mass spectrometry was run in split mode (ratio 1:20). The fibre remained in the injector for 2 min at 250 °C and the flow rate of the helium carrier gas was1.0 ml/ min. during desorption. The volatile compounds were separated on a DB-Wax column (60 m x 0.25 mm x 0.25 mm; J&W Scientific, Folsom, CA, USA). The oven temperature was held at 40 °C for 2 min (desorption period), increased to 70 °C by increment at 5 °C/min and held for 1 min. Then The temperature was increased to 240 °C by increment at 10 °C/ min. for a total run time of 30 min. The MS was set to record at 33-450 atomic mass units (threshold 1000) at a sampling rate of 1.11 scans/ s.

Identification of volatiles was based on the comparison of the mass spectra of unknown compounds with those in the National Institute of Standards and Technology, and Wiley Registry of Mass Spectral Data mass spectral databases. GC Retention indices were determined using n-alkane series (C_{10}-C_{26}) as described Sulejmani et al., (2020) [27]. The relative concentration of a compound in tea sample was calculated as follows: the following equation (1):

$$C_i(\mu g/\ 100 \mu g\ tea) = \frac{C_{IS}}{A_{IS}} * A_{\ i} \quad (1)$$

where, C_i; concentration of compound C_{IS}; concentration of internal standard A_{IS}; peak area of internal standard $A_{\ i}$; peak area of compound.

2.3.3 Sensory analysis

Ranking test, one of the quality-quantity tests, was applied to the teas (O and NO samples) produced using the oxygenated and the non-oxygenated waters (each individually) by dynamic liquid extraction method. The eight panelists (5 female and 3 male) who love Turkish black tea consumption, the age range was between 20 and 25, were selected at the Food Engineering Department of KSU. Four groups were formed (each group consisted of two panelists). One group's sensory analysis evaluation time lasted 30 minutes, and the total analysis evaluation time of the panelists took two hours. The analysis of the samples produced using oxygenated water was performed two hours in the morning, and the analysis of the samples using non-oxygenated water was performed two hours in the afternoon. The panelists were asked to evaluate color, aroma, brightness, and odor characteristics on a score range from 1 (dislike extremely) to 10 (like extremely). O and NO samples with the highest score in total were determined. Then, “scoring difference-from-control” test, one of difference tests, was conducted to determine the differences between the teas (O and NO samples) produced by the dynamic liquid extraction process and the tea (R sample) brewing by the traditional process. R sample was presented to the panelists; O and NO samples that received the highest total scores in the ranking test were asked to compare with R sample. The teas were scored

on a twenty-point scale for the color, aroma, brightness, and odor properties. All sensory properties of R sample were accepted as 20 points each [28, 29].

2.3.5 Statistical analyses

Statistical analyzes were carried out with the SPSS program (version 27). Variance analyzes were performed as one-way ANOVA and general linear model (GLM). Significant differences between the samples at each time and the times of each sample were determined by the Duncan multiple comparison test. Differences were considered significant at the $p<0.05$ level [30].

3 RESULTS AND DISCUSSION

3.1 Volatile compounds

A total of 35 components, including 14 aldehydes, 11 alcohols, 7 ketones, 2 aliphatic hydrocarbons (AHC) and 1 acid group, were determined in black tea samples. Although all 35 components were detected in O and NO samples, a total of 33 components were detected in the R. The number of volatile components varies greatly depending on the tea type and brewing conditions (temperature, quantity, tea type, etc.). Chen et al., (2022) identified a total of 55 volatile compounds in 44 types of black tea, including 10 esters, 9 aldehydes, 9 alcohols, 8 alkenes, 7 heterocyclics, 5 ketones, 5 alkanes and 2 ethers [31], but it was identified a total of 110 volatile compounds in Keemun black tea, including 16 aldehydes, 37 hydrocarbons, 18 alcohols, 9 heterocyclic compounds, 13 ketones, 16 esters and 1 carboxylic acid group by Su et al., (2022) [32]. In another study, a total of 157 volatile compounds were identified, including alcohol, ester, ketone, aldehyde, aliphatic hydrocarbon, aromatic hydrocarbon, acid, terpene, furan and sulfur group [33].

3.1.1 Aldehydes group

The concentrations of aldehyde group compounds in the teas are given in Table 1. The compounds with the highest and the lowest concentration were benzaldehyde (159.08 µg/g) in O sample at 120s and 3-methylbutanal (2.09µg/g) in NO sample at 30s. The two compounds with the highest concentration after benzaldehyde were hexanal, and 2-hexanal, (E)-. 2,4 hexadienal, furfural compounds identified in O and NO samples were not detected in R sample. Butanal, 2-methyl-, butanal, 3-methylbutanal, hexanal, octanal, safranal, (Z)-citral, benzaldehyde compounds in O and NO samples at all times were detected and also in R sample. The concentrations of butanal, 2-methyl-, butanal, 3-methyl-, 2-hexanal, (E)-, nonanal, (Z)-citral, benzaldehyde compounds in O, NO and R samples were different from each other, only the concentration of octanal compound in O sample at 60s was the same as that in R sample ($p<0.05$).

The number and concentrations of aldehyde group components vary widely due to reasons such as the tea growing region, tea processing process, tea size, brewing method, time, temperature and type of water used in brewing [34-36].

Chen et al., (2022) determined that the compounds with the highest concentration were 2-methylbutanal (23.21%), 2-methylpropanal (17.12%) and 3-methylbutanal (16.97%) among the aldehyde group compounds in black teas brewed at different brewing volumes, incubation times and temperatures but 3-methylbutanal had the lowest concentration in the present study [31]. Higher concentrations of heptanal, (Z)-4-heptenal, 2-hexenal, (E, E)-2,4-heptadienal, (E, E)-2,4-hexadienal, and (E)-2-octenal may be favorable to maintain the freshness of black tea [37].

Total concentrations of aldehyde group compounds in the teas are given in Figure 2. A fluctuating result was obtained in total concentration values depending on time. The highest (415.23 µg/g) and the lowest (213.24 µg/g) values were detected in O samples at 120 and 90s. and it was 329.05 µg/g in R sample. The total aldehyde concentrations in all O samples (except for 60 and 120 seconds), and NO samples were determined to be lower than the R sample. Statistically, the total aldehyde concentrations in O and NO samples in each time were different from each other and from R sample ($p<0.05$). Liu et al., (2021) reported that the total content of aldehydes decreased with increasing brewing temperature and that the main aldehydes were hexanal, benzaldehyde and (E)-2-hexenal, which have herbal and flower aromas [33]. Meng et al., (2021) stated that common and important volatile substances in tea, floral-scented compounds such as benzene acetaldehyde, contribute to the fresh and sweet aroma [38]. In addition, Kang et al., (2019) determined that the benzene acetaldehyde compound provides the strongest aroma with a pleasant smell in some black teas [39].

Table 1. Aldehyde group component concentration (µg/g) values of R, O and OS samples

t	S	Components*					
		1	2	3	4	5	6
30	O	3.35 $^{bX+}$	2.43 $^{aX+}$	26.59 ^{aX}	52.37 $^{bY+}$	13.94 ^{eY}	99.85 ^{dY}
	NO	2.73 ^{aX}	2.09 ^{aX}	26.26 ^{aX}	48.22 ^{bY}	4.58 ^{aX}	12.99 ^{bX}
60	O	6.59 ^{dY}	5.69 ^{dY}	42.41 $^{cX+}$	28.79 ^{aY}	8.68 ^{dZ}	27.32 ^{cY}
	NO	3.08 ^{bX}	2.31 ^{bX}	27.32 ^{bX}	50.10 $^{cY+}$	6.18 ^{cX}	9.65 ^{aX}
90	O	3.76 $^{cX+}$	2.60 ^{bX}	27.06 ^{aX}	57.08 ^{cY}	5.46 $^{aX+}$	9.38 ^{aX}
	NO	3.47 ^{cX}	2.74 $^{cX+}$	29.79 $^{cX+}$	60.74 $^{dY+}$	4.68 ^{aX}	nd
120	O	7.17 $^{eY+}$	5.95 ^{eY}	43.84 $^{dX+}$	87.76 ^{dY}	5.85 ^{bX}	19.42 ^{bY}
	NO	5.63 ^{dY}	4.84 ^{dX}	32.34 ^{dX}	nd	6.88 $^{dX+}$	nd
150	O	3.17 ^{aX}	2.72 ^{cX}	28.32 ^{bX}	28.02 ^{aY}	6.08 $^{eX+}$	19.07 $^{bY+}$
	NO	5.67 ^{dY}	5.57 ^{eY}	37.99 $^{eX+}$	4.07 ^{aX}	5.74 ^{bX}	17.89 ^{cY}
1200	R	5.48 ^{Z}	5.33 ^{Z}	101.46 ^{Z}	5.89 ^{Z}	8.81 ^{Z}	16.23 ^{Z}
		7	8	9	10	11	12
30	O	3.92 $^{cY+}$	nd	9.76 $^{bX+}$	3.91 ^{aX}	5.12 ^{c}	77.43 ^{aX}
	NO	2.20 ^{aY}	5.77 ^{aX}	8.13 ^{aX}	7.98 $^{cX+}$	nd	84.44 $^{aX+}$
60	O	5.41 $^{eY+}$	14.74 ^{bY}	14.81 ^{dY}	8.90 $^{dX+}$	8.39 $^{dY+}$	158.60 ^{dY}
	NO	4.19 ^{cY}	nd	9.76 ^{bX}	6.35 ^{aX}	2.83 ^{aY}	82.43 ^{aX}
90	O	2.21 ^{aY}	nd	9.95 $^{bX+}$	4.72 ^{bX}	3.44 ^{bY}	87.65 ^{cX}
	NO	2.60 ^{bY}	6.13 ^{bX}	8.15 ^{aX}	6.52 $^{bX+}$	5.00 $^{bY+}$	95.95 $^{bX+}$
120	O	4.88 ^{dY}	18.08 ^{cY}	14.17 $^{cX+}$	10.02 $^{eX+}$	8.50 $^{dY+}$	159.08 $^{dY+}$
	NO	5.87 $^{eY+}$	8.57 ^{cX}	13.69 ^{cX}	9.76 ^{dX}	5.98 ^{cY}	123.70 ^{cY}
150	O	3.12 ^{bY}	7.81 ^{aX}	7.88 ^{aX}	8.14 $^{cX+}$	3.29 ^{aY}	81.21 ^{bX}
	NO	4.42 $^{dY+}$	11.48 ^{dY}	15.89 $^{dX+}$	6.40 ^{abX}	8.51 $^{dY+}$	152.87 ^{dY}
1200	R	nd	13.63 ^{Z}	16.79 ^{Z}	11.97 ^{Z}	nd	118.32 ^{Z}
		13	14				
30	O	nd	nd				
	NO	10.28 $^{aX+}$	2.45 ^{aX}				
60	O	12.60 $^{aX+}$	nd				
	NO	10.48 ^{aX}	2.76 ^{cX}				
90	O	nd	nd				
	NO	11.20 $^{bX+}$	2.59 ^{bX}				
120	O	27.68 ^{bY}	2.86 ^{dX}				
	NO	15.36 ^{cX}	nd				
150	O	12.51 ^{aX}	2.58 ^{bX}				
	NO	22.22 ^{dY}	nd				
1200	R	19.70 ^{Z}	5.46 ^{Z}				

*1) Butanal, 2-Methyl-, 2) Butanal, 3-Methyl-, 3) Hexanal, 4) 2-Hexenal, (E)-, 5) Octanal, 6) Nonanal, 7) 2,4 Hexadienal, Trans- ,Trans -, 8) Benzeneacetaldehyde, 9) Safranal, 10) (Z)-Citral, 11) Furfural, 12) Benzaldehyde, 13) 2,4-Heptadienal, (E,E)-, 14) Pentanal

t: time (s), S: sample, R: the tea prepared using non-oxygenated water by the classical tea brewing process; accepted as reference sample, nd: not detected. O and OS refer the teas prepared using oxygenated and non-oxygenated waters by the dynamic tea extraction proceses.

"a-e" series show the differences between the times for each sample, and "X<X+<Z<Y<Y+" sorting indicates the triple comparison between the tea samples (O and OS) extracted by dynamic processes at each time with reference sample (R), statistically ($p<0.05$).

3.1.2 Alcohol group

Alcohols were the second most important volatile compounds in the teas. The concentrations of alcohol group compounds are given in Table 2. The major and minor alcohols in the samples were linalool (56.18 µg/g in R sample) and geraniol (2.22 µg/g in NO sample at 30 s).

1-Pentanol, 1-hexanol, 2-ethylhexanol, linalool, trans-linalool oxide, 1-octanol, 1-nonanol, benzeneethanol, benzenemethanol were detected in all teas. Concentrations of linalool and nerol in R sample were higher than all O and NO samples ($p<0.05$).

Table 2. Alcohol group component concentration (μg/g) values

t	S	Components *					
		1	2	3	4	5	6
30	O	6.36^{bY+}	27.34^{aY}	23.75^{aX+}	32.79^{aX}	8.85^{bX}	20.84^{cX+}
	NO	5.26^{bY}	3.03^{X}	21.36^{aX}	33.79^{aX+}	12.44^{bX+}	7.09^{aX}
60	O	13.03^{cY}	4.36^{cX}	33.43^{cZ}	49.22^{cX+}	25.29^{eY}	26.43^{eZ}
	NO	4.54^{aX}	4.46^{X}	24.86^{cX}	36.09^{bX}	9.92^{aX}	13.89^{bX}
90	O	6.50^{bY+}	nd	24.71^{bX+}	36.98^{bX}	10.71^{cX}	15.61^{aX}
	NO	5.92^{cY}	nd	22.28^{bX}	40.36^{cX+}	14.92^{cX+}	16.28^{cX+}
120	O	14.86^{dY+}	nd	36.90^{dY}	49.59^{cX+}	5.32^{aX}	23.73^{dX+}
	NO	7.21^{dY}	4.31^{bX}	32.47^{dX}	45.11^{dX}	19.87^{dY}	22.51^{dX}
150	O	4.28^{aX}	nd	24.66^{bX}	32.35^{aX}	11.79^{dX}	16.53^{bX}
	NO	9.69^{eY}	5.71^{dX}	37.37^{eY}	52.86^{eX+}	26.81^{eY}	29.99^{eY}
1200	R	5.07^{Z}	6.23^{Z}	33.47^{Z}	56.18^{Z}	15.40^{Z}	26.01^{Z}

t	S	7	8	9	10	11
30	O	24.98^{aX+}	6.87^{bY}	13.68^{aX}	6.89^{aX}	8.62^{aX}
	NO	2.22^{aX}	4.65^{bX}	13.64^{aX}	8.89^{bY}	14.11^{bY}
60	O	54.53^{cY}	7.11^{bX+}	60.95^{dY+}	19.79^{dY}	24.13^{dY+}
	NO	31.80^{dX}	5.89^{cX}	17.99^{bY}	8.46^{aZ}	13.42^{aY}
90	O	26.41^{bX+}	5.86^{aX+}	15.82^{bX}	8.21^{bX}	12.76^{cZ}
	NO	2.86^{bX}	4.34^{aX}	21.77^{cY}	9.23^{cY}	15.53^{cY}
120	O	nd	10.52^{cX+}	50.18^{c}	18.11^{cY+}	31.52^{eY+}
	NO	39.95^{eX+}	8.37^{dX}	nd	12.42^{dY}	23.03^{dY}
150	O	nd	28.35^{dY+}	14.29^{aX}	7.93^{bX}	10.64^{bX}
	NO	29.32^{cX}	12.12^{eY}	24.15^{dY}	14.40^{eY}	26.56^{eY}
1200	R	48.28^{Z}	11.00^{Z}	16.44^{Z}	8.46^{Z}	12.77^{Z}

*1) 1-Pentanol, 2) 1-Hexanol, 3) 1-Hexanol, 2-ethyl-, 4) Linalool, 5) Trans-linalool Oxide, 6) 1-Octanol, 7) Geraniol, 8) 1-Nonanol, 9) 3-Hexen-1-ol, (Z)-, 10) Benzeneeethanol, 11) Benzenemethanol

t: time (s), S: sample, R: the tea prepared using non-oxygenated water by the classical tea brewing process; accepted as reference sample, nd: not detected. O and OS refer the teas prepared using oxygenated and non-oxygenated waters by the dynamic tea extraction proceses.

"a-e" series show the differences between the times for each sample, and "X<X+<Z<Y<Y+" sorting indicates the triple comparison between the tea samples (O and OS) extracted by dynamic processes at each time with reference sample (R), statistically ($p<0.05$).

Total concentrations of alcohol group compounds in the teas are given in Figure 3. The total varied from 150.78 to 318.17 μg/g for O samples, 126.47-268.95 μg/g for NO samples, and was 239.28 μg/g for R sample. In all times, the totals in O and NO samples were different from each other, and not different between R sample and O sample at 120s ($p<0.05$). Similarly, Chen et al., (2022) and Liu et al., (2021) found that the linalool compound to be the highest [31, 33]. Geraniol and cis linalool oxide compounds provide the pleasant smell and strongest aroma in all teas [39, 40].

3.1.3 Ketones, AHC's and acid groups

Table 3 shows the concentrations of ketone (compound 1-7), AHC (compound 8-9) and acid (compound 10) group compounds. 2(4H)-benzofuranone, had the lowest concentration (4.61 µg/g in O sample at 30s).

Table 3. Ketone group component concentration (µg/g) values

t	S	Components*						
		1	2	3	4	5	6	7
30	O	19.79 ^{cY}	24.89 ^{bX}	20.41 ^{aX}	12.23 ^{aX}	18.90 ^{aX}	4.61 ^{aX}	21.73 ^{cY}
	OS	18.97 ^{bX}	29.59 ^{xX}	33.04 $^{aX+}$	14.92 $^{aX+}$	23.18 $^{bX+}$	7.40 ^{cY}	14.99 ^{eX}
60	O	27.53 ^{eY}	35.46 $^{dX+}$	48.26 $^{dX+}$	26.94 ^{cY}	37.96 ^{eY}	16.37 $^{eY+}$	nd
	OS	18.13 ^{aX}	24.93 ^{aX}	33.46 ^{aX}	15.34 ^{bX}	21.51 ^{aX}	4.80 ^{aX}	10.06 ^{bX}
90	O	23.51 ^{dY}	23.64 ^{aX}	36.66 ^{cX}	13.60 ^{bX}	19.51 ^{bX}	5.35 $^{bX+}$	18.57 ^{bY}
	OS	18.74 ^{bX}	28.08 ^{bX}	36.00 ^{bX}	16.19 $^{bX+}$	26.65 $^{cX+}$	6.27 ^{bY}	14.45 ^{dX}
120	O	13.07 ^{aX}	26.15 ^{cX}	48.41 ^{dX}	30.93 ^{dY}	36.66 ^{dZ}	12.74 $^{dY+}$	25.68 $^{dY+}$
	OS	22.00 ^{cY}	34.16 $^{dX+}$	47.23 ^{cX}	22.14 ^{dX}	30.27 $^{dX+}$	9.82 ^{dY}	9.37 ^{aX}
150	O	19.21 ^{bX}	26.36 ^{cX}	29.44 ^{bX}	13.69 ^{bX}	21.45 ^{cX}	6.24 ^{cZ}	12.84 ^{aX}
	OS	nd	44.13 $^{eX+}$	52.65 $^{dX+}$	27.15 ^{eY}	40.01 ^{eY}	11.53 $^{eY+}$	11.11 ^{cX}
1200	R	44.63 ^{Z}	51.93 ^{Z}	59.68 ^{Z}	24.10 ^{Z}	36.54 ^{Z}	6.83 ^{Z}	16.96 ^{Z}

* 1) alpha.Ionene, 2) Beta-ionene, 3) 6-Methyl-5-hepten-2-one, 4) 6-Methyl-3.5-heptadien-2-one, 5) 3.5-Octadien-2-one, (E,E)-, 6) 2(4H)-Benzofuranone, 7) 2-Heptanone

t: time (s), S: sample, R: the tea prepared using non-oxygenated water by the classical tea brewing process; accepted as reference sample, nd: not detected. O and OS refer the teas prepared using oxygenated and non-oxygenated waters by the dynamic tea extraction proceses.

"a-e" series show the differences between the times for each sample, and "X<X+<Z<Y<Y+" sorting indicates the triple comparison between the tea samples (O and OS) extracted by dynamic processes at each time with reference sample (R), statistically ($p<0.05$).

The compound with the highest concentration was 6-methyl-5-hepten-2-one (59.68 µg/g) identificated in R sample. The other two compounds with the highest concentration were determined as α-ionone and β-ionone. The concentrations of those three compounds in the R sample were higher than all O and NO samples. Also, 2-heptanone and α-ionone could not be detected at 60. s of O sample and 150. s of NO sample, respectively. And it was determined that the concentrations of all ketone compounds in R were statistically higher than those in the O and NO samples; except 3,5-octadien-2-one and 2(4H)-benzofuranone compounds in O sample at 120s and NO sample at 150s, respectively ($p<0.05$). β-ionone, one of the main volatile compounds, has the highest ACI (aroma character effect) value as strong odorants [40-42]. α-ionone (clove) contributes to the strong floral, caramel-like and moderate honey-like odors of black tea, which has a strong aroma intensity and a moderate effect of odor activity in different harvest seasons [33, 43, 44]. Total concentrations of the ketone groups in all samples are given in Table 4. The total concentration in the R sample is 240.63 µg/g, it varied between 122.52-193.60 µg/g and 128.21-186.56 µg/g in the O and NO samples. It was found that there were significant differences between R and other samples ($p<0.05$).

It was identified two compounds (decane and dodecane) from AHC group in the teas. Decane could not be detected in NO samples at 120 and 150s and dodecane in O sample at 120s. Decane had the highest concentration (39.19 µg/g) at 30. seconds of NO sample, and dodecane (68.72 µg/g) in the R sample. Statistically, as there was statistically no difference between NO sample at 90th second and R sample for concentration of decane, at other times O and NO samples were different from each other and from the R sample. it was determined that the concentrations of decane in O sample and dodecane in NO sample were significant different during all times ($p<0.05$). Total concentrations of the compounds in AHC group are given in Table 4. The total concentration in the R sample was 102.96 µg/g, and it was in the range of 27.82-105.44 µg/g in the O and NO samples. The totals in O and NO samples at each time were different from each other and from R sample as well ($p<0.05$).

Table 4. Aliphatic hydrocarbon and acid group component concentration (µg/g) values

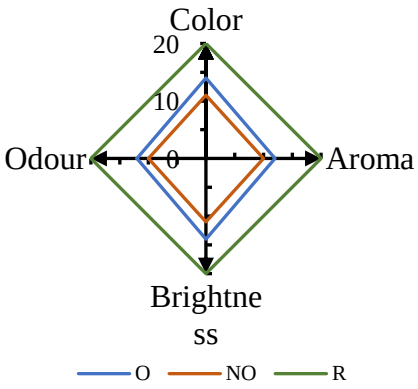
t	S	Components *		
		1	2	3
30	O	30.24 ^{cX}	nd	15.69 ^{bX}
	NO	39.19 ^{cY}	66.25 $^{dX+}$	16.47 $^{bX+}$
60	O	20.85 ^{aX}	28.06 ^{aX}	25.54 ^{dZ}
	NO	36.61 ^{bY}	56.25 $^{bX+}$	15.00 ^{aX}
90	O	38.33 ^{dY}	66.12 $^{dX+}$	14.73 ^{aX}
	NO	34.50 ^{aZ}	59.89 ^{cX}	17.31 $^{cX+}$
120	O	21.59 ^{bX}	61.57 ^{cX}	22.40 $^{cX+}$
	NO	nd	78.52 ^{eY}	22.10 $^{dX+}$
150	O	38.96 ^{eY}	56.73 $^{bX+}$	14.98 ^{aX}
	NO	nd	27.83 ^{aX}	25.61 ^{eZ}
1200	R	34.24 ^{Z}	68.72 ^{Z}	25.93 ^{Z}

*1) Decane, 2) Dodecane, 3) Methyl salicylate

t: time (s), S: sample, R: the tea prepared using non-oxygenated water by the classical tea brewing process; accepted as reference sample, nd: not detected. O and OS refer the teas prepared using oxygenated and non-oxygenated waters by the dynamic tea extraction proceses.

"a-e" series show the differences between the times for each sample, and "X<X+<Z<Y<Y+" sorting indicates the triple comparison between the tea samples (O and OS) extracted by dynamic processes at each time with reference sample (R), statistically ($p<0.05$).

Methyl salicylate acid is the only compound in the acid group and was determined in all tea samples. Its highest concentration (25.93 µg/g) was detected in the R sample. But there were no differences between O sample at 60s and R sample and between NO sample at 150s and R sample. Moreover, the concentrations of methyl salicylate acid in NO samples shown different at all times ($p<0.05$). It is one of the main aromatic compounds found in high amounts in black teas [31, 32, 40, 42].



	O	NO	R
Color	Y	X	Z
Aroma	XY	X	Z
Brightness	Y	X	Z
Odor	XY	X	Z

"X<XY (closed to X)<Y<Z" sequence shows the differences between the samples, statistically ($p<0.05$).

R: black tea prepared with the classical tea brewing process (Reference sample), O: black tea prepared using oxygenated water by the dynamic tea extraction process, NO: black tea prepared using non-oxygenated water by the dynamic tea extraction process

Figure 2. The sensory attributes of the black teas

3.2 Sensory analysis

In the ranking test applied to the teas (O and NO samples) produced using the oxygenated and the non-oxygenated waters (each separately) with the dynamic liquid extraction method, it was determined that the best samples were

the samples at time120s (not shown data). In "scoring difference-from-control" test conducted to determine the differences between the best teas determined in the ranking test and the tea (R sample) brewing by the traditional process, O sample received the highest scores compared to NO sample but fell 6-8 points behind the R sample in each attribute (color, brightness, odor and aroma). The results are given in Figure 2. Statistically, the color and brightness values of O and NO samples were different from each other and from R. The aroma and odor values of O and NO teas were similar to each other but different from R. It was determined that the oxygenated water was more effective in color and brightness than aroma and odor ($p<0.05$).

4 CONCLUSION

A food process was designed to obtain ready-to-drink Turkish black tea and shorten brewing times. In the process, oxygenated and non-oxygenated waters were used as solvents and the effects on tea quality were investigated. By sensory and volatile component analysis, it was determined how close the teas produced with the designed process were to Turkish black tea brewed with the traditional method. The use of oxygenated water in tea brewing has not been encountered in previous studies. Oxygenated water was not used in the classical method, because the presence of dissolved oxygen in water cannot be mentioned at a temperature of approximately 85°C. However, as in the present process, it must be used in closed-loop systems, or a different process must be applied for open systems. A positive correlation was observed between sensory and volatiles analysis. It was seen in the sensory analysis that the use of oxygenated water compared to non-oxygenated water was more effective although the difference between the waters was small. The data were obtained well below the brewing time of the classical method. The study was carried out by keeping pure Turkish tea type, size, temperature, and tea-water ratio constant. In the future, studies on these parameters can be carried out to make the system more efficient or it can be studied on blending with teas having a short brewing time such as Ceylon tea to reduce extraction time.

Author Contributions

Kübra DOĞRU: Conceptualization, Methodology, Software, Validation, Formal analysis, Investigation, Resources, Data curation, Writing - Original Draft, Writing - Review & Editing, Visualization, Supervision, Project administration, Funding acquisiton

Ahmet Levent İNANÇ: Conceptualization, Methodology, Software, Validation, Formal analysis, Investigation, Resources, Data curation, Writing - Original Draft, Writing - Review & Editing, Visualization, Supervision, Project administration, Funding acquisiton

All authors read and approved the final manuscript.

Conflict of interest

No conflict of interest was declared by the authors.

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