International Journal of Food Science and Nutrition
www.foodsciencejournal.com
ISSN: 2455-4898
Received: 20-05-2024, Accepted: 10-06-2024, Published: 01-07-2024
Volume 9, Issue 3, 2024, Page No. 1-6

Analysis methods of coffee volatile compounds

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Abstract
Coffee is one of the types of beverages that are most consumed around the world. During the processing of coffee, from roasting to extraction, a wide range of chemical reactions occur that affect the color as well as the flavor and aroma. As a result, several efforts have been made to comprehend the nature of this intriguing issue. This review used an integrated approach to research in order to give readers a comprehensive understanding of the widely used coffee aroma analysis techniques. This review was intended to serve as a foundation for subsequent application in the beverage sector, particularly in Vietnam. It did so by offering useful information regarding qualitative and quantitative research in terms of coffee scent.

Keywords: Coffee aroma, analytical methods, applications

Introduction

Coffee is one of the most important commodities that is only placed after petroleum and it is widely sold in the world throughout the years in many forms, including green, roasted, instant, and ready-to-drink coffee, thus providing an important source of income for people who are mainly from developing countries. Although there has been a decrease in sales in 2020 and 2021, due to the COVID-19 pandemic, the coffee market is predicted to increase in the future. The main reasons for the growth of coffee beverages not only depend on the psychological effects of coffee, including the feelings of relaxation, calmness, and ease of drowsiness [1] but also lie in the flavors of the coffee, which are mainly taste and aroma. However, the aroma is believed to be more important in perceived sensory characteristics, thus, affecting overall acceptance of the coffee.

Aroma has been the subject of studies for approximately a hundred years. First, the aroma-detecting mechanism in the body involves chemical detection in the olfactory receptors in the nasal cavity, which eventually provides electrochemical signals to the aroma-sensing area in the brain. From understanding the reason behind the smelling perception, researchers have developed many methods along with equipment that mimics the smell perception and thus, use them for the analysis of aroma – known as volatile organic compounds (VOCs). Until now, there have been more than 7000 VOCs found in food in general, and more than 1000 volatile compounds contribute to the unique flavor of coffee [2]. With the development of technology, many researchers have introduced innovative ways to overcome problems that are found in the old methods, which involve problems with efficiency, extraction, sensitivity, price, time, etc. Furthermore, many studies solved the long-term question of the correlation between coffee volatile compounds and their sensory characteristics; however, those studies only provided a small amount of information about the whole picture of the unpredictable nature of VOCs. Therefore, this paper will summarize the use of popular analytical instruments and identify volatile chemicals.

Analytical techniques of coffee volatile compounds

Comprehensive studies of coffee aroma are formidable tasks due to the unpredictable nature of VOCs. There has been much research on coffee aroma, but the overall picture is not yet fully described. Over the past 100 years, many analytical methods and instruments were developed to overcome these problems in coffee analysis. Depending on the analytical purpose, the selected approach can benefit the overall result of the research on coffee aroma by tackling the unpredictable nature of VOCs for extraction, detection, and characterization. Since most of the analytical methods were built based on the human odor-sensing mechanism in the body, 3 most popular methods used for aroma analysis were developed, including Gas Chromatography-based methods, Electric Nose, and analysis using Proton Transfer Reaction – Mass Spectrometry (PTR-MS) [3]. This part will focus on reviewing the use of those analytical methods of coffee VOCs.

1. Analysis of coffee volatile compounds using GC-based techniques

To be sensed in the human body, coffee aroma compounds must be ingested, then they react with olfactory receptors and be detected in the brain. Similarly, in coffee aroma analysis, the VOCs must be collected in either the vapor, liquid or solid form of the coffee, then the collected VOCs from the sample are brought to the GC column for separation and eventually, the VOCs are detected by the detectors and interpreted on the computer in the form of the chromatogram.

2.1 Coffee volatile extraction techniques

Coffee aroma isolation is the first step that takes advantage of the solubility and volatility of the VOCs. Aroma compounds have different thermodynamic properties, and different reactions with the coffee matrix and solvent; therefore, several approaches used to isolate substances in a coffee sample were developed. The most popular methods are Solvent-assisted extraction (SAE) Static Headspace (SHS), Dynamic Headspace (DHS), Headspace-Microextraction (HS-SPME), and Sorptive Extraction with

Headspace Sorptive Extraction (HSSE) and Stir Bar Sorptive Extraction (SBSE).

a. Solvent-assisted extraction (SAE)

Many methods utilized solvents to extract the VOCs in food in general, including supercritical fluid extraction (SFE), pressurized-fluid extraction, Soxhlet extraction, solvent-assisted flavor evaporation (SAFE), etc [4]. Overall, those methods commonly utilize the mass transfer properties, matrix effects, and solubility of VOCs [5] to extract the sample using a suitable solvent, either polar or nonpolar, then followed up by the dehydration and evaporation using N2[6]. The sample containing VOCs is directly injected into the GC column for separation and further analysis. Research about coffee VOCs using different organic solvents was reported. For example, either dichloromethane or hexane is used in Soxhlet extraction; tertiary butyl methyl ether solvent [7], methylene chloride or pentane was suggested in liquid-liquid extraction of coffee brew, and supercritical CO2 was used in Supercritical Fluid Extraction method. Solvent extraction methods can extract either volatile or semi-volatile compounds; however, they usually harm the extracted sample since the evaporation step can degrade the VOCs or even make new VOCs through a chemical reaction [8].

b. Static headspace extraction (SHS)

It is one of the popular methods used in aroma analysis due to the robust and non-selective characteristics during VOCs isolation. Compared to other headspace sampling methods, SHS does not require the use of solid-phase materials, solvents, or sorbents, which means that this method can utilize all the VOCs [9, 10], and it highly resembles the coffee ingestion process in the human body. Generally, the process of SHS sampling involves a sample placed in a vial with complete isolation, in which a relatively high temperature is applied to evaporate the VOCs in the vial, which occurs in 30-60 minutes to reach equilibrium, then the sample is taken out for injection into GC column [11]. There have been many researches related to the utilization of SHS, including aroma analysis and differentiation of distinct compounds in Black Ivory coffee, in which had successfully identified the key aroma components in elephant dung coffee in Black Ivory Coffee Company (Chiang Rai, Thailand) and Coffea Arabica L. in Chandanpur, Nepal. Furthermore, SHS was chosen for the observation of selected VOCs during the brew of espresso coffee using different temperatures [12]. However, the method is efficient only when the researcher wants to measure VOCs with high volatility since SHS has low sensitivity followed by the lack of extracted compounds in the sample.

c. Dynamic headspace (DHS)

The dynamic headspace method utilizes hydrophilic characteristics and low vapor pressure of volatile compounds. Due to this reason, DHS can efficiently extract the VOCs from food samples, and it is widely used for coffee aroma analysis. Using the inert gas, usually Nitrogen or Helium, it purges with a consistent flow rate into the sampling vial and the VOCs will evaporate and be trapped in the absorbent trap. The absorbent trap will later be desorbed with a thermal desorption unit (TDU), and VOCs from that will be injected into the GC column. An application of multi volatile-method (MVM) using sequential dynamic headspace sampling coupled with a fully evaporated technique (FET) was suggested as an innovation of DHS, which was considered a promising approach in coffee aroma sampling due to the utilization of different trap absorbents, with various purging volumes to evaporate the substances with low vapor pressure (VP < 0.000088). This method succeeded in the identification of 658 volatile compounds in Americano-style brewed Arabica coffee [13].

d. Headspace solid-phase microextraction (HS – SPME)

This is considered the most popular technique for coffee aroma analysis. The coffee brew is agitated using a temperature (50–60◦C) for a fixed time (5-30 minutes). After that, the fiber coating is inserted into the heated sample and VOCs from the coffee brew will migrate into the fiber coating until it reaches equilibrium in about 20-30 minutes. The fiber is then inserted into the GC inlet for desorption and then separation of coffee aroma in the GC column before detection by detectors [3]. The efficiency of HS-SPME mostly depends on the polarity of the fiber coatings, therefore many fiber coatings are developed, including Divinylbenzene, Carboxen, Polydimethylsiloxane, Carbowax, and Polyacrylate Fiber. However, the most sensitive and efficient fiber coating is the combination of Divinylbenzene-Carboxen-Polydimethylsiloxane (CAR/DVB/PDMS) due to high recoveries of analytes with variable structure and polarity [14]. HS-SPME is commonly used for the differentiation of coffee products and identification of coffee quality, due to having no requirement of solvent and high sensitivity. HS- SPME was suggested for the identification of aroma quality indicators in fermented coffee [15,16], volatile analysis of single bean [17], identification of defective indicators in coffee [18] and the difference between roasted Arabica and Robusta coffee. Despite the advantages of HS-SPME, the careful selection and handling of fiber coatings should be carried out since the coating fibers can strongly influence the aroma extraction.

e. Sorptive Extraction

Sorptive Extraction methods are emerging as a potential category of aroma-extracting techniques. Two main sorptive techniques are popularly used in coffee aroma extraction including stir bar sorptive extraction (SBSE) and headspace sorptive extraction (HSSE). These methods focus on the adsorption of VOCs in the sample using a polymer-coated magnetic stir bar. This approach is applied either in the liquid samples (SBSE) or in the headspace of the samples (HSSE). In Bicchi *et al*. study, sorptive extraction was proved to be superior in coffee VOCs recovery efficiency, due to higher sorptive phase volume, compared to conventional SHS, HS-SPME, and IS-SPME (In-sample SPME). These methods, however, only have two types of coating fibers: PDMS and EG-Silicone; therefore, limiting the detected volatile spectrum and resulting in the low recovery rate of polar molecules [19]. Further utilization of different materials for the sorptive material - polyurethane - was researched and the result was promising [20]; however, this area still needs to be further studied in the future.

1.2 Separation and detection of coffee volatile compounds

The coffee VOCs after being sampled will be inserted into a GC column for separation before analysis, in which 3 popular methods for coffee aroma analysis with Gas Chromatography include 1-dimensional GC (1D-GC), 2-dimensional GC (GCxGC) and GC coupled with Olfactometry (GC-O). In 1-dimensional GC, the columns are made from either polar or nonpolar materials. The most popular static phase polar columns are made from polyethylene glycols which are: DB-WAX, HP-FFAP, SolGel-WAX, SUPELCO WAX, and ZB-WAX while the poplar non-polar columns are DB-5, HP-1, and HP-5MS, which were also reported to have similar effects compared to polar ones [21]. Although 1-dimensional GC is a powerful tool for VOCs analysis, the data from the sample is sometimes too complex to understand on a conventional chromatogram, therefore, 2-dimensional GC is used to solve the problems. The application of GCxGC was found in studies of D. Ryan (2006) (BPX5 (1st column) and BP20 WAX (2nd column)) and in I.D. Fisk and others (2012) (Varian VF-5MS column (1st column) and Agilent DB-1701 column (2nd column)), utilized the combined polarities of different column types for separation [7,22]. The VOCs after going through the GC column will then be ionized and detected by detectors. Many types of detectors are used in the coffee analysis that includes: Flame Ionization Detector (FID), Mass Spectrometry Detector (MSD), and Olfactory Detector (OD) [3] and they are chosen based on their unique features such as FID is cheaper and more widely applied for target analysis of coffee VOCs that contain carbon-hydrogen bond [23], while MS is highly recommended for identification of overall VOCs in coffee aroma analysis through retention time and molecular mass spectrum, and OD is used to correlate coffee VOCs with their perceived sensory attributes [24]. In coffee aroma analysis, it is the most common to see the application of GC-MS, in which the VOCs after traveling in the column, will be ionized by an electron impact (EI) ionization mode, and the ions are detected in the Quadrupole Mass Spectrometer, which is a mass analyzer [25,26]. After the ionization, the recording system, is mainly scan mode or single/multiple ions monitoring modes (SIM/MIM) or ion extraction mode (IEM), will interpret the VOCs data on a chromatogram in form of the mass spectrum (MS) and retention indexes (RI) then compared them to the reference libraries (NIST, WILEY, etc.) for identification of the VOCs. Furthermore, the Quadrupole-Time-of-Flight Mass Spectrometry detector has been recently developed and preferred due to the rapidity, efficiency, and accuracy of the approach. This detection method is highly efficient when paired with GCxGC as it has in-time data provision with reliable, continual data which can solve the overlap of peaks based on 3-dimensional ion chromatograms [19]. A study suggested the use of the nitrogen-phosphorus detection method (NPD) coupled with GCxGC [22], which compared NPD to the ToF-MS and FID in coffee VOCs detection using GCxGC methods. The result showed that using GCxGC-NPD was more sensitive, effective, and reproducible than that of GCxGC-FID and it was also simpler in the characterization of Nitrogen-containing compounds compared to GCxGC-ToF- MS. However, the studies about GCxGC methods in coffee aroma analysis are still limited and they should be extensively studied in the future based on the advantages this method has. Finally, Olfactory Detector is used for the sensorial characterization of coffee VOCs by diverting the VOCs in the GC column, using humid air or inert gas, to the olfactometer for sensorial detection. In this analytical model, the coffee VOCs are divided by 2 parts after separation in the GC column, one is transferred to the sniffing port for qualitative analysis using a non-destructive thermal conductivity and one is transferred to the FID or MS for quantitative analysis [27]. The most popular olfactometer methods that are used to collect and process the data from the olfactometry are Aroma Extract Dilution Analysis (AEDA), Odor activity values (OAV), and Odor Spectrum Value (OSV) [28]. After this step, the collected data might be further processed to meet the requirement of the analysis, including differentiation, detection of quality indicators, etc.

2. Analysis of coffee volatile compounds using E-Nose

GC-based methods are powerful tools and suitable for quantitative analysis of roasted coffee aroma; however, they are usually expensive and laborious. Furthermore, future studies also require the characterization of the substances with their perceived sensory attributes. Therefore, researchers developed Electronic Nose to compensate for the weakness in coffee VOCs analysis, which is the characterization of the sensory attributes of VOCs [25,29,30]. At first, E-nose will detect and discriminate the aroma through statistical analysis of chemical resistance in the sample provided by Headspace sampling methods. The data is consequently used to study the correlation between physicochemical and sensory characteristics of coffee aroma through multivariate tools.

The coffee VOCs before being analyzed in the E-nose will be collected via headspace methods (SHS and SPME) [3]. The collected VOCs from the sample will be applied to the E-Nose sensor using a range of sensor types, including Metal Oxide Sensors (MOS), conducting polymer sensors, electrochemical sensors, and mass spectrometry sensors. Although many options for the sensor types are suggested, MOS is widely used for coffee aroma analysis due to its high sensitivity to detect a wide range of chemical compounds, fast recovery, low cost, and high reproducibility [31]. After the application of E-nose on the sample, the coffee VOCs database is recorded based on features including steady-state, transient parameters, and features from the phase space [32,33], and the recorded data then requires further analysis techniques to recognize the trends in the coffee sample (pattern recognition analysis), such as principal component analysis (PCA), linear discriminant analysis (LDA), machine learning (ML) including artificial neural networks (ANN), multilayer perceptron (MLP), etc. In the PCA method, it can transform the data from multidimensional into 2-dimension and gather highly related samples into groups to observe the correlation. This method is widely used as it simplifies the correlation, and reduces the work to make the data more understandable. LDA, on the other hand, searches for vectors that are unique for each VOC class and creates a linear combination of those vectors that have the largest mean differences between the classes. ML techniques, rather than finding similarities or differences, use statistical analysis for identifying patterns [34], which can recognize and correlate VOCs with their sensory attributes [29] or even predict the sensory characteristics of roasted coffee with ML method (support vector machine) and the deep convolutional neural network (DCNN) with an accuracy of up to 77% as YT Chang and others (2021) suggested [35]. Many researchers applied MOS to study the VOCs of coffee, as it was reported in the study of Romani and others (2012), 10 temperature-moderate MOS were used, along with ANN to develop an automatic roasting system and reproducible quality testing model [36]. Michishita and others (2010) also used E-nose in their study about the prediction of sensory descriptors using αFOX4000 E-nose equipped with 18 MOS sensors in 3 chambers with ANN techniques [29]. In addition, Wakhid and others (2020) also suggested the use of a similar e-nose along with ML statistical analysis to classify civet and non-civet coffee [30]. Furthermore, C. G. Viejo and others (2021) studied the assessment of coffee aroma profile and intensity, and also reported on the use of 9 different MOS coupled with ML and ANN techniques for statistical analysis [37]. As a result, it was claimed to be a reliable and effective tool in coffee aroma analysis, considering the low price of the E-nose and its portability.

3. Analysis of coffee volatile compounds using PTR-ToF-MS methods

Although GC-based analytical methods are a powerful tool for coffee aroma analysis, it is often time-consuming, and this resulted in the development of another approach that also utilizes Mass Spectrometry – known as Proton-transfer-reaction-mass spectrometry (PTR-MS). This method is basically Direct Injection MS (DI-MS) that has been popularly used for aroma analysis in recent years due to the dynamic and sensitive nature of the method. In this model, the reagent ion (namely protonated water) will first be ionized by an ion source (a hollow cathode discharge source) and transferred to a drift tube through electric fields. On the other hand, VOCs collected from the coffee will also be injected into the drift tube and the volatile compounds will react with the ions from the ion source. The drift tube is filled with buffer gas to control the reaction between two reagents. After that, the products from the reaction in the drift tube will be detected by the analyzers, including Quadrupole Mass Spectrometer (QMS) or Time-of-Flight-Mass Spectrometer (ToF-MS). While QMS identifies VOCs based on the mass number of an ion *m*, divided by its charge number, *z*, ToF-MS utilizes the traveling energy of an ion in a vacuum environment. The latter is reported with many applications in the coffee analytical field due to the advantages when paired with PTR-MS, such as: not requiring sample preparation, direct measure of VOCs without calibration, high sensitivity, and higher mass resolution analysis as well as in-time response [38,39]. This method can be applied in studies about changes during coffee roasting due to fast response as witnessed in A.N. Gloess and others (2012), PTR-ToF-MS was successfully used to demonstrate the correlation of time-temperature with VOCs during the roasting of different coffee species. Furthermore, PTR-ToF- MS was also suggested in the analysis of instant coffee aroma during reconstitution to measure the intensity and can further improve the quality of the product [40,41]. In addition, PTR-ToF-MS was also found in the investigation of the effects of water temperature and pressure on the VOCs during Espresso Coffee making and the results demonstrated the polar compounds were more affected by the temperature. Despite the advantages PTR-ToF-MS might have, the size of the equipment is one of its weaknesses since it cannot be used directly on the field. Furthermore, the lack of sensitivity towards the chemical isomers is also one of the reasons that limit the use of PTR-ToF-MS.

Conclusion and Recommendation

In conclusion, coffee scent has been a fascinating subject because of the continuous rise in consumption throughout the globe. In an effort to fully comprehend the nature of the scent of roasted coffee and apply it to linked businesses, several researchers have made an effort to examine the volatile molecules found in coffee. Because of this, numerous well-liked analytical methods were created to effectively study the aroma of coffee, both quantitatively (GC-based methods with various detectors) and qualitatively (Olfactometer, E-Nose), and as a result, the causes of variations in coffee were identified, including variations in coffee species and origins, coffee growing climates, coffee processing methods, coffee roasting, and preservation.

Future studies about coffee aroma may consider the improvement of analytical methods, which should focus on individual beans to reduce VOCs variation from the whole batch. Moreover, advanced artificial intelligence or computer learning algorithms should also be developed for qualitative analysis to reduce the variables in real-life sensory tests as people's senses can be subjective and easy to be fatigued. Several plans suggested for the analysis of coffee VOCs include isolation and extraction of volatile compounds that contribute most to the aroma of coffee and use them as a flavor enhancement in the food industry. Another approach in the future of coffee aroma analysis is the removal of undesirable VOCs resulting from defects during production as this can solve the manufacturing efficiency problems of coffee and benefit the industry. Finally, researchers can comprehensively study the quality indicators from every processing step, especially in the post-harvest steps, then apply the results to produce desirable coffee products. In Vietnam, although the studies about its coffee staple, Robusta, are still limited, this subject has great potential to discover considering that Robusta is one of the most common goods exported by Vietnam in the last decade and the application from studies of coffee can improve the quality; thus, increasing the sales of Vietnamese coffee staple, leading to the growth of the country.

Acknowdgement

We would like to express our special thanks to all co-authors for their helpful comments and guidance during preparation of this manuscript. We also appreciate everyone who involved directly and indirectly in this study.

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