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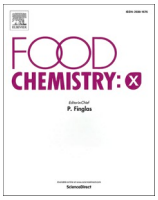

The impact of different drying methods on the physical properties, bioactive components, antioxidant capacity, volatile components and industrial application of coffee peel

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ABSTRACT

This study evaluated the effects of hot air drying (HAD), microwave drying (MD), vacuum drying (VD), sun drying (SD) and vacuum freeze drying (VFD) on the physical properties, bioactive components, antioxidant capacity, volatile components and industrial application of coffee peel. The results showed VFD could retain the appearance color, total phenolics (19.49 mg GAE/g DW), total flavonoids (9.65 mg CE/g DW), caffeine (3.15 mg/g DW), trigonelline (2.71 mg/g DW), and antioxidant capacities of fresh sample to the greatest extent, but its operating cost was significantly higher than other treatments and total volatile components were in the minimum levels. HAD and SD exhibited the highest loss rates of total phenols and antioxidant capacities, exceeding 50%. MD offered the lowest operating cost, superior retention of bioactive components, and the richest variety and quantity of volatile compounds. Therefore, it is recommended to use MD to dehydrate the coffee peel in actual production.

1. Introduction

Coffee (*Coffea arabica L.*), belonging to the Rubiaceae family, is the second most traded commodity after oil and one of the world's most popular beverages (Lee et al., 2023). In the 2020/21 crop year, more than 166 million 60 kg bags of coffee were consumed globally, and the scale of consumption has continued to grow at a rate of 1.3–4.4% per year over the last few years (Blumenthal et al., 2022). However, this consumption only represents coffee beans, which account for only 20% of the total weight of fresh coffee fruit. The remaining 80% corresponds to the by-products produced during coffee processing, such as coffee peel, silverskin, parchment and mucilage (Oliveira et al., 2021). Among these, coffee peel is the most important by-product of coffee harvesting and processing, accounting for approximately 45–55% of the whole fresh weight of the coffee fruit. Unfortunately, a substantial portion of the coffee peel is typically discarded without appropriate treatment, aside from a small portion used for composting or conversion into animal feed, which not only gives rise to severe environmental problems in coffee-growing countries but also undermines the sustainability of agricultural production (Blumenthal et al., 2022). Coffee peel has been demonstrated to be a safe food ingredient containing many high-value components (e.g., proteins, fibers, polysaccharides, bioactive components, and flavor compounds) (Gemechu, 2020). These components not only give coffee peel the potential to become a natural source of functional ingredients and nutrients for the industry but also confer antioxidant, anti-inflammatory, anti-diabetic, cholesterol-lowering, and antibacterial properties (Bondam, da Silveira, dos Santos, & Hoffmann, 2022; Duangjai et al., 2016). Nevertheless, the high moisture content (over 80% w.b.) of fresh coffee peel poses a tremendous challenge for the storage and transportation of the items, significantly limiting the widespread use of coffee peel (Thy Minh Kieu, Kirkman, Minh, & Quan Van, 2020). Thus, it is crucial to remove moisture from coffee peels through drying in the subsequent production and processing of high-value-added products.

Coffee peel is commonly dehydrated by sun drying (SD). However, SD is susceptible to weather conditions that can lead to inadequate drying or even corruption, resulting in lower product quality. In addition, SD is also prone to contamination by foreign substances like dust

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and debris, and coffee peel can come into contact with fungi, birds, and insects, leading to microbial growth and a decrease in flavor quality (Dong et al., 2017). As a result, researchers have developed other drying methods, including hot air drying (HAD), infrared drying, vacuum freeze drying (VFD), vacuum drying (VD) or microwave drying (MD). Additionally, unconventional methods such as ultrasonic, pulsed electric field drying, high-pressure assisted drying, and various drying methods have produced foods with higher nutritional and sensory attributes (Yu et al., 2022). Regrettably, initial investment and operating costs of these methods are very high, leading to their occasional use in large-scale industrial implementations.

Generally, the heat-sensitive components in plant raw materials undergo varying degrees of degradation and transformation during drying (Liu et al., 2021). Characteristics of drying products can also be influenced by drying treatments (Yu et al., 2022). Several studies have evaluated the physicochemical effects of drying on coffee peel. For example, compared with SD and HAD samples, VFD and VD treatments could contain high levels of total phenols, flavonoids, anthocyanidins and antioxidant activities (Jiamjariyatam et al., 2022; Thy Minh Kieu et al., 2020). Moreover, different drying techniques can also greatly affect the volatile compounds (Wang et al., 2022). Aroma is crucial, influencing the flavor and commercial appeal of agricultural products (Song et al., 2020). However, the references on coffee peel drying were minimal and mainly focused on researching total phenols, flavonoids, anthocyanidins, and antioxidant activities. There were few studies on the effects of different drying methods on the volatile compounds, primary flavors and industrial applications of coffee peel. How to select the appropriate drying technology for the characteristics of coffee peel to dry with low cost, low energy consumption, and high efficiency is an urgent problem.

Therefore, five drying techniques (HAD, SD, MD, VD, and VFD) were used in this study to analyze the effect on physical properties, bioactive components, antioxidant capacity, and volatile components of coffee peel. Furthermore, the relationship between operating costs and drying methods of coffee peel in industrial applications was assessed, and the current range of coffee peel applications was summarized. The overall aim was to provide drying technology options for the recycling and high-value utilization of coffee peel waste and determine the most efficient drying method for industrial production.

2. Materials and methods

2.1. Chemicals

Standards (gallic acid, catechin, caffeine, rutin, trigonelline, chlorogenic acid, neochlorogenic acid, cryptochlorogenic acid, protocatechuic acid and caffeic acid, and 2-octanol) were purchased from Aladdin (Shanghai, China). High-performance liquid chromatography (HPLC) grade acetonitrile and trifluoroacetic acid were purchased from Sigma-Aldrich (Milan, Italy). *N*-alkanes (C_3-C_{25}) were bought from O2si (South Carolina, USA). All other reagents were analytical grade and purchased from Aladdin (Shanghai, China) or Macklin (Shanghai, China).

2.2. Coffee peel samples

Fresh Arabica coffee fruits were harvested in March 2022 from an organic coffee production base in Lujiangba, Baoshan, Yunnan Province, Southwest China (25°4′N, 99°11′E; 799.50 m above sea level). The fresh coffee fruits were peeled manually after washing. Then, the fresh peel was immediately transported to the laboratory and frozen at −40 °C until drying experiments. The initial moisture content of the fresh peel was 83.14 ± 0.09% wet basis.

2.3. Drying experiments

The samples were defrosted at room temperature (19 ± 3 °C) before drying experiments. Coffee peel was selected without apparent signs of injury or disease, divided into 150 g each and then evenly placed on clean trays. The coffee peel samples were dried using five methods until the samples had a constant weight. The individual drying conditions were determined on the basis of the pre-experiments. For HAD, a hot air oven (DHG-9140B, Hangpei, Shanghai, China) was heated to 85 °C, and then the samples were placed in the oven and dried at 85 °C and 1 m/s air speed for 9.18 h. For MD, samples were put in a microwave oven (RWBZ-08S, Suenrui, Nanjing, China) and dried at 560 W for 2.52 h. VD was performed using a vacuum oven (DZF-6090, Hangpei, Shanghai, China) at 0.07 MPa and 90 °C for 9.18 h and the drying process was performed as HAD. For SD, coffee peels were spread evenly on a tray and dried under natural sunlight (Daily sun exposure time 8 h, Mid-day temperature 24 ± 2 °C, Southwest wind level 2, relative humidity 18 ± 4%) for 102.22 h. The VFD samples were pre-frozen at −60 °C and then dried by a freeze-dryer (FD-1B-50, Lanyi, Shanghai, China) for a total of 27.33 h, where the condenser temperature and chamber pressure were −60 °C and 10 pa, respectively. Each drying process was repeated 3 times. The dried coffee peel samples were vacuum-packed with an automatic vacuum packing machine (DZ-300/2SA, Ruixiang, Shanghai, China) to prevent moisture retention. The samples were kept at −20 °C until the following experiment. Selected parts of the coffee peel samples prepared by different drying methods were pulverized with a universal high-speed pulverizer (FW-80, Tianjin Test Instrument Co., Ltd., Tianjin, China), filtered through a 40-mesh sieve, and then packed in closed plastic bags, and stored at −40 °C for determination of color, bioactive components and volatile components.

2.4. Physical properties

2.4.1. Specific energy consumption (SEC) and yields

The specific energy consumption (*SEC*) is crucial in evaluating the efficiency of different drying methods. The energy consumption for drying a kilogram of fresh coffee peel was calculated according to equation (1) (Surendhar, Sivasubramanian, Vidhyeswari, & Deepanraj, 2019).

$$SEC = \frac{E}{M} \tag{1}$$

where *SEC*, E and M were the specific energy consumption (kWh/kg), energy consumption (kWh) obtained from an electric meter (kWh) and fresh coffee peel weight (kg), respectively.

The recorded variation of samples mass before and after drying was used to evaluate their yields in percentage following Mondal, Rangan, and Uppaluri (2019) reported work, equation (2).

$$Yield(\%) = \frac{M1}{M2} \tag{2}$$

where M_1 and M_2 were weight of the coffee peel before and after drying, respectively.

2.4.2. Color measurement

The color parameters (L, a, and b values) of fresh and dried samples were quantified using a colorimeter (WSC-S, Shanghai Physical Optical Instrument Co., Ltd, Shanghai, China). The color difference (ΔE) was then calculated using equation (3) (An et al., 2022).

$$\Delta E = \sqrt{(L^* - L)^2 + (b^* - b)^2 + (a^* - a)^2} \tag{3}$$

where L* = 91.30, a* = −0.92 and b* = 3.74 were the color values of the standard white plate.

2.4.3. Scanning electron microscopy (SEM)

The microstructure of the dried coffee peel was analyzed following the methodology described by Xu et al. (2020) with some modifications. The sample was broken into small pieces with tweezers and secured to the sample holder using double-sided tape. The sample was then sprayed with gold under a vacuum to make it conductive. These samples were observed by a scanning electron microscope (SEM, VEGA3, Tesken Co., Ltd., Prague, Czech Republic) at 500x magnification.

2.5. Determination of bioactive components

2.5.1. Preparation of extracts

The coffee peel extracts were prepared based on the method of An et al. (2016). 5 g of the powder samples were mixed with 100 mL of 80% methanol and extracted using an ultrasonic bath (CH-06 M, Weineng, Suzhou, China) for 30 min at room temperature with assistance. The extract was centrifuged at 8000 rpm for 10 min by a centrifuge (TGL-16ar, Feige, Shanghai, China), and the residue was re-extracted twice as described above. The supernatant from each round of extraction was collected using a rotary evaporator (RE-200B, Yarong Biotechnology, Shanghai, China), and the extracts were concentrated under vacuum at 40 °C. Then, the concentrates were stored in a brown glass bottle (500 mL) at 4 °C to determine total phenols, total flavonoids, antioxidant activity and individual phenolic compounds content.

2.5.2. Total phenolic and total flavonoid contents

The total phenolic content (TPC) assay was conducted according to the Folin-Ciocalteu reaction described by Chumroenphat, Somboonwatthanakul, Saensouk, and Siriamornpun (2021) with slight modifications. Briefly, Folin-Ciocalteu reagent (0.5 mL) and 10% Na_2CO_3 (1.5 mL) were added to the diluted sample extract (0.1 mL). The TPC of the sample was quantified by constructing standard curves with different concentrations of gallic acid at 760 nm. The total flavonoid content (TFC) was determined following the method of Sun et al. (2023). Initially, diluted sample extracts (1 mL) were combined with 5% $NaNO_2$ (0.15 mL), 10% $AlCl_3$ and 1 M NaOH (1 mL). The mixed solution was then left to stand in the dark for 5 min to complete the reaction. The TFC in the samples was quantified by generating a standard curve with different concentrations of catechin at 510 nm. TPC and TFC were calculated as equivalents of gallic acid and catechin per g of dry weight, expressed as mg GAE/g DW and mg CE/g DW, respectively.

2.5.3. Individual phenolic compounds

For quantitative analysis of individual phenolic compounds, extracts were filtered through a 0.22 μm regenerated cellulose membrane prior to HPLC analysis. Based on the method developed by Chen, Ma, and Kitts (2018), an Agilent 1260 instrument (Agilent Technologies Inc., Palo Alto, CA, USA) with a reversed-phase C18 column (100 mm × 2.1 mm, id, 1.7 μm particle size) was utilized. The mobile phase consisted of two components: solvent A (distilled water with 0.1% (v/v) trifluoroacetic acid) and solvent B (acetonitrile with 0.1% (v/v) trifluoroacetic acid). Gradient elution conditions were as follows: 0–10 min, 95–80% A; 10–13.5 min, 80% A; 13.5–18 min, 80–95% A; 18–21 min, 95–5% A; 21–23 min, 5–95% A; 23–25 min, 95% A. The mobile phase flow rate was 0.2 mL/min. The column temperature was 25 °C and the injection volume was 1.5 μL. The contents of caffeine, rutin, trigonelline, chlorogenic acid, neochlorogenic acid, cryptochlorogenic acid, protocatechuic acid and caffeic acid (Shanghai Aladdin Biochemical Technology Co., Ltd., China) were quantified by external standard method, respectively based on the dry weight.

2.6. Antioxidant activity assessment

The antioxidant activities of coffee peel were detected using DPPH, ABTS and FRAP kits (Jiancheng, Nanjing, China). The determination of three antioxidant activities was carried out according to the manufacturer's instructions. For DPPH free radical scavenging activity, diluted sample extracts of coffee peel (0.1 mL) were added to 4 mL of 0.14 m mol/l DPPH methanol solution, left to react for 30 min in the dark, followed by measurement of absorption at 517 nm. To measure ABTS free radical scavenging activity, diluted sample extracts (0.4 mL) were added to 3.6 mL of the ABTS solution, left to react for 30 min in the dark, followed by absorption measurement at 734 nm. For ferric reducing antioxidant power (FRAP), diluted sample extracts (0.1 mL) were mixed with the FRAP solution 4.0 mL for 15 min under 37 °C. The FRAP was measured by monitoring the absorbance at 593 nm. The standard curves of the three antioxidant activities were plotted with Trolox and the results were expressed as Trolox equivalents per g of coffee peel (dry weight) (mg TE/g DW).

2.7. Analysis of the volatile compounds

2.7.1. Extraction of volatile compounds by HS-SPME

Following to Yi et al. (2018) method with minor modifications, an extraction bottle (10 mL) containing the sample (2 g) was added to saturated sodium chloride solution (5 mL) and 2-octanol internal standard solution (50 μL, 0.415 mg/mL). The sample vials were preheated at 70 °C for 30 min using a solid-phase microextraction device, and then SPME fibers (50/30 μm CAR/PDMS/DVB; Supelco Co., PA, USA) were inserted into the headspace of the sample and extracted for 30 min.

2.7.2. Quantification of volatile components by GC–MS

For GC conditions, the inlet temperature was set to 250 °C, the carrier gas was helium (purity > 99.99%), and the flow rate was 1.0 mL/min. Volatile compounds were qualitatively and quantitatively determined using a GCMS-QP2010 gas chromatography-mass spectrometer equipped with a DB-WAX column (0.25 μm, 30.0 m × 250 μm; Agilent Co., CA, USA). The initial column temperature was 50 °C (5 min hold), then ramped to 150 °C at 5 °C/min (5 min hold), then 5 °C/min to 250 °C (5 min hold). The total runtime of the GC program was 45 min. For MS conditions, the electron ionization (EI) energy was 70 eV, the ion source temperature was 230 °C, the quadrupole temperature was 150 °C, and the mass scan range was 33–400 *m/z*. Preliminary identification of volatile compounds by comparing MS data using the NIST 5.0 database. The integrated report will be accepted if the match exceeds 80%. Identification confirmation was done by comparing the retention index (RI) to the RI of alkane standard solutions (C_3-C_{25}, Shanghai, China). The relative amount of each volatile compound was determined by calculating the ratio of the peak area to the peak area of the internal standard.

2.8. Economic assessment

The operating costs are analyzed based on a cycle time of 1 kg of fresh coffee peel dried to constant weight. These costs involve expenses for electricity consumption and equipment depreciation. The cost of electricity consumption is calculated based on the unit price of electricity for non-household customers in Kunming City, Yunnan Province, China, in the first half of 2022 (0.49 ¥/kWh). The depreciation cost of equipment is calculated based on the implementation regulations of the Enterprise Income Tax Law of the People's Republic of China.

2.9. Statistics analysis

All experiments were performed in three replications at least. Orthogonal partial least squares discriminant analysis (OPLS-DA) and variable importance in projection (VIP) were performed using SIMCA-P software (Version 14.1, Umetrics, Sweden). Histograms were plotted in Origin 2021 pro (Microcal Software, Inc., Northampton, NC, USA). Duncan test and one-way analysis of variance were conducted using SPSS (SPSS 24.0 for windows). Statistical difference was determined when $P < 0.05$.

3. Results and discussion

3.1. Comparison of drying time, specific energy consumption (SEC) and yields

Drying time, *SEC* and yields are closely related to industrial production. As shown in Table 1, the drying time of VFD was 27.33 ± 1.07 h, with the highest *SEC* (22.01 ± 0.37 kWh/kg). Although the drying time of HAD was also long (9.18 ± 0.15 h), its *SEC* was relatively low (6.70 ± 0.02 kWh/kg). This result was primarily due to the low power of the hot air drying equipment. VD shortened the drying time of the samples compared to HAD. However, energy consumption was significantly higher than HAD ($P < 0.05$). This was mainly because VD equipment needed a vacuum pumping process, dramatically increasing energy consumption. MD experienced the lowest energy consumption and drying time. This was attributed to the internal heating of materials during MD, which dramatically improved the heat transfer efficiency (An et al., 2022). SD used solar energy for drying, and although there was no production cost, the drying cycle was too long, susceptible to natural conditions such as weather, and unsuitable for large-scale industrial production. In terms of yields, the SD sample showed the highest yields. This result could be due to the low drying efficiency of SD, which made the sample insufficiently dried (Felsot, Rosen, & Chemistry, 2004). The yields of the MD sample were also second only to SD, which could be due to the inhomogeneous nature of microwave drying. Compared with MD, the yields of HAD and VD decreased slightly ($P > 0.05$). The lowest yields were observed in VFD, which may be due to the direct sublimation of moisture in the material by freeze-drying, which could minimize the moisture content of the material and thus minimize the yields (Le et al., 2022).

3.2. Analysis of the visual characteristics and color changes

The visual characteristics and color parameters of the coffee peel before and after drying by different methods are indicated in Fig. 1 and Table S1. In terms of shape, Fig. 1A suggests the VFD sample kept almost the same shape as the fresh coffee peel, while the shapes of HAD, MD, VD and SD were shrunk. The color of the coffee peel also changed significantly due to the different drying conditions through visual observation (Fig. 1B). Compared with fresh samples, the VFD sample was lighter, the samples treated by MD were light orange, the samples obtained by SD were close to brown yellow, while the color of samples treated by HAD and VD was similar (light brown). Regarding the color parameters, compared with the fresh sample, the L and a value of all treated samples decreased significantly, while the b values increased significantly ($P < 0.05$). These changes could be attributed to the browning reaction and degradation of polyphenols during drying (Rahimmalek & Goli, 2013). The lowest a value and the highest ΔE value may be due to the low drying rate and long heat exposure time of HAD, resulting in high pigment degradation and maximum color change (Chen et al., 2017). The maximum b value was observed in the SD-treated sample, indicating that the SD sample's yellow color was significant. This may be due to the induction of enzyme hydrolysis in a longer duration and lower temperature. Compared with HAD and SD, the negative effect on the color was less observed in the MD sample, possibly due to the high heat transfer efficiency and fast drying speed during MD treatment (Xu et al., 2020). A more considerable L value than MD was observed in VD. Chen et al. (2020) obtained consistent results that dried saffron obtained by vacuum drying is brighter than other thermal drying methods. The retention rate of natural pigment in VFD sample was the highest, and the sample was red, probably due to the low temperature and vacuum environment during drying (Sheng et al., 2016).

3.3. Effect of drying methods on microstructure

The microstructure can be used to judge the structure of dried products and evaluate their quality (Zhu et al., 2022). As shown in Fig. 2, the SEM of the VFD sample presented a honeycomb structure. Xu et al. (2022) found a similar structure in freeze-dried roses. This structure in VFD samples can be attributed to the direct sublimation of water from the frozen material into the gas phase under vacuum, which could effectively prevent the collapse of the solid matrix during freeze-drying, maintain structural stability, and thus form a porous honeycomb structure. Similar porous structures were also found in MD samples, but due to the uneven distribution of temperature and moisture diffusion during MD, the honeycomb structures were more irregular than VFD samples. Collapse and contraction of cell tissues were observed in HAD and SD samples, with closely arranged structures. Compared with HAD and SD samples, the collapse and contraction strength of cell tissue of the VD sample decreased, and a small proportion of pores were observed, which could be explained by the shorter drying time and lower oxygen concentration in the vacuum drying chamber (Jin et al., 2017; Xu et al., 2021).

3.4. Effect of drying methods on TPC, TFC and individual phenolic compound contents

Coffee peel has attracted much attention due to its rich active ingredients (Esquivel & Jimenez, 2012). The results of TPC and TFC in coffee peel under different treatments are illustrated in Fig. 3A. The TPC of fresh coffee peel was 21.10 ± 1.96 mg GAE/g DW, similar to the report of Duangjai et al. (2016). Many researchers have obtained different TPC results, which may be caused by different heredity, varieties, climates and regions of coffee peel (Heeger, Kosinska-Cagnazzo, Cantergiani, & Andlauer, 2017). Compared with the fresh sample, the TPC of coffee peel after VFD treatment was slightly reduced ($P > 0.05$), and the TPC of other dried samples was significantly decreased ($P < 0.05$). The TPC in the VFD sample was closer to that in the fresh sample, possibly because VFD prompt the formation of ice crystals in the plant matrix leading to greater destruction of the plant cell structure, thus making it easier for organic solvents to extract polyphenols (Long, Zhang, Mujumdar, & Chen, 2022). In addition, vacuum and freezing conditions could also effectively reduce the degradation of polyphenols (Kayacan et al., 2020). During hot drying, HAD and SD caused higher TPC loss rates of 71.2% and 65.87%, respectively. Xu et al. (2020) pointed out that the stability of phenolic compounds was vulnerable to the adverse effects of heat treatment and oxidation. During HAD and SD, the continuous drying time, low drying efficiency and large oxygen exposure area significantly aggravated the loss of polyphenols (Jamjariyatam et al., 2022). In contrast, VD and MD treatments resulted in relatively low TPC loss, with 37.30% and 52.60% loss rates, respectively. In the process of VD, the vacuum would produce a hypoxic

Table 1
Drying time (h), specific energy consumption (*SEC*) (kWh/kg) and yield (%) of coffee peel among different drying methods.

Parameters	HAD	MD	VD	SD	VFD
Drying time	9.18 ± 0.15^{c}	2.52 ± 0.04^{a}	5.45 ± 0.21^{b}	102.22 ± 4.32^{e}	27.33 ± 1.07^{d}
Specific energy consumption (*SEC*)	6.70 ± 0.02^{c}	4.31 ± 0.01^{b}	8.89 ± 0.03^{d}	0.00 ± 0.00^{a}	22.01 ± 0.37^{e}
Yields	19.84 ± 0.02^{b}	19.82 ± 0.11^{b}	19.83 ± 0.08^{b}	20.24 ± 0.17^{c}	18.86 ± 0.05^{a}

Note: HAD: hot air drying, MD: microwave drying, VD: vacuum drying, SD: sun drying, VFD: vacuum freeze drying. Data are presented as means ± SD (n = 3). Different letters (a-e) in the same row were significantly different ($P < 0.05$) (Duncan's test), where a was the lowest value.

Fig. 1. Photos of coffee peel (A) and powder (B) in different drying methods. HAD: hot air drying; MD: microwave drying; VD: vacuum drying; SD: sun drying; VFD: vacuum freeze drying.

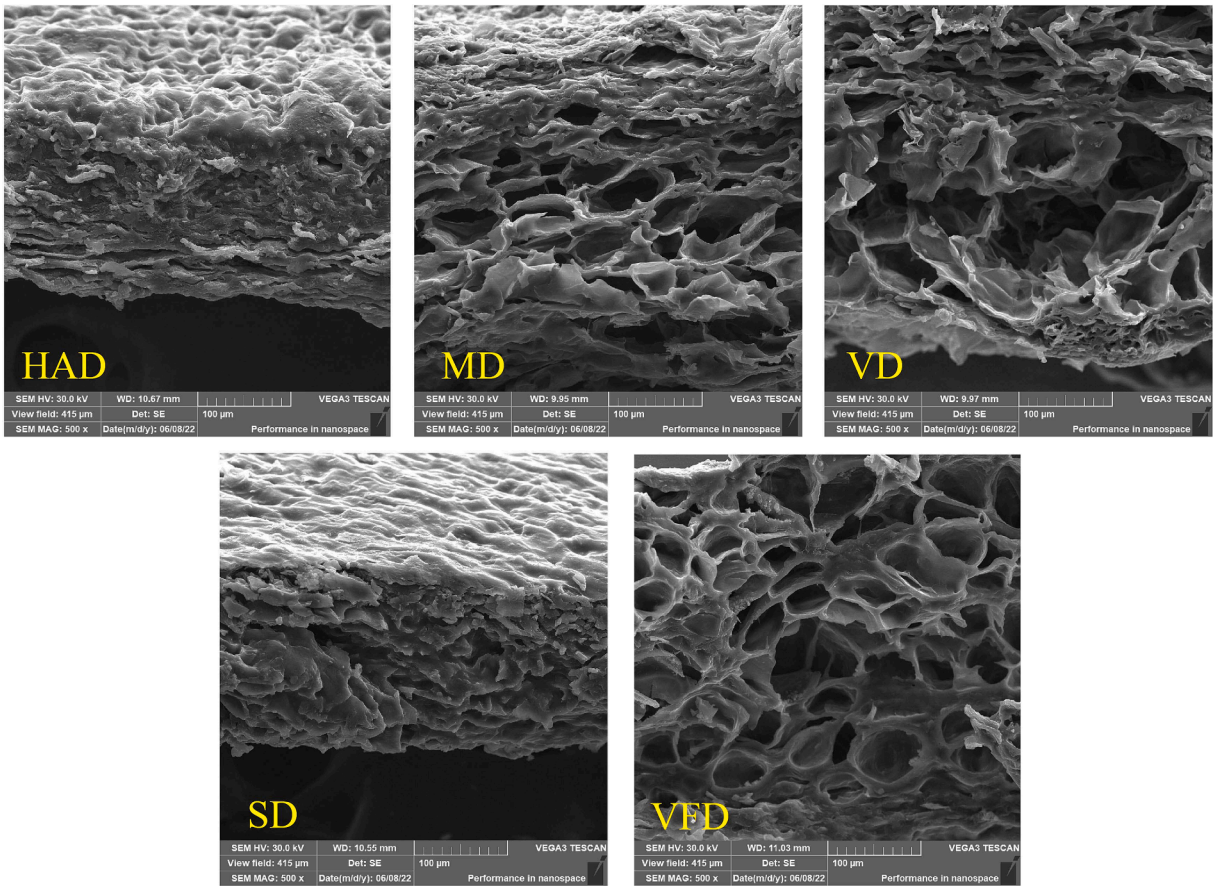

Fig. 2. Scanning electron microscope images of coffee peel cross-sectional view with different drying methods. HAD: hot air drying; MD: microwave drying; VD: vacuum drying; SD: sun drying; VFD: vacuum freeze drying.

environment, which could effectively reduce the activities of polyphenol oxidase and degrading enzymes, thus reducing the loss of polyphenols. The short drying time of MD may lead to lower heat load and phenol degradation (Zielinska & Zielinska, 2019).

The TFC of coffee peel decreased significantly after drying, compared with fresh samples ($P < 0.05$). SD, HAD and VD all displayed higher TFC loss rates ($P < 0.05$) with 64.72%, 59.76% and 48.46%, respectively, which were associated with higher drying temperature or longer drying time (Wojdylo et al., 2014). The loss rate of TFC by VFD and MD was relatively low, 16.09% and 28.46%, respectively, and there was no significant difference between them ($P > 0.05$). Based on the research results of Rahath Kubra, Kumar, and Rao (2016), appropriate microwave radiation can lead to cell decomposition and facilitate the extraction of flavonoids easier to obtain, which would explain the higher TFC content of MD samples.

As illustrated in Fig. 3C, eight phenolic substances in the sample were determined. Caffeine, trigonelline and chlorogenic acid were the most abundant phenolic compounds in the coffee peel, corresponding

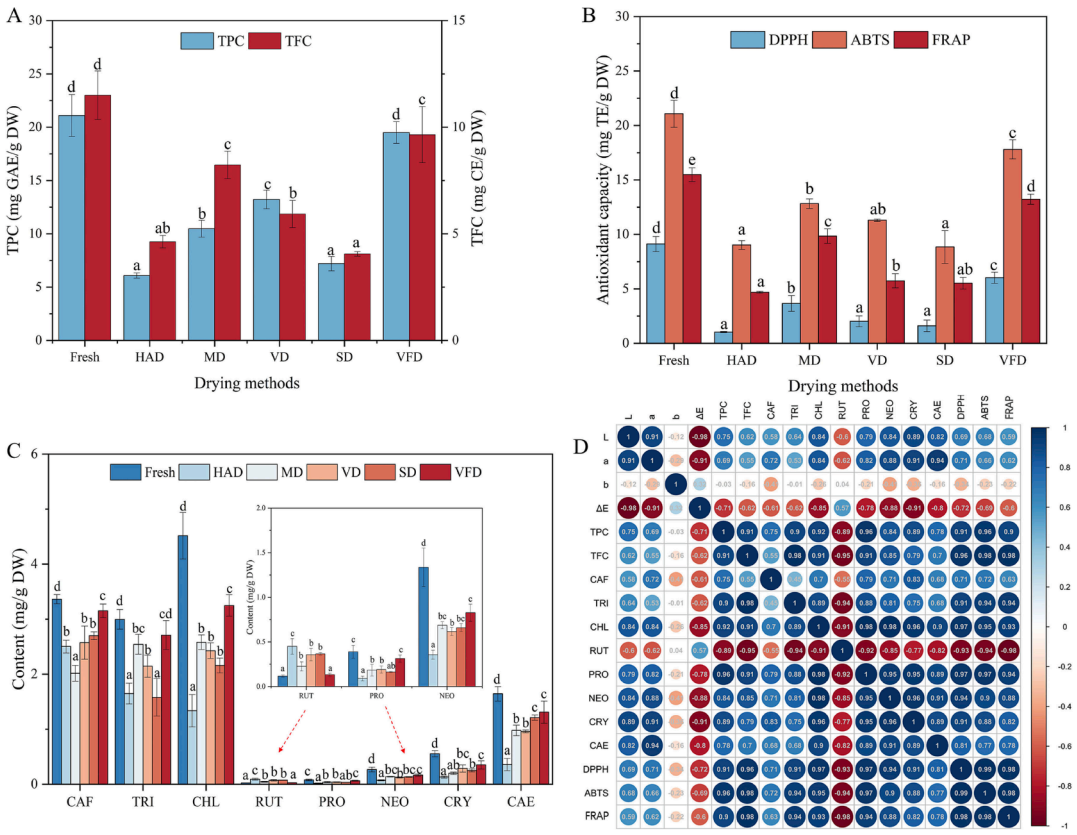

Fig. 3. Total phenolic and total flavonoid contents (TPC and TFC) (A), antioxidant activities (B), contents of individual phenolic compounds (C) and correlation analysis (D) of fresh and dried coffee peel. HAD: hot air drying; MD: microwave drying; VD: vacuum drying; SD: sun drying; VFD: vacuum freeze drying. CAF: caffeine; TRI: trigonelline; CHL: chlorogenic acid; RUT: rutin; PRO: protocatechuic acid; NEO: neochlorogenic acid; CRY: cryptochlorogenic acid; CAE: caffeic acid.

with results from Thy Minh Kieu et al. (2020), who previously reported abundant caffeine and chlorogenic acid in coffee peel. The caffeine content was highest in the VFD samples (3.15 ± 0.13 mg/g DW). The caffeine content in SD, VD and HAD was similar, ranging from 2.50 ± 0.12 mg/g DW to 2.70 ± 0.07 mg/g DW, with no significant difference ($P > 0.05$). This result could be explained by the relatively stable structure of caffeine (Dong et al., 2017). However, the caffeine content of the samples dried by the MD method was the lowest (2.02 ± 0.15 mg/g DW) ($P < 0.05$). This might be because a certain amount of microwave radiation penetration would destroy the structure of caffeine, resulting in a decrease in content. Chlorogenic acid, neochlorogenic acid, cryptochlorogenic acid, protocatechuic acid and caffeic acid of different treated samples had the same change trend. Compared with the fresh sample, the sample after drying was significantly reduced ($P < 0.05$). VFD treatment had the highest retention rate of chlorogenic acid, neochlorogenic acid, cryptochlorogenic acid and protocatechuic acid, followed by VD, MD, SD, and HAD samples had the lowest retention rate. This showed that longer drying time and higher drying temperature would accelerate the degradation of these phenolic acids, and a vacuum and freezing environment would help to retain their content (Dorta, Gloria Lobo, & Gonzalez, 2012; Kayacan et al., 2020). Compared to the VFD treatment, the rutin content increased significantly in the HAD, MD and VD treatments ($P < 0.05$). It should be noted that the rutin content of HAD, MD, VD and SD was 2.38, 0.70, 1.67 and 1.75 times higher than that of VFD, respectively. This phenomenon was also observed in dried fresh jujube fruit (Liu et al., 2022). As the main phenolic compound of coffee peel, trigonelline had the lowest loss rate of the VFD sample compared with the fresh sample, which indicated that the low temperature and dry environment were conducive to maintaining the trigonelline. During the hot drying process, the loss rates of TPC caused by HAD and SD were 44.98% and 47.30%, respectively. In contrast, the loss rate of trigonelline by MD was lower, which might be due to the lower thermal load caused by the shorter drying time of MD (Rahath Kubra, Kumar, Jagan Mohan Rao, & nutrition, 2016).

3.5. *Effect of drying methods on antioxidant activities*

As seen in Fig. 3B, three antioxidant activities in different coffee peels had the same trend. The values of DPPH, ABTS and FRAP of dehydrated coffee peel samples were significantly lower than those of fresh coffee peel ($P < 0.05$), which was consistent with the results obtained by An et al. (2016); Kayacan et al. (2020) on persimmon and Chinese ginger. The highest DPPH, ABTS and FRAP values were observed in VFD samples, followed by MD, VD and SD, whereas samples after HAD had the lowest radical scavenging capacity. VFD sample had the most potent antioxidant activities, which might be due to the vacuum and low-temperature environment during the drying process, thereby reducing the decomposition of antioxidants by light, heat and oxygen (Stamenkovic et al., 2019). Moreover, the DPPH, ABTS and FRAP values of MD samples were also higher, indicating that moderate microwave treatment could also effectively maintain the antioxidant activity of the coffee peel sample. Plant materials' antioxidant activity generally depends on their bioactive components' content. Thus, the correlation between TPC, TFC and individual phenolic compounds of coffee peel and the antioxidant indices were analyzed. As presented in Fig. 3D, the correlation analysis showed that DPPH, ABTS scavenging capacity and FRAP reducing capacity displayed a higher positive correlation with TPC ($r = 0.88, 0.94, 0.82$), TFC ($r = 0.95, 0.96, 0.96$), trigonelline ($r = 0.95, 0.96, 0.96$), chlorogenic acid ($r = 0.90, 0.92, 0.90$), protocatechuic acid ($r = 0.90, 0.92, 0.90$) and neochlorogenic acid ($r = 0.82, 0.76, 0.80$). Similar changes were found in the study of Deng et al. (2019).

3.6. Effect of drying methods on volatile compounds

The total ion chromatography (TIC) plots and detailed information on various compounds in the coffee peel are shown in Fig. 4A and Table 2. In this experiment, a total of 80 volatile compounds were identified in all treatment groups, including 21 aldehydes, 11 alcohols, 10 esters, 8 terpenes, 7 ketones, 6 furans, 5 acids, 5 alkenes, 4 alkanes and 3 pyrroles. Among them, 48, 57, 64, 63, 58 and 52 aroma compounds were identified in fresh, HAD, MD, VD, SD and VFD samples, respectively. As shown in Fig. 4B, all samples had a similar composition of aroma types, but the concentrations of each type differed. This difference was attributed to the different degrees of fatty acid degradation, glycoside hydrolysis, and merged reactions during the various drying processes of the samples (Liu et al., 2022). Aldehydes, alcohols, esters, terpenes and furans were the main volatile compounds detected in coffee peel, contributing to the typical floral, fruity and tea-like flavors. Some compounds have also been reported in previous studies, such as hexanal, heptanal, (E)-2-Heptenal, cinnamaldehyde, decanal, phenylethyl aldehyde, 1-Octanol, benzyl alcohol, phenethyl alcohol, nerol, 3,7-Dimethyl-1,6-octadien-3-ol, and indole (Pua et al., 2021), but geranyl acetone, (E)-2-Decen-1-ol, 2-Acetylfuran, 5-Methyl furfural and methyl salicylate were not reported in high concentrations in previous studies.

Considering the complex correlation of the data set, OPLS-DA was used to determine the correlation between the different treatments and volatiles (Fig. 4C). The independent variable fit index (R^2x) in this analysis was 0.868, the dependent variable fit index (R^2y) was 0.98, and the model prediction index (Q^2) was 0.959, with R^2 and Q^2 exceeding 0.5, indicating that acceptable model fit results. As shown in Fig. 4D, the intersection of the Q^2 regression line with the vertical axis was less than zero, meaning that there was no overfitting of the model, the model validation was valid, and the results were considered acceptable for the discriminative analysis of coffee peel aroma in different treatment groups. Fig. 4C shows that fresh and VFD samples were located in the same quadrant, and the VFD sample presented similar contents and types of volatile compounds as the fresh sample, indicating that VFD was more favorable for the retention of volatile compounds in the fresh coffee peel, which was similar to the results of An et al. (2016). The HAD and VD treatments were located in similar regions, suggesting that the volatile flavor components of the two treatments were similar. In the samples of HAD and VD, there was no significant difference in the total

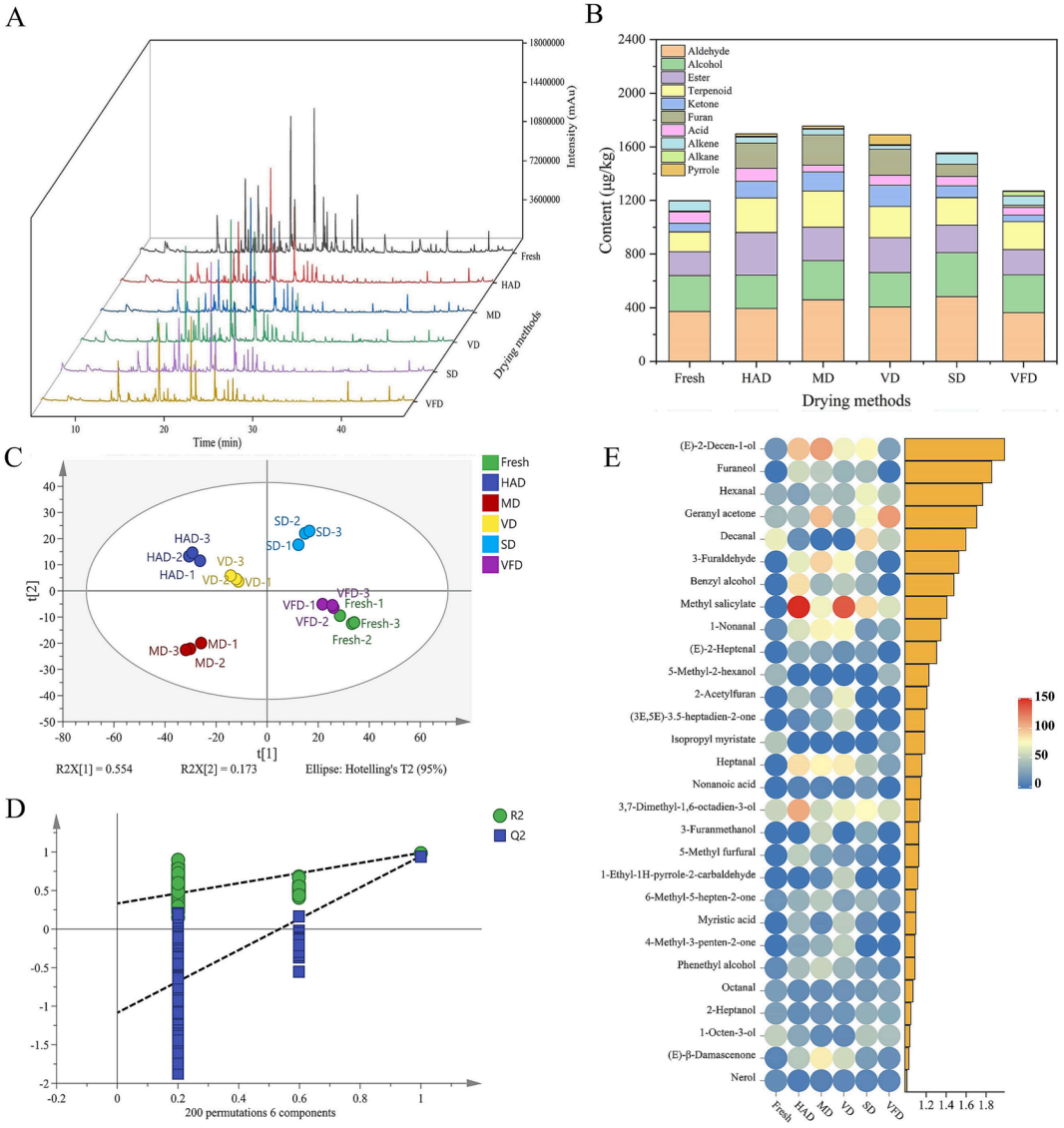

Fig. 4. The total ion chromatography (TIC) plots (A), contents of different types (B), score plots of the OPLS-DA model (C), cross-validation plot by 200 permutation tests (D) and heat map and scores with VIP > 1 (E) of volatile compounds for fresh and dried coffee peel. HAD: hot air drying; MD: microwave drying; VD: vacuum drying; SD: sun drying; VFD: vacuum freeze drying.

Table 2
Comparisons of the detected volatile components in fresh and dried coffee peel samples.

NO	[a] Compounds	[b] RI	CAS	Quantification (μg/kg DW)					
				Fresh	HAD	MD	VD	SD	VFD
	Aldehydes								
1	Hexanal	1081	66-25-1	31.69 ± 5.21^{a}	24.60 ± 8.06^{a}	42.37 ± 6.81^{bc}	38.65 ± 3.74^{b}	69.37 ± 10.30^{d}	46.24 ± 8.54^{c}
2	Heptanal	1192	111-71-7	0.00 ± 0.00^{a}	87.37 ± 9.95^{d}	79.16 ± 3.83^{d}	82.78 ± 8.47^{d}	46.11 ± 3.86^{c}	24.34 ± 1.02^{b}
3	(E)-2-Heptenal	1235	505-57-7	0.00 ± 0.00^{a}	33.12 ± 2.83^{d}	27.48 ± 1.70^{d}	14.63 ± 2.31^{b}	22.60 ± 0.83^{c}	0.00 ± 0.00^{a}
4	Octanal	1269	124-13-0	22.30 ± 4.67^{c}	11.99 ± 2.60^{a}	14.27 ± 2.59^{ab}	16.17 ± 4.85^{b}	18.58 ± 1.13^{b}	25.61 ± 3.59^{c}
5	1-Nonanal	1378	124-19-6	12.04 ± 0.64^{a}	60.14 ± 9.53^{d}	79.68 ± 13.62^{e}	77.36 ± 5.32^{e}	19.20 ± 2.11^{b}	29.21 ± 0.98^{c}
6	trans-2-Heptenal	1331	18829-55-5	0.00 ± 0.00^{a}	0.00 ± 0.00^{a}	4.98 ± 0.21^{c}	0.00 ± 0.00^{a}	7.02 ± 0.51^{d}	2.55 ± 0.12^{b}
7	(E)-2-Octenal	1424	2548-87-0	14.97 ± 3.78^{d}	9.07 ± 1.18^{b}	10.74 ± 0.18^{bc}	6.45 ± 1.14^{a}	15.68 ± 1.60^{d}	8.83 ± 0.73^{ab}
8	Tridecanal	1784	10486-19-8	27.35 ± 0.79^{e}	17.89 ± 2.35^{d}	7.43 ± 0.13^{a}	11.20 ± 1.02^{c}	8.72 ± 0.46^{b}	19.20 ± 0.87^{d}
9	(E,E)-2,4-Heptadienal	1488	4313-03-5	18.67 ± 0.13^{c}	0.00 ± 0.00^{a}	24.89 ± 0.25^{d}	7.74 ± 0.39^{b}	27.05 ± 4.41^{e}	0.00 ± 0.00^{a}
10	Decanal	1524	112-31-2	68.32 ± 8.87^{c}	14.98 ± 1.05^{b}	0.00 ± 0.00^{a}	0.00 ± 0.00^{a}	89.63 ± 6.48^{d}	50.32 ± 6.01^{c}
11	Undecanal	1605	112-44-7	25.63 ± 0.36^{d}	0.00 ± 0.00^{a}	0.00 ± 0.00^{a}	19.04 ± 0.45^{b}	0.00 ± 0.00^{a}	23.32 ± 0.12^{c}
12	phenylethyl aldehyde	1642	122-78-1	16.63 ± 1.21^{a}	42.72 ± 2.24^{e}	59.34 ± 2.98^{f}	32.32 ± 1.65^{c}	41.18 ± 5.33^{d}	24.86 ± 0.71^{b}
13	2,6,6-Trimethyl-1,3-cyclohexadiene-1-aldehyde	1656	116-26-7	0.00 ± 0.00^{a}	19.95 ± 2.37^{c}	25.37 ± 0.09^{d}	16.58 ± 0.32^{b}	0.00 ± 0.00^{a}	0.00 ± 0.00^{a}
14	Cinnamaldehyde	2004	104-55-2	0.00 ± 0.00^{a}	24.68 ± 0.42^{f}	8.13 ± 0.09^{c}	16.55 ± 3.71^{e}	9.17 ± 1.30^{d}	5.46 ± 0.05^{b}
15	trans-Cinnamaldehyde	2013	14371-10-9	6.98 ± 0.10^{a}	16.32 ± 2.87^{d}	7.98 ± 0.21^{b}	19.81 ± 1.21^{e}	6.42 ± 0.46^{a}	11.72 ± 2.34^{c}
16	Pyrrole-2-carboxaldehyde	2037	1003-29-8	0.00 ± 0.00^{a}	10.76 ± 0.53^{b}	25.89 ± 2.87^{d}	14.59 ± 0.22^{c}	0.00 ± 0.00^{a}	0.00 ± 0.00^{a}
17	Pentadecanal	2049	2765-11-9	0.00 ± 0.00^{a}	19.64 ± 1.06^{b}	0.00 ± 0.00^{a}	0.00 ± 0.00^{a}	0.00 ± 0.00^{a}	0.00 ± 0.00^{a}
18	Stearaldehyde	2379	638-66-4	35.31 ± 4.12^{d}	24.83 ± 0.78^{c}	17.37 ± 2.08^{a}	22.86 ± 1.36^{bc}	38.70 ± 0.31^{e}	25.24 ± 0.58^{c}
19	N-Methylpyrrole-2-carboxaldehyde	1622	1192-58-1	0.00 ± 0.00^{a}	10.94 ± 0.06^{b}	34.66 ± 1.03^{d}	27.70 ± 2.35^{c}	0.00 ± 0.00^{a}	0.00 ± 0.00^{a}
20	(4E,8E)-5,9,13-trimethyl-4,8,12-tetradecatrienal	2382	66408-5-7	26.78 ± 1.77^{e}	0.00 ± 0.00^{a}	21.40 ± 2.16^{d}	35.65 ± 4.06^{f}	6.00 ± 0.11^{b}	17.46 ± 1.32^{c}
21	α-Hexylcinnamaldehyde	2431	101-86-0	28.45 ± 3.75^{c}	0.00 ± 0.00^{a}	0.00 ± 0.00^{a}	19.09 ± 2.47^{b}	0.00 ± 0.00^{a}	21.87 ± 1.56^{b}
	Total aldehydes			**334.53 ± 23.78**^{a}	**427.83 ± 26.43**^{b}	**491.16 ± 38.53**^{c}	**479.18 ± 42.47**^{bc}	**424.37 ± 31.25**^{b}	**337.22 ± 15.75**^{a}
	Alcohols								
22	5-Methyl-2-hexanol	1176	111768-09-3	35.56 ± 4.09^{b}	0.00 ± 0.00^{a}	0.00 ± 0.00^{a}	0.00 ± 0.00^{a}	0.00 ± 0.00^{a}	37.27 ± 3.84^{b}
23	2-Heptanol	1302	543-49-7	23.45 ± 1.80^{c}	13.02 ± 0.52^{a}	16.42 ± 1.13^{b}	17.38 ± 0.43^{b}	32.65 ± 2.27^{e}	29.09 ± 0.71^{de}
24	1-Hexanol	1388	111-27-3	36.98 ± 5.12^{d}	0.00 ± 0.00^{a}	0.00 ± 0.00^{a}	14.70 ± 0.71^{b}	23.22 ± 0.42^{c}	35.11 ± 0.38^{d}
25	3-Octanol	1393	589-98-0	22.70 ± 0.94^{c}	0.00 ± 0.00^{a}	0.00 ± 0.00^{a}	18.63 ± 2.06^{b}	0.00 ± 0.00^{a}	14.04 ± 2.21^{b}
26	1-Octen-3-ol	1436	3391-86-4	50.36 ± 4.82^{d}	27.88 ± 1.44^{c}	19.82 ± 0.77^{b}	11.04 ± 0.26^{a}	45.75 ± 3.49^{cd}	42.37 ± 6.08^{c}
27	(E)-2-Decen-1-ol	1474	18409-18-2	17.32 ± 2.38^{a}	98.63 ± 7.45^{de}	111.01 ± 11.27^{e}	70.28 ± 5.07^{c}	78.35 ± 6.83^{c}	24.36 ± 0.85^{b}
28	2,3-Dimethylcyclohexan-1-ol	1529	1502-24-5	0.00 ± 0.00^{a}	0.00 ± 0.00^{a}	36.27 ± 2.84^{b}	0.00 ± 0.00^{a}	0.00 ± 0.00^{a}	0.00 ± 0.00^{a}
29	2,3-Butanediol	1583	513-85-9	5.33 ± 0.02^{a}	20.11 ± 3.13^{d}	26.92 ± 0.15^{e}	9.24 ± 0.48^{b}	13.07 ± 1.26^{c}	10.18 ± 0.63^{b}
30	1-Octanol	1588	111-87-5	7.30 ± 0.16^{a}	39.66 ± 5.19^{d}	12.12 ± 1.88^{b}	13.13 ± 1.23^{b}	21.57 ± 2.09^{c}	12.23 ± 0.77^{b}
31	Benzyl alcohol	1827	100-51-6	0.00 ± 0.00^{a}	87.44 ± 9.03^{d}	37.30 ± 2.35^{b}	48.50 ± 3.54^{c}	34.40 ± 4.35^{b}	0.00 ± 0.00^{a}
32	Phenethyl alcohol	1954	60-12-8	11.54 ± 0.32^{a}	40.57 ± 3.58^{e}	53.63 ± 4.72^{f}	36.40 ± 3.15^{de}	22.41 ± 0.89^{c}	13.44 ± 0.18^{b}
	Total alcohols			**231.64 ± 10.68**^{a}	**298.96 ± 23.12**^{c}	**313.48 ± 35.36**^{c}	**267.64 ± 15.68**^{b}	**271.42 ± 15.89**^{b}	**258.17 ± 19.47**^{ab}
	Esters								
33	Methyl salicylate	1727	119-36-8	0.00 ± 0.00^{a}	152.79 ± 16.98^{d}	70.39 ± 12.35^{b}	136.47 ± 10.69^{d}	88.76 ± 9.78^{c}	61.00 ± 5.34^{b}

(*continued on next page*)

Table 2 (*continued*)

NO	[a] Compounds	[b] RI	CAS	Quantification (μg/kg DW)					
				Fresh	HAD	MD	VD	SD	VFD
34	2-Ethyl-3-hydroxyhexyl 2-methylpropanoate	1974	74367-31-0	21.36 ± 3.85^{d}	0.00 ± 0.00^{a}	0.00 ± 0.00^{a}	0.00 ± 0.00^{a}	7.63 ± 1.79^{bc}	12.80 ± 2.13^{c}
35	Isopropyl myristate	2056	110-27-0	45.89 ± 6.30^{c}	0.00 ± 0.00^{a}	0.00 ± 0.00^{a}	0.00 ± 0.00^{a}	0.00 ± 0.00^{a}	26.70 ± 2.15^{b}
36	Methyl palmitate	2228	112-39-0	0.00 ± 0.00^{a}	22.58 ± 2.03^{e}	13.55 ± 0.68^{bc}	15.74 ± 0.82^{cd}	18.43 ± 3.84^{d}	0.00 ± 0.00^{a}
37	Diisobutyl phthalate	2534	84-69-5	75.53 ± 6.62^{e}	32.42 ± 2.99^{a}	38.77 ± 4.43^{b}	48.20 ± 5.66^{cd}	57.10 ± 4.80^{d}	55.00 ± 5.89^{d}
38	Furaneol	1662	98-00-0	0.00 ± 0.00^{a}	56.65 ± 7.36^{d}	49.06 ± 6.35^{c}	35.33 ± 3.27^{b}	38.77 ± 4.48^{b}	0.00 ± 0.00^{a}
39	Bis(2-ethylhexyl) adipate	2409	103-23-1	11.03 ± 2.13^{a}	16.89 ± 0.75^{c}	21.13 ± 3.12^{d}	10.22 ± 0.78^{a}	14.36 ± 1.47^{bc}	11.53 ± 0.46^{a}
40	Octyl 4-methoxycinnamate	2753	5466-77-3	18.12 ± 1.03^{d}	5.66 ± 0.42^{b}	27.31 ± 3.75^{e}	0.00 ± 0.00^{a}	0.00 ± 0.00^{a}	11.87 ± 2.11^{c}
41	L-Ascorbyl dipalmitate	2028	28474-90-0	6.52 ± 0.72^{a}	26.88 ± 3.46^{d}	29.94 ± 2.15^{d}	15.30 ± 0.16^{b}	18.83 ± 0.63^{c}	8.44 ± 1.00^{a}
42	Propane acid 2-methyl-3-hydroxy-2,4,4-trimethyl-pentyl ester	2576	74367-34-3	45.12 ± 7.28^{d}	0.00 ± 0.00^{a}	0.00 ± 0.00^{a}	0.00 ± 0.00^{a}	11.34 ± 0.08^{b}	23.87 ± 0.25^{c}
	Total esters			**223.57 ± 32.53^{a}**	**313.88 ± 35.23^{c}**	**250.14 ± 15.63^{ab}**	**261.27 ± 20.36^{ab}**	**255.21 ± 10.44^{ab}**	**211.23 ± 13.85$_{a}$**
	Terpenoids	1554							
43	3,7-Dimethyl-1,6-octadien-3-ol		78-70-6	55.12 ± 8.76^{a}	106.96 ± 15.33^{d}	55.29 ± 4.98^{a}	67.95 ± 8.63^{bc}	76.16 ± 11.34^{c}	58.11 ± 6.54^{a}
44	α-Terpineol	1686	98-55-5	0.00 ± 0.00^{a}	33.18 ± 4.39^{d}	13.46 ± 2.54^{b}	24.44 ± 3.18^{c}	0.00 ± 0.00^{a}	0.00 ± 0.00^{a}
45	Geraniol	1841	106-24-1	0.00 ± 0.00^{a}	19.55 ± 1.87^{d}	7.01 ± 0.09^{b}	10.35 ± 0.69^{c}	0.00 ± 0.00^{a}	0.00 ± 0.00^{a}
46	Nerol	1782	106-25-2	13.61 ± 1.58^{e}	4.87 ± 1.01^{a}	6.50 ± 0.43^{b}	10.03 ± 1.26^{d}	8.68 ± 0.36^{c}	9.01 ± 0.05^{cd}
47	(E)-β-Damascenone	1850	23726-93-4	8.12 ± 0.21^{a}	46.75 ± 4.46^{d}	82.41 ± 10.36^{f}	57.13 ± 3.88e	21.49 ± 1.06^{c}	14.07 ± 0.45^{b}
48	Geranyl acetone	1883	3796-70-1	38.33 ± 3.51^{a}	40.96 ± 2.88^{a}	100.02 ± 15.69^{cd}	39.27 ± 4.39^{a}	72.52 ± 7.21^{b}	112.19 ± 19.37^{d}
49	α-Ionone	1904	127-41-3	23.01 ± 1.88^{e}	0.00 ± 0.00^{a}	4.22 ± 0.04^{b}	12.13 ± 1.17^{d}	8.28 ± 0.36^{c}	13.39 ± 0.41^{d}
50	Myrcene	1162	123-35-3	0.00 ± 0.00^{a}	5.64 ± 0.14^{c}	1.69 ± 0.02^{b}	10.21 ± 0.98^{d}	18.06 ± 0.36^{e}	0.00 ± 0.00^{a}
	Total terpenoids			**138.19 ± 7.63^{a}**	**257.92 ± 23.10^{cd}**	**270.59 ± 31.64^{d}**	**231.51 ± 20.87^{bc}**	**205.19 ± 18.33^{b}**	**206.79 ± 12.48^{b}**
	Ketones								
51	4-Methyl-3-penten-2-one	1147	141-79-7	0.00 ± 0.00^{a}	23.48 ± 1.43^{b}	33.14 ± 4.89^{c}	49.24 ± 4.06^{d}	0.00 ± 0.00^{a}	0.00 ± 0.00^{a}
52	3-Hydroxy-2-butanone	1296	513-86-0	0.00 ± 0.00^{a}	3.82 ± 0.07^{b}	5.26 ± 2.16^{cd}	6.11 ± 0.78^{d}	3.29 ± 0.20^{b}	5.29 ± 0.13^{cd}
53	1-Octen-3-one	1323	4312-99-6	47.99 ± 7.04^{d}	0.00 ± 0.00^{a}	0.00 ± 0.00^{a}	0.00 ± 0.00^{a}	24.65 ± 4.21^{c}	11.95 ± 1.03^{b}
54	6-Methyl-5-hepten-2-one	1349	110-93-0	15.63 ± 1.11^{a}	33.36 ± 3.13^{c}	48.11 ± 3.86^{d}	24.41 ± 5.21^{b}	36.23 ± 4.34^{c}	20.39 ± 3.34^{b}
55	(3E,5E)-3.5-heptadien-2-one	1538	18402-90-9	0.00 ± 0.00^{a}	9.26 ± 0.36^{b}	27.11 ± 1.85^{c}	55.11 ± 6.31^{d}	0.00 ± 0.00^{a}	0.00 ± 0.00^{a}
56	Acetophenone	1635	98-86-2	0.00 ± 0.00^{a}	28.53 ± 3.88^{f}	22.15 ± 4.31^{ef}	8.25 ± 1.23^{c}	12.43 ± 0.69^{d}	5.18 ± 0.11^{b}
57	2-Pentadecanone	2036	2345-28-0	0.00 ± 0.00^{a}	25.85 ± 1.53^{f}	7.43 ± 0.45^{c}	15.07 ± 0.36^{e}	10.94 ± 0.11^{d}	0.00 ± 0.00^{a}
	Total ketones			**63.62 ± 12.09^{b}**	**124.29 ± 13.74^{de}**	**143.20 ± 11.36^{e}**	**158.19 ± 10.70^{e}**	**87.54 ± 9.36^{c}**	**42.81 ± 3.96^{a}**
	Furans								
58	3-Furaldehyde	1432	498-60-2	0.00 ± 0.00^{a}	66.68 ± 4.74^{c}	91.30 ± 10.63^{d}	73.46 ± 8.01^{c}	34.31 ± 5.96^{b}	0.00 ± 0.00^{a}
59	5-Methyl furfural	1565	620-02-0	0.00 ± 0.00^{a}	49.70 ± 9.67^{d}	27.05 ± 5.36^{c}	19.61 ± 4.14^{bc}	12.37 ± 3.08^{b}	0.00 ± 0.00^{a}
60	5-Hydroxymethylfurfural	2502	67-47-0	0.00 ± 0.00^{a}	10.89 ± 0.83^{c}	0.00 ± 0.00^{a}	16.13 ± 3.28^{d}	5.02 ± 0.04^{b}	0.00 ± 0.00^{a}
61	3-Furanmethanol	1673	4412-91-3	0.00 ± 0.00^{a}	0.00 ± 0.00^{a}	53.17 ± 6.69^{c}	0.00 ± 0.00^{a}	29.11 ± 1.09^{b}	0.00 ± 0.00^{a}
62	2-Acetylfuran	1492	1192-62-7	0.00 ± 0.00^{a}	42.47 ± 7.38^{c}	25.35 ± 3.21^{b}	68.77 ± 5.76^{d}	0.00 ± 0.00^{a}	0.00 ± 0.00^{a}
63	2-Pentylfuran	1237	3777-69-3	5.74 ± 0.20^{a}	17.36 ± 1.21^{e}	27.88 ± 0.97^{f}	13.45 ± 0.52^{d}	9.44 ± 0.45^{c}	8.36 ± 1.09^{bc}
	Total furans			**5.74 ± 0.20^{a}**	**187.10 ± 17.38^{d}**	**224.75 ± 23.63^{e}**	**191.42 ± 15.18^{d}**	**90.25 ± 8.91^{c}**	**8.36 ± 1.09^{b}**
	Acids								
64	Hexanoic acid	1829	142-62-1	0.00 ± 0.00^{a}	0.00 ± 0.00^{a}	3.46 ± 0.03^{b}	11.38 ± 0.88^{d}	8.35 ± 0.63^{c}	0.00 ± 0.00^{a}
65	Nonanoic acid	2126	112-05-0	0.00 ± 0.00^{a}	6.83 ± 0.51^{bc}	7.41 ± 1.08^{c}	5.79 ± 0.84^{b}	19.87 ± 0.46^{d}	0.00 ± 0.00^{a}
66	Decanoic acid	2259	334-48-5	21.02 ± 3.18^{d}	17.86 ± 2.04^{d}	15.91 ± 0.67^{c}	2.72 ± 0.11^{a}	10.19 ± 0.25^{b}	17.69 ± 1.38^{d}

(*continued on next page*)

Table 2 (*continued*)

NO	[a] Compounds	[b] RI	CAS	Quantification (μg/kg DW)					
				Fresh	HAD	MD	VD	SD	VFD
67	Myristic acid	2733	544-63-8	0.00 ± 0.00^{a}	37.19 ± 2.51^{c}	11.28 ± 2.19^{b}	49.10 ± 5.63^{d}	13.96 ± 2.36^{b}	0.00 ± 0.00^{a}
68	Pentadecanoic acid	2828	1002-84-2	10.36 ± 1.49^{de}	4.15 ± 0.21^{a}	12.58 ± 1.88^{e}	8.62 ± 0.76^{cd}	21.52 ± 1.81	7.84 ± 0.20^{b}
	Total acids			**31.38 ± 2.08^{b}**	**66.03 ± 8.34^{cd}**	**50.63 ± 4.34^{c}**	**77.61 ± 6.36^{d}**	**73.90 ± 4.68^{d}**	**25.53 ± 1.82^{a}**
	Alkenes								
69	2-Octene	886	111-67-1	35.15 ± 4.58^{c}	0.00 ± 0.00^{a}	0.00 ± 0.00^{a}	0.00 ± 0.00^{a}	13.32 ± 1.65^{b}	34.65 ± 6.39^{c}
70	1-Ethyl-5-methylcyclopentene	1299	97797-57-4	41.55 ± 3.08^{e}	13.55 ± 0.97^{b}	8.36 ± 0.22^{a}	12.36 ± 1.38^{b}	38.09 ± 5.01^{de}	30.32 ± 4.15^{c}
71	1,5-Cyclodecadiene	1743	75023-40-4	0.00 ± 0.00^{a}	11.33 ± 0.45^{d}	8.96 ± 0.50^{c}	5.98 ± 0.08^{b}	16.15 ± 1.38^{e}	0.00 ± 0.00^{a}
72	2-Nonene	1221	17003-99-5	0.00 ± 0.00^{a}	3.56 ± 0.06^{b}	18.63 ± 3.14^{c}	0.00 ± 0.00^{a}	0.00 ± 0.00^{a}	0.00 ± 0.00^{a}
73	1,3-Hexadiene	1287	61142-36-7	0.00 ± 0.00^{a}	7.28 ± 0.89^{d}	7.25 ± 0.03^{d}	6.31 ± 0.52^{cd}	9.71 ± 1.25^{e}	2.31 ± 0.22^{b}
	Total alkenes			**76.70 ± 8.12^{e}**	**35.72 ± 3.96^{b}**	**43.20 ± 4.66^{c}**	**24.65 ± 3.08^{a}**	**77.28 ± 10.41^{e}**	**64.94 ± 9.33^{de}**
	Alkanes								
74	Heptadecane	1742	1560-89-0	0.00 ± 0.00^{a}	0.00 ± 0.00^{a}	0.00 ± 0.00^{a}	0.00 ± 0.00^{a}	0.00 ± 0.00^{a}	5.71 ± 0.36^{b}
75	Octadecane	1805	593-45-3	0.00 ± 0.00^{a}	0.00 ± 0.00^{a}	3.70 ± 0.23^{b}	0.00 ± 0.00^{a}	0.00 ± 0.00^{a}	11.95 ± 1.85^{c}
76	Heneicosane	1894	629-94-7	0.00 ± 0.00^{a}	0.00 ± 0.00^{a}	0.98 ± 0.02^{b}	0.00 ± 0.00^{a}	0.00 ± 0.00^{a}	9.06 ± 0.43^{c}
77	Cyclohexadecane	2053	295-65-8	0.00 ± 0.00^{a}	6.45 ± 0.74^{d}	0.00 ± 0.00^{a}	5.34 ± 0.11^{cd}	3.10 ± 0.06^{b}	0.00 ± 0.00^{a}
	Total alkanes			**0.00 ± 0.00^{a}**	**6.45 ± 0.74^{e}**	**4.68 ± 0.65^{cd}**	**5.34 ± 0.11^{de}**	**3.10 ± 0.06^{b}**	**26.72 ± 4.21^{f}**
	Pyrroles								
78	Indole	2438	120-72-9	1.35 ± 0.08^{a}	5.89 ± 0.06^{e}	5.13 ± 0.23d	2.18 ± 0.21^{b}	3.40 ± 0.75^{c}	2.97 ± 0.21^{bc}
79	1-Ethyl-1H-pyrrole-2-carbaldehyde	1624	2167-14-8	0.00 ± 0.00^{a}	0.00 ± 0.00^{a}	12.22 ± 0.41^{b}	51.66 ± 7.38^{c}	0.00 ± 0.00^{a}	0.00 ± 0.00^{a}
80	1-(2-furanylmethyl)-1h-pyrrol	1846	1438-94-4	0.00 ± 0.00^{a}	11.81 ± 0.24^{b}	0.00 ± 0.00^{a}	19.72 ± 1.23^{c}	0.00 ± 0.00^{a}	0.00 ± 0.00^{a}
	Total pyrroles			**1.35 ± 0.08^{a}**	**17.70 ± 2.24^{d}**	**17.35 ± 0.85^{d}**	**73.57 ± 8.34^{e}**	**3.40 ± 0.55^{c}**	**2.97 ± 0.21^{bc}**
	General Total			**1106.71 ± 68.78^{a}**	**1744.19 ± 87.33^{cd}**	**1805.61 ± 114.98^{d}**	**1775.23 ± 91.26^{cd}**	**1520.07 ± 88.65^{b}**	**1123.76 ± 46.36^{a}**

Note: HAD: hot air drying, MD: microwave drying, VD: vacuum drying, SD: sun drying, VFD: vacuum freeze drying. Data are presented as means ± SD (n = 3). Different letters (a-f) in the same row were significantly different ($P < 0.05$) (Duncan's test), where a was the lowest value.

[a] Volatile compounds detected were integrated with the GC-MS automatic deconvolution system and compared with the standard mass spectrum in the NIST 5.0 library.

[b] RI, retention index.

volatile compounds content ($P > 0.05$), but fewer volatile compounds were detected in the samples of HAD. This might be because hot air heating accelerates the loss of volatile compounds (Dong et al., 2017). In contrast, the MD and SD treatments were located in different quadrants and could be clearly distinguished from the other treatments. The SD had the lowest total content of volatile compounds. This could be due to the fact that solarization was conducted at low temperatures, inhibiting the production of volatile compounds (Zhu et al., 2022). The most volatile substances and the highest content were found in MD, showing that microwave treatment favored the production of volatile compounds in coffee peel (Tian, Zhao, Huang, Zeng, & Zheng, 2016).

To further analyze the contribution of different treatments to coffee peel flavor, 29 differential aroma substances were screened according to the criterion of VIP > 1 (Fig. 4E and Table 2S). (E)-2-Decen-1-ol (VIP = 1.99) and furaneol (VIP = 1.86), as the most dominant discriminating substances, were produced by the oxidative degradation of unsaturated fatty acids during drying and contributed to the fruity and caramel aromas in the samples (Xu et al., 2022; Yang et al., 2022). The two substances were detected in the hot-dried samples (HAD, MD, VD and SD) and exhibited the highest content in the HAD and MD samples. Compared to the VFD sample, hot drying also promoted the production of 3-furaldehyde, 2-furanmethanol, heptanal, 5-methylfurfural, 2-hexenal, (3E,5E)-3,5-heptadien-2-one, and 4-Methyl-3-penten-2-one, bringing burnt and fatty aromas (Li et al., 2023). All these substances were detected in HAD samples and their contents were significantly higher than in the other treatments ($P > 0.05$). Although most substances were also detected in the VD and MD samples, their content was significantly lower than in the HAD treatment. 5-methyl-2-hexanol and isopropyl myristate were detected only in the fresh and VFD samples, giving them a fresh and floral flavor. Moreover, it was also shown that these compounds were unstable and easily degraded during thermal drying. Some fruit and tea aroma components were also detected among the discriminative flavor compounds. For example, 1-nonenal, (E)-β-damascenone and phenyl ethanol in dried dates imparted a strong honey and sweet aroma to coffee peel (Liu et al., 2022). Three substances were detected in all treatments, and the contents of MD, HAD and VD samples were significantly higher than those of VFD and SD, especially in MD samples. Geranyl acetone, benzyl alcohol, 3,7-dimethyl-1,6-octadien-3-ol, nonanoic acid, phenyl ethanol, and myristic acid in black tea were also found in coffee peel (Qu et al., 2019). Compared with the fresh sample, except for the dynamic changes of benzyl alcohol and myristic acid, the content of other volatile substances increased after drying, which presented that drying treatment was helpful to the formation of black tea flavor in coffee peel. 1-Octanol and nerol in hawthorn, as well as 1-octen-3-ol and 2-heptanol in rosehip, were also detected (Demir, Yildiz, Alpaslan, & Hayaloglu, 2014; Zhu & Xiao, 2018). These substances offered a strong floral, fruity and earthy scent. The flavor reminded one of rose, lavender, hawthorn and hay, giving the final products a sweet herbaceous taste. The contents of these flavor substances were lower in HAD, MD and VD samples but higher in

SD and VFD samples. This may be caused by thermal decomposition during drying (Tian et al., 2016).

The above results exhibited that compared with the fresh sample, the VFD treatment was more conducive to the retention of the original volatile flavor in the fresh sample, and hot drying increased the total content and quantity of volatile compounds in the coffee peel, especially the MD treatment. In hot drying, the SD sample had the most obvious earthy and herbaceous aroma, and HAD, MD and VD had pronounced caramel, fat, honey and sweet smell. In contrast, the HAD sample had the most significant caramel and fat scent, and the MD sample had the most pronounced honey and sweet flavors.

3.7. Economic evaluation and current application of coffee peel

Table S3 presents the operating costs of different drying methods during operation. Obviously, MD was the most economically attractive among all the drying methods. VFD exhibited the least favorable economic feasibility due to its expensive equipment and extended drying time. Although VD has higher operating costs than HAD, VD can significantly reduce drying time, bring more bioactive components and produce more volatile flavors. SD utilizes solar energy and incurs no production costs. But the drying cycle was too long and susceptible to weather and other natural conditions, making it unsuitable for large-scale industrial production. Therefore, it was recommended to employ MD to dehydrate the coffee peel. In practical production, VFD could be chosen to obtain more raw bioactive components to produce high additional products. Considering the equipment investment and production cost, MD was more suitable for a wide range of industrial production than other drying methods.

The coffee peel contains rich nutrients, bioactive components and volatile components. This makes it possible to produce high-value-added products. Through literature search and market research, it can be found that coffee peel can be used in many fields. As shown in Table S4, coffee peel has been used to produce tea, fruit wine, mixed fruit juice drinks, refreshing drinks and other foods for ordinary food. In terms of feed, coffee peel extracts can be used as an additive in animal feed to reduce the use of hormones and antibiotics in the breeding process. The coffee peel can serve as a functional food additive. Incorporating dried coffee powders into baked goods significantly enhanced their protein and dietary fiber content, as well as their antioxidant activity. The coffee peel extracts can be added to the polymer composite as a plasticizer of the polymer matrix to improve the performance of the composite. The modified dried coffee peel can be used as a matrix to produce biochar, biosorbent, enzyme, mushroom and biofuel. In addition, the coffee peel can also be used as a source of dietary fiber, prebiotics, organic acids, lignin, antioxidants, antibacterial agents, spices and aromatic compounds, enabling coffee peel to be used in the food, pharmaceutical, cosmetic and other industries.

4. Conclusions

The effects of five different drying methods (HAD, MD, VD, SD, VFD) on the physical properties, bioactive components, antioxidant capacity, volatile components and industrial application of coffee peel were evaluated. The results showed differences in the quality of coffee peel under different drying methods. The VFD treatment could better maintain the appearance, color, total polyphenols, total flavonoids, antioxidant activity, most phenolic compounds and original volatile components in the fresh sample than other technologies. Nonetheless, its drying time was longer and energy consumption was higher. Although hot drying (HAD, MD, VD, SD) had a higher loss of color, internal structure, bioactive components and antioxidant activity of coffee peel than VFD treatment, its operating cost was generally low, and it also produced pleasant flavors such as caramel, fat, floral fragrance, grass and honey. Compared with HAD and SD, MD and VD treatments significantly reduced the drying time and increased the types and contents of bioactive components and volatile compounds. The results indicated that MD or VD could be an effective alternative to conventional sun drying. Considering its drying efficiency, biological activity maintenance and production cost, MD had the broadest market prospects. However, microwave drying also had many shortcomings, such as insufficient drying. Considering the economic benefits and overall product quality, we plan to combine MD with other drying technologies to address the shortcomings of microwave drying, further improve the drying technical parameters, and continuously provide high-quality coffee peel raw materials for the market and processing enterprises.

CRediT authorship contribution statement

Dongsheng Hu: Data curation, Visualization, Writing – original draft. **Xiaogang Liu:** Writing – review & editing. **Yuyue Qin:** Writing – review & editing. **Jiatong Yan:** Writing – review & editing. **Rongmei Li:** Investigation. **Qiliang Yang:** Investigation.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Ethical guidelines

Ethics approval was not required for this research.

Date availability statement

Research data are not shared.

Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.fochx.2023.100807.

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