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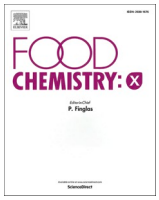

Changes in chemical composition, volatile compound, and bioactive compounds retention in shallots (*Allium ascalonicum* L.) under different drying methods

Jiranan Ratseewo [a], Theeraphan Chumroenphat [b], Hua Li [c], Sirithon Siriamornpun [d,*]

[a] *Division of Food Innovation and Technology, Faculty of Liberal Arts and Science, Sisaket Rajabhat University, Sisaket 33000, Thailand*
[b] *Aesthetic Sciences and Health Program, Faculty of Thai Traditional and Alternative Medicine, Ubon Ratchathani Rajabhat University, Ubonratchathani 34000, Thailand*
[c] *Department of Cuisine and Nutrition, Yangzhou University, Yangzhou, Jiangsu Province 225127, China*
[d] *Research Unit of Thai Food Innovation, Department of Food Technology and Nutrition, Mahasarakham University, Kantarawichai, Maha Sarakham 44150, Thailand*

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ABSTRACT

Shallots (*Allium ascalonicum* L.) are widely used in culinary and medicinal applications due to their rich bioactive compounds and health benefits. This study examines the effects of freeze drying, sun drying, and hot air drying at 40, 60, and 80 °C on chemical and bioactive compounds in shallots. Hot air drying at 80 °C had the highest total phenolic and flavonoid contents. Freeze drying increased syringic, *p*-coumaric, protocatechuic, vanillic, and ferulic acids. Quercetin was highest in freeze drying. Apigenin increased in samples that was dried by hot air drying at 80 °C. Proline was the major amino acid; arginine increased by 34 % in sun drying. Fourier Transform Infrared Spectroscopy (FTIR) revealed polysaccharides, lipids-proteins, and amino acids. Eucalyptol was the predominant volatile component in fresh shallot and most preserved by freeze drying. The results underscore the significance of choosing suitable drying methods to preserve shallot bioactive compounds and antioxidant properties.

1. Introduction

Shallot (*Allium ascalonicum* L.) is an important economic crop and spice that is popularly consumed and cultivated around the world, especially Asia and Southeast Asia such as China, Vietnam, Indonesia, and Thailand. Shallots are a specific shade of red, and have a spicy flavor. Shallot is recognized as spices that also enhance health and taste of food. It has been used as an herbal, medicine, and culinary spice. It is rich in phytochemicals such as phenolics, flavonoids and volatile compounds (Lu et al., 2011; Sfara et al., 2009; Thuy et al., 2019). These compounds have potential for health benefits, prevent of many diseases related to inflammation and malignancy, angiogenesis pathways (Ruksiriwanich et al., 2022) and anti-carcinogenic properties (Ravindranath et al., 2023).

Phytochemicals, including phenolics, flavonoids, and volatile compounds, are recognized for their health benefits. Phenolic compounds are commonly found in vegetables and spices (Chumroenphat et al., 2021; Ratseewo et al., 2016), with shallots containing *p*-coumaric acid as the major constituent (Ruksiriwanich et al., 2022). Flavonoids are abundance in shallot, especially quercetin (Ruksiriwanich et al., 2022). Shallot has high moisture that affects long-term storage, causing them to be processed into shallot powder through drying. Dried shallots are important due to their versatile applications, nutritional benefits, and practical advantages. Drying significantly extends their shelf life, reducing spoilage and making them a reliable pantry staple, especially where fresh produce is out of season. In recent years, dried shallot has been used in industries. They are widely used in the food industry for ready-to-eat meals, spice blends, and seasonings. Their nutritional benefits, culinary usefulness, and convenience make dried shallots essential in both household and commercial kitchens. However, the application of heat to plants during processing results in changes in heat-sensitive compounds. In a recent study, the firmness and bioactive components of raw purple shallots were examined in relation to the application of mild heat and calcium chloride treatment (Thuy et al., 2019). Furthermore, Tian et al. (2021) analyzed the volatile compound changes in fried shallot oil at various frying temperatures, emphasizing

* Corresponding author.
E-mail address: sirithons@hotmail.com (S. Siriamornpun).

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the influence of thermal processing on flavor profiles. In addition, the processing of shallot powder has been studied by using fillers in the production of shallot powder using a drum dryer to help stabilize flavor and antioxidants (Setyadjit & Sukasih, 2015). However, effect of different drying methods on phytochemicals of shallot is limited. Volatile compounds are essential in defining the aroma, flavor, and medicinal properties of spices. Key volatiles, such as eucalyptol, β-pinene, and fenchone, contribute to their distinct sensory characteristics while also exhibiting antimicrobial, anti-inflammatory, and antioxidant properties (Sfara et al., 2009; Silva et al., 2012; Singh et al., 2020). However, drying methods can significantly modify volatile compound profiles and bioactivity (Ratseewo et al., 2016; Tian et al., 2021). Examining these changes is essential for optimizing drying techniques, enabling the selection of the most suitable method to preserve desired properties and flavor.

Dehydration is important both for prolonging the shelf life and for processing plants. Most recent studies have been focused on affected drying methods on phytochemicals in other plants. Chumroenphat et al. (2021) investigated the effects of freeze drying, hot air drying, and sun drying on turmeric, observing significant variations in curcuminoid retention and antioxidant activity. Among the methods, sun drying resulted in the greatest curcumin degradation, followed by hot air drying and freeze drying. However, vanillic and ferulic acid contents increased, particularly in hot air- and sun-dried samples. Additionally, total phenolic compounds and antioxidant activity were enhanced after drying. Similarly, Sharma et al. (2015) examined the impact of high-temperature, short-duration drying on onions, reporting that heating at 120 °C for 30 min maximized total phenolic and flavonoid content in red-skinned varieties. Quercetin and its glucosides peaked at 120 °C before declining at 150 °C, while sugar content continuously decreased with increasing temperature. Despite these findings, the impact of these drying methods on shallot, specifically, remains underexplored.

Both the food industry and consumers are interested in antioxidants because of their positive health effects. The number of antioxidants that foods contain when heated to different temperatures should therefore be investigated. Therefore, studies on the effect of different drying methods on polysaccharides, amino acids, volatile compounds, bioactive compounds and antioxidant activities, FTIR analysis including microscope of shallot are still limited. This research aims to investigate the impact of various drying methods on the comprehensive quality parameters of shallot, including polysaccharides, amino acids, volatile compounds, bioactive compounds, antioxidant activities and microscope. By comparing methods such as freeze drying, hot air drying (40 °C, 60 °C and 80 °C), and sun drying, this study seeks to identify the most effective method for preserving the bioactive properties of shallot. The findings of this study will contribute significantly to the food processing industry by providing a guideline on the effect drying method for shallot.

2. Materials and methods

2.1. Chemicals and reagents

The following compounds were purchased from Sigma-Aldrich Co. (St. Louis, MO, U.S.A.): apigenin, rutin, quercetin, kaempferol, and myricetin; standards of phenolic acids, such as sinapic, protocatechuic, caffeic, p-hydroxybenzoic, syringic, vanillic, ferulic, *p*-coumaric, chlorogenic, and gallic acids. Flavonoid standards, including apigenin, rutin, quercetin, kaempferol, and myricetin, were used in the study. Polysaccharide standards were sourced from Sigma-Aldrich Co. (St. Louis, MO, USA). Additionally, Sigma-Aldrich Co. (St. Louis, MO, USA) supplied the amino acid standards, which comprised lysine, histidine, leucine, phenylalanine, tryptophan, valine, arginine, isoleucine, methionine, and threonine. Merck provided HPLC-grade acetonitrile, methanol, and acetic acids (Darmstadt, Germany). Other solvents and substances of an analytical grade were used in this study.

2.2. Sample preparation

Shallots were purchased from a local farm of shallot in Yang Choom Noi District, Sisaket Province, Thailand: *Latitude: 15.1186° N, Longitude: 104.3221° E.* Shallot bulbs were collected according to their similar physical qualities (e.g., size). The second thin layer of the skin were cleaned and wiped dried with sanitary paper towels. After that, the shallots bulbs were sliced and sorted in 4 °C in aluminum foil bag for further analysis. The sliced shallot bulbs were divided into six portions. One portion was kept fresh and used as the control sample (Fresh). The remaining five portions were subjected to different drying methods: hot air drying (HA) at 40 °C, 60 °C, and 80 °C; freeze drying (FD); and sun drying (SD). The samples were separated for three samples of another treatment.

2.2.1. Hot air drying (HA)

Three shallot samples were dried by HA using a hot-air oven (UFE 600, Memmert, Memmert Company, Germany). The drying methods have been modified and described by Ratseewo et al. (2020). The drying conditions involved a set-air velocity of 1.5 m/s at 40 °C, 60 °C, and 80 °C (drying temperature) for 24 h, 20 h, and 16 h, respectively. The samples were dried until the moisture content was 3 % (db). These samples were recorded as HA40, HA60 and HA80.

2.2.2. Freeze drying (FD)

Shallot slices were freeze-dried using a Scanvac CoolSafe (model 100–9 Pro, LaboGene ApS, Denmark) device. Before being dried in equipment, the shallot samples were frozen outside of the freeze-dryer at −50 °C for 12 h. They were then moved to a heating plate and cold trap, where they were chilled to −100 °C while an absolute *v*acuum of 20 Pa was applied. Before being evaluated, the freeze-dried samples were kept in sealed bags. These samples had an FD record.

2.2.3. Sun drying (SD)

Stainless steel racks were subjected to six days of solar drying at an average temperature of 36 °C and relative humidity (RH) of 46–50 % (±1 %) for 2–3 days. The samples were kept in sealed bags in storage at 4 °C.

2.3. Sample extraction

The extraction process was done, with minor modifications, according to Chumroenphat et al. (2021). Briefly, one gram (ground) of sample for each sample was extracted three times with 20 ml of 80 % methanol at a ratio of 1:20 (*w/v*) and shaken at 150 rpm at room temperature for 12 h. The extracts were used for the determination of total antioxidant activity, total phenolic content and total flavonoid content.

2.4. Determination of total phenolic content

The total phenolic content (TPC) in shallots was assessed using the Folin–Ciocalteu method, following the procedure outlined by Ratseewo et al. (2022). The TPC results were expressed in milligrams of gallic acid equivalents (mg GAE) per 100 g of dry weight (DW). All experiments were conducted in triplicate.

2.5. Measurement of total flavonoid content

The total flavonoid content (TFC) in shallots was determined through a colorimetric method, as described by Ratseewo et al. (2020). The TFC values were reported as milligrams of rutin equivalents (mg RE) per 100 g of dry weight (DW), and the analyses were repeated three times for accuracy.

2.6. Evaluation of antioxidant activity

2.6.1. DPPH radical scavenging assay

The antioxidant potential of shallots was assessed using the 2,2-diphenyl-1-picrylhydrazyl (DPPH) radical scavenging method, following the procedure of Ratseewo et al. (2020). A 0.1 ml sample of the extract was combined with 3 ml of DPPH solution (0.001 M in methanol), and the mixture was incubated in the dark at room temperature for 30 min. The absorbance was then recorded at 517 nm using a Synergy HT microplate reader (BioTek Instruments, USA). All measurements were performed in triplicate, and the percentage of DPPH radical inhibition was calculated using the formula:

$(\%) = [1 - (A(\text{sample}) - A(\text{control}))] \times 100.$

2.6.2. Ferric reducing antioxidant power (FRAP)

The FRAP values of shallots was determined using the method described by Kubola and Siriamornpun (2008). The FRAP reagent was freshly prepared by mixing acetate buffer (300 mM, pH 3.6), TPTZ solution (10 mM TPTZ in 40 mM HCl), $FeCl_3{\cdot}6H_2O$ (20 mM), and 12 ml distilled water in a 10:1:1 ratio. The mixture was then warmed to 37 °C. A 60 µl sample extract was combined with 180 µl of the prepared FRAP reagent. After a 4 min incubation at 37 °C, the absorbance was recorded at 593 nm using a Synergy HT microplate reader (BioTek Instruments, USA). All analyses were conducted in triplicate. FRAP *v*alues for the rice samples were calculated using a $FeSO_4$ standard curve and expressed as µmol Fe(II) per gram of dry weight (g DW).

2.7. Determination of phenolic compounds and flavonoids by HPLC

Briefly, one gram of ground sample for each sample was extracted three times with 10 ml of 80 % methanol acidified with 1.0 N HCl (85,15, *v*/v) at a ratio of 1:10 (*w/v*) and shaken at 180 rpm at room temperature for 2 h (Ratseewo et al., 2020). After filtration, the mixed filtrates were evaporated to dryness under vacuum at 40 °C. The quantification and identification of phenolic acids and flavonoids were carried out using high-performance liquid chromatography (HPLC). The system used consisted of Shimadzu LC-20 AC pumps, an SPD-M20A diode array detector (DAD), and an Inetsil ODS-3 C18 column (4.6 mm × 250 mm, 5 µm) (Hichrom Limited, Berks, UK). Gradient elution was performed following the method outlined by Ratseewo et al. (2020). UV-diode array detection was set at 280 nm for phenolic acids and 370 nm for flavonoids. Individual phenolic acids and flavonoids were identified by comparing their retention times and UV absorption spectra to those of authentic standards. All analyses were performed in triplicate.

2.8. Extraction and determination of amino acids

Amino acids were extracted from shallot samples following the method of Chumroenphat et al. (2021) and analyzed using a Shimadzu LCMS-8030 triple quadrupole mass spectrometer in electrospray ionization (ESI) mode, with modifications from Chumroenphat et al. (2021). The ground samples (200 mg) were extracted with 10 ml of methanol for 3 min using a sonication probe (50 % amplitude, 50 s on, 50 s off cycle) (Sonics VCX-750, Vibra cell®, Sonics and Materials Inc., Newtown, CT, USA). After centrifugation at 19,000 ×*g* for 10 min, the pellet was re-extracted. The supernatants were then evaporated under vacuum at 40 °C. This residue was re-dissolved in 5 ml of methyl alcohol. The mixture was filtered via a 0.22 µm nylon membrane before LC/MS/MS analysis. A Shimadzu HPLC system was used under the following conditions: flow rate of 0.2 ml/min, autosampler at 4 °C, and column oven at 38 °C. The mobile phases included (A) demineralized water with 0.1 % formic acid and (B) 50 % methanol in demineralized water with 0.1 % formic acid. Amino acid identification was based on comparison with ten authentic standards, including threonine, isoleucine, tryptophan, and others. All analyses were performed in triplicate.

2.9. Determination of polysaccharide

Sugars were extracted in triplicate following the method of Sharma et al. (2015) with slight modifications. About 1 g of shallot (ground) was mixed with 50 ml of 80 % ethanol and refluxed for 1 h. After filtration through a Buchner filter, the *v*olume was readjusted to 50 ml with 80 % ethanol. The samples were then concentrated below 50 °C using a rotary evaporator under reduced pressure. The concentrated extracts were diluted with 10 ml of water and stored at 20 °C. Before analysis, the extract was filtered through 0.2 µm syringe filters. For analysis, 20 µl of extract was injected into an Agilent Zorbax carbohydrate column (150 mm × 4.6 mm) protected by an Agilent NH_2 precolumn. The elution was performed with acetonitrile:water (75,25, *v*/v) at 30 °C and a flow rate of 1 ml/min. The analysis used a refractive index detector and a Shimadzu 10 A-VP series chromatograph with a Rheodyne manual injector. Chromatograms were processed using Shimadzu Class-VP software, and each injection was repeated two to three times.

2.10. Determination of volatile compounds

The samples were ground, and 0.2 g was placed in vials, which were sealed with aluminum rubber septa (Supelco, Bellefonte, PA, USA) for headspace analysis (Ratseewo et al., 2016). GC–MS analysis was performed using a Shimadzu GC-2010 chromatograph paired with a GC/MS-QP2010 (Shimadzu, Japan). The samples were run through Rtx-5Ms and Rtx-5 fused-silica capillary columns (30 m length, 0.25 mm internal diameter, 0.25 µm film thickness, Restek, PA, USA). Helium served as the carrier gas at a constant pressure of 134.2 kPa. The injector was set at 250 °C with a split ratio of 1:5. The temperature program started at 80 °C, ramped to 250 °C at 10 °C/min, and was held for 2 min. The ion source temperature was 200 °C, transfer line 250 °C, with ionization energy at 70 eV, and mass spectra were recorded in the range of 35–550 u.

2.11. FTIR analysis

Shallot sample FTIR spectra were obtained with an FTIR instrument (Frontier) fitted with a Diamond/KRS-5 crystal composite (1 bounce) and a UATR accessory (Perkin Elmer, USA). Without requiring any additional processing, a turmeric powder could be analyzed directly using UATR-FTIR spectra. The spectral data encompassed the 4000–400 cm^{-1} spectral band and were obtained from 32 scans with a spectral resolution of 4 cm^{-1}. A force gauge of 110 units was used to measure all of the samples. The program also captured and automatically removed a background spectrum.

2.12. Microstructure of shallots

A scanning electron microscope (SEM) (Hitachi, TM4000plus, Japan) fitted with the TM 4000plus program was used to assess structural alterations. The shallots underwent vacuum mode SEM examination after being coated with gold.

2.13. Statistical analysis

SPSS software was used to calculate the means, standard deviations (SD), and perform one-way ANOVA on both unheated and processed samples. Each experiment was conducted in triplicate. Duncan's test was applied to determine significant differences, with a confidence level of 95 % ($p < 0.05$).

3. Results and discussion

3.1. The total phenolic content (TPC)

The total phenolic content of dried shallots is presented in Fig. 1A.

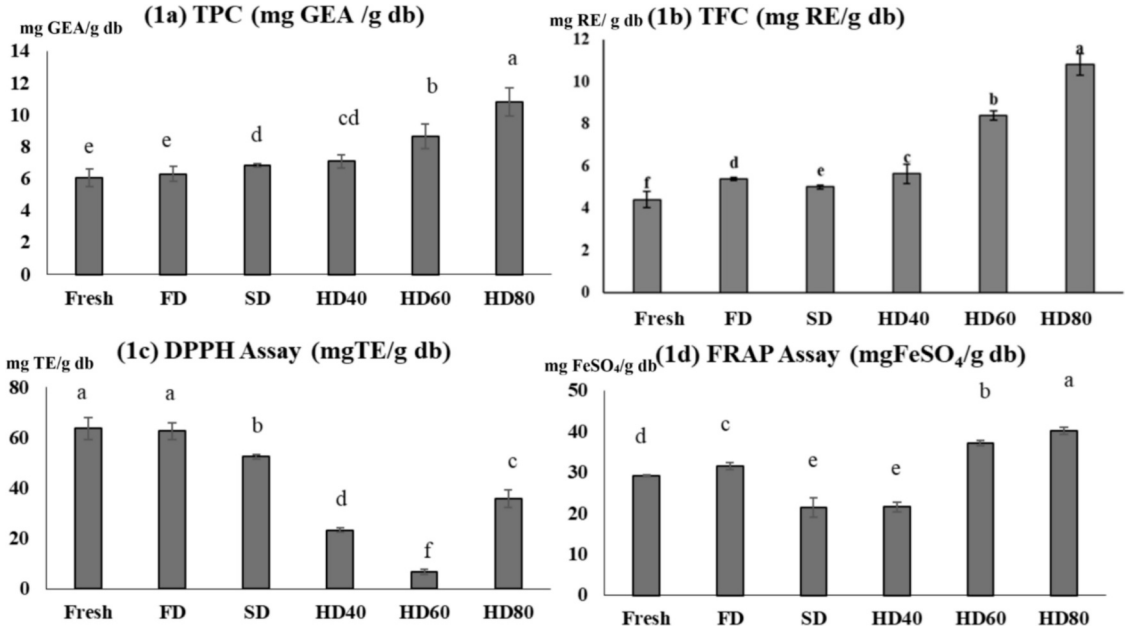

Fig. 1. Effect of different drying methods on TPC (1a), TFC (1b), DPPH (1c) and FRAP (1d) of shallots. Different letters significantly different at $p < 0.05$ within each sample.

The result found that sample HD80 (10.83 mg GEA/g db) had the highest content of phenolics, followed by HD60, while fresh shallot had the lowest TPC. The FD sample was not significantly TPC. While TPC was significantly increased after heating methods of shallot, for instance, the dried shallot, HD80 increased by 43.95 %, followed by HD60 (29.99 %) when compared to fresh shallot. Thuy et al. (2019) reported that dried shallot had a high content of TPC at 50 °C (13.52 mg GAE/g). Similarly, TPC was increased in a study by Abdou Bouba et al. (2012) that studied the drying of onions using solar drying, which had a higher total phenolic content when compared to fresh onions. The increase in total phenolic content (TPC) of shallots after high-temperature drying is consistent with previous findings. Sharma et al. (2015) reported a significant increase in phenolic content in high-temperature dried onions, indicating that heat treatment promotes the release of phenolic compounds. Different heat treatment methods, such as boiling, stir-frying, frying, and roasting, have been demonstrated to release phenolic compounds by cleaving ester bonds and glycosylation or generating Maillard reaction products, which contributed to the observed increase in phenolic content. Similarly, Ratseewo et al. (2020) also showed that heat exposure degrades cell wall components, including pectin, protein, and fiber, or causes their structure to collapse, resulting in the release of bound phenolic compounds. These findings suggest that high-temperature drying increases TPC in shallots via the degradation of cell structure and the formation of new phenolic derivatives during heating. Freeze-drying (FD) did not significantly increase the total phenolic content (TPC) due to low temperature and vacuum conditions, which inhibited the activity of polyphenol oxidase (PPO) and oxidative transformation. Although FD effectively preserved the biological compounds, reduced degradation and maintained the structure of the samples, it could preserve the heat-sensitive and oxidizable compounds, resulting in a greater retention of TPC than the addition of TPC, and the effect on TPC varied depending on the characteristics of each plant species (Chumroenphat et al., 2021; Yang et al., 2024).

3.2. The total flavonoid content (TFC)

Flavonoids are an important groups of polyphenol compounds because of its potential pharmacological and biological activities such as anti-inflammatory, anti-cancer, anti-viral and antioxidant (Ferreira et al., 2016). Changes in the total flavonoid contents of shallots as affected by dehydration treatments are shown in Fig. 1B. The result found that HD80 had the highest total flavonoid content, followed by HD60 and HD40, while fresh shallot had the least amount of TFC. Total flavonoid content was significantly increased in all dried shallots when compared to fresh shallot. Total flavonoid content increased after heating methods were used. This is in agreement with the research of Sharma et al. (2015), who studied onion drying by hot air drying at 120 °C for 30 min. The highest total flavonoid content was observed in red onion cultivars treated by hot air drying (120 °C). Thermal drying of plant samples affected these compounds. This indicated that these compounds can be released when the cell wall structures have broken covalent bonds (Ratseewo et al., 2020), like flavonoids. The formation of Maillard reaction products or the glycosylated bonds and esterified may contribute to the enhance in TPC and TFC after heating, and the compounds may depend on the plant cultivar (Aoyama & Yamamoto, 2007). The Maillard reaction can occur even at low drying temperatures (40–80 °C) during hot air drying, leading to the formation of intermediate Maillard reaction products. Previous studies have reported the presence of these compounds at significant levels, up to 55.8 mg/kg under similar conditions, namely 40–60 °C hot air drying (Tang et al., 2023). These Maillard reaction products possess antioxidant properties, which may contribute to the observed increase in total phenolic content (TPC) and total flavonoid content (TFC) (Nooshkam et al., 2019).

3.3. Antioxidant activities

The antioxidant activities of dried shallots, as affected by various dehydration methods, were determined by DPPH radical scavenging activity and FRAP assay (Fig. 1C and B, respectively). DPPH radical scavenging activity in shallot samples was found that fresh and FD sample showed the highest inhibition of DPPH radical, while other dried samples, i.e. SD, HD40, HD60 and HD80, had lower the DPPH radical scavenging activity. In this study, this result suggested that the FD is one of the treatments that have retain the antioxidant activity using DPPH assay when compared to fresh shallot. Antioxidant activity by FRAP assay of dried shallot samples was found that HD80 (40.18 mg $FeSO_4$/g db) had the highest antioxidant activity, followed by HD60 (37.26 mg $FeSO_4$/g db), FD (31.64 mg $FeSO_4$/g db) and Fresh (29.19 mg $FeSO_4$/g

db) (Fig. 1D), while other dried samples, SD and HD40, had lowest FRAP activity. Dried shallots treated with high-temperature dryings (HD80 and HD60) significantly increased their antioxidant activity by FRAP assay, approximately 27.35 % and 27.66 %, respectively. The results of the study were similar to those of the flavonoid content in dried shallots, which showed that HD80 and HD60 had high TFC. This compound is one of antioxidant effect in dried shallots. Fresh shallot was observed to have a to have a high content of bioactive compounds, especially phenolics and flavonoids, which have strong antioxidant activity. A similar result was reported in dried onion, which presented the highest antioxidant activity by the FRAP assay at high temperatures (Sharma et al., 2015). Many research studies focus on drying methods or specific bioactivities that can guide the optimal selection of drying techniques for dried plants. Another study by Chumroenphat et al. (2021) investigated the effects of freeze drying and low-temperature drying on turmeric and found substantial differences in the retention of curcuminoids and antioxidant activity. Sharma et al. (2015) reported the decrease in antioxidant activity of dried onion by DPPH and FRAP assays at 150 °C deepened on onion variety. The increase in TPC and TFC, we have further explained the increase. As for DPPH, the difference in results may be explained because the DPPH method is different from FRAP, resulting in values that do not follow the trend. The mechanism of each assay varies based on the chemical properties and function of the target compound. DPPH is a stable free radical commonly used to evaluate free radical scavenging capacity, whereas the FRAP assay measures the reducing potential of antioxidants by reacting with a ferric tripyridyltriazine (Fe^{3+}–TPTZ) complex, forming a colored ferrous complex (Kubola & Siriamornpun, 2008). There are many methods for measuring antioxidant activity. Each method of analysis has different ways of inhibiting free radicals, resulting in different expressions of the value of inhibiting free radicals Ratseewo et al., 2020).

3.4. Phenolic acids

Study of composition and concentration of phenolic acids, including gallic acid, protocatechuic acid, *p*-hydroxybenzoic acid, vanillic acid, chlorogenic acid, caffeic acid, syringic acid, ferulic acid, sinapic acid and *p*-coumaric acid by HPLC according to a method minor modified from Ratseewo et al. (2020) was present in Fig. 2. The group of phenolic acids that were found in the highest amounts in all shallot samples was *p*-coumaric acid (143–714 μg/g), syringic acid (69–639 μg/g), and caffeic acid (48–278 μg/g). The results explore that most samples of shallots detected these phenolic acids except for sinapic acid, which was not detected only in the HD60 and HD80 dried shallots. In the case of fresh shallot, gallic acid, caffeic acid, and sinapic acid were observed to have the highest content in fresh shallots, while when dying treated with shallots, their content was significantly decreased. FD revealed the highest amounts of chlorogenic acid, syringic acid, and *p*-coumaric acid among all samples, with an increase in concentration from the fresh sample of approximately 1.67, 1.99, and 1.68 times, respectively. While drying with other heat methods resulted in a decrease in the number of compounds. Protocatechuic acid and ferulic acid showed similar increases in FD, although not to the greatest extent among all the samples. In the case of hot air drying, HD80 had the highest vanillic acid and protocatechuic acid compared to all samples, with approximately 2 and 12.54 times more increases than fresh samples, respectively. Protocatechuic acid and ferulic acid were also increased after all drying methods. The study also demonstrated that the HD40 sample had the greatest *p*-hydroxybenzoic acid content of any sample, having grown 1.28 times from the fresh sample. Moreover, the results from this study also showed that the HD40 sample showed the highest content of ferulic acid in among all samples and increased by 8.85 times from the fresh sample. The results of this study suggested that drying at both low and high temperatures, including freezing, was the cause of the rise in a content of several phenolic acids. The results were consistent with the study of changes in phenolic acids in hot air drying of rice at 60 °C (Ratseewo et al., 2020). Additionally, after heat treatment in food processing, protocatechuic acid was discovered increasing in this study. The largest amount was identified in shallot samples that were dried at 80 °C. This may be explained by the partitioning of vanillin (vanillyl alcohol) and vanillic acid. The reaction of the precursor, veratraldehyde, with heat produces vanillin, vanillic acid, and protocatechuic acid. The partition may suggest that heat treatment is capable of converting vanillin or veratraldehyde into vanillic acid and protocatechuic acid (Converti et al., 2010). Moreover, phenolic compounds, which are usually present in plants, are a group of compounds with a wide variety of forms and large molecular sizes. It is possible to break down cell walls and release these bioactive chemicals through both cold and heat processing. The drying processes have the potential to break weak chemical or covalent bonds from the delivery polymer or to degrade big molecular weight bioactive components like lignin, reducing them to small molecules. Thus, there is an increase in content of some phenolic acids while some bioactive compounds reduce (Ratseewo et al., 2020).

3.5. Flavonoids

The comparative analysis between drying methods namely; freeze drying, hot air drying and sun drying methods of shallots reveals distinct advantages for each approach (Fig. 3). The results showed that quercetin, rutin, apigenin and kaempferol were the most predominant flavonoids in fresh shallot. The high concentration of quercetin in shallot was also reported (Ruksiriwanich et al., 2022). This study assessed the flavonoid content in shallots samples to various drying methods, highlighting significant differences and improvements in preserving these compounds. The HPLC analysis revealed substantial variations in flavonoid content across different drying methods, exhibition the advantages of FD and HD at various temperatures. HD40 exhibited the highest rutin content at 107.94 ± 9.00 μg/g, followed by those HD80 with 85.31 ± 1.35 μg/g. In contrast, fresh shallots had significantly lower rutin levels at 19.65 ± 8.26 μg/g, while freeze-dried shallots contained 24.32 ± 1.59 μg/g, and sun-dried shallots had 23.16 ± 0.86 μg/g. The FD method is higher in preserving heat-sensitive compounds like quercetin, significantly outperforming fresh, SD, and HD methods. For apigenin content, HD80 showed exceptionally high levels at 1779.37 ± 313.56 μg/g, with significant amounts also found in HD60 samples at 509.70 ± 25.53 μg/g. Fresh shallots had much lower apigenin levels at 12.30 ± 0.98 μg/g, sun-dried shallots contained 11.66 ± 0.44 μg/g, and apigenin was not detected in freeze-dried samples. High temperature drying methods (HD80 and HD60) significantly enhance the content of specific bioactive compounds such as apigenin, surpassing fresh and SD methods.

Rutin was presented the highest content in HD40 followed by HD80 and HD60, respectively, which showed the higher content when compared to fresh shallot. Quercetin has the highest content in FD followed by HD80 and SD, which significantly increased to approximately 9.30, 5.34 and 3.91 times, respectively. Hot air drying showed the positive effect on apigenin. Apigenin was observed that has the highest content in HD80 followed by HD60 and HD40, with was also significantly increased from fresh shallot approximately 144.66, 41.44, 1.48 times, respectively. Myricertin was not detected in all samples, while kaempferol was found in all samples. Kaempferol was significantly increased after drying in only HD40 samples (1.12 times) when compared to fresh shallot, while it was significantly decreased in the rest samples. The result was found that HD40 dried shallot had the remarkably significantly highest content of rutin (5.49 times) and kaempferol (1.48 times) when compared to all samples, respectively.

The improvements observed with FD and HD methods have significant practical implications for the food industry, particularly regarding the preservation and enhancement of flavonoids in shallots. Flavonoids such as rutin, quercetin, and apigenin are known for their potent antioxidant properties, which contribute to the health benefits of shallots (Ruksiriwanich et al., 2022). By effectively preserving and enhancing

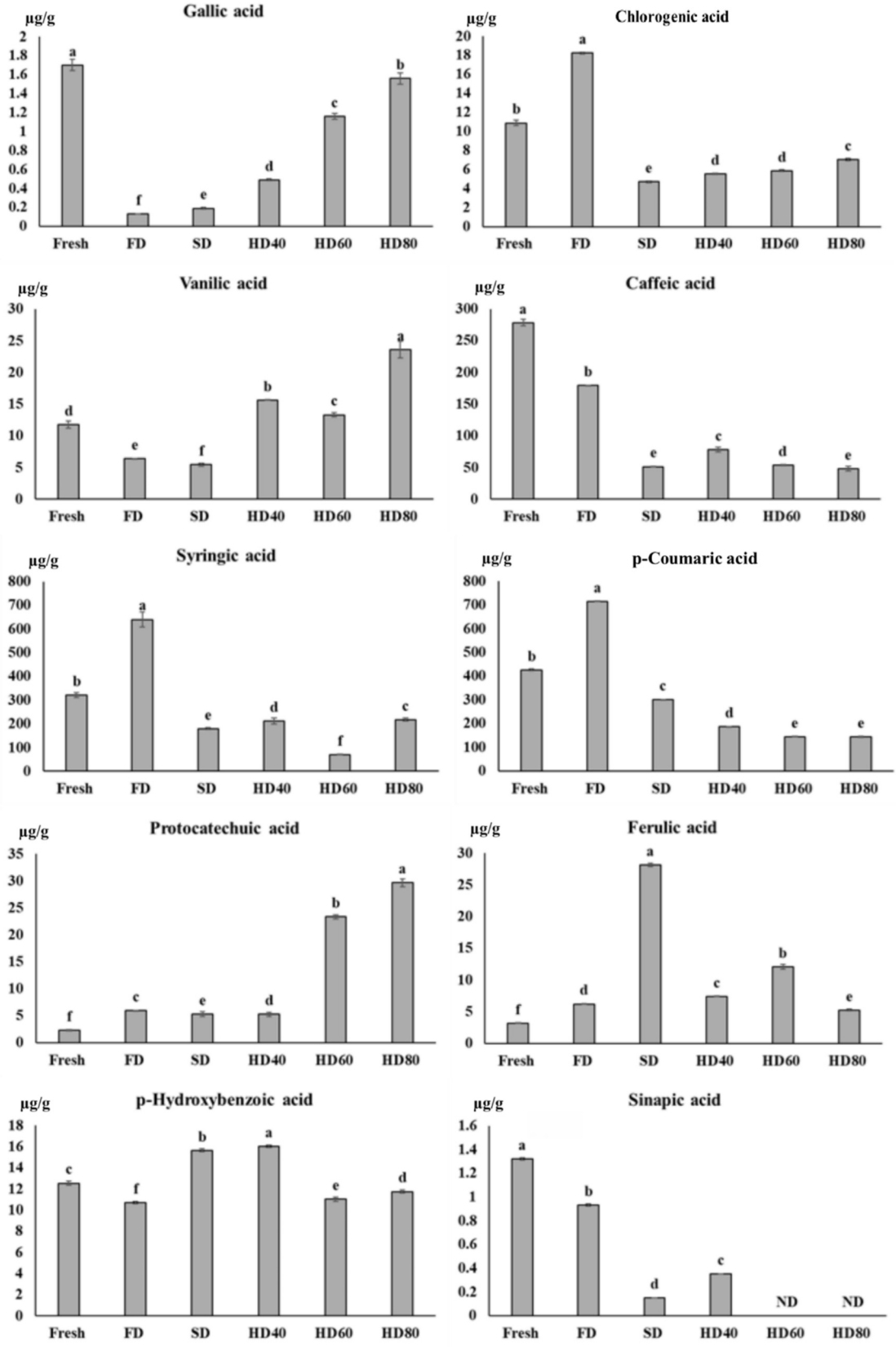

Fig. 2. Effect of different drying methods on phenolic acids of shallots. Different letters significantly different at $p < 0.05$ within each sample.

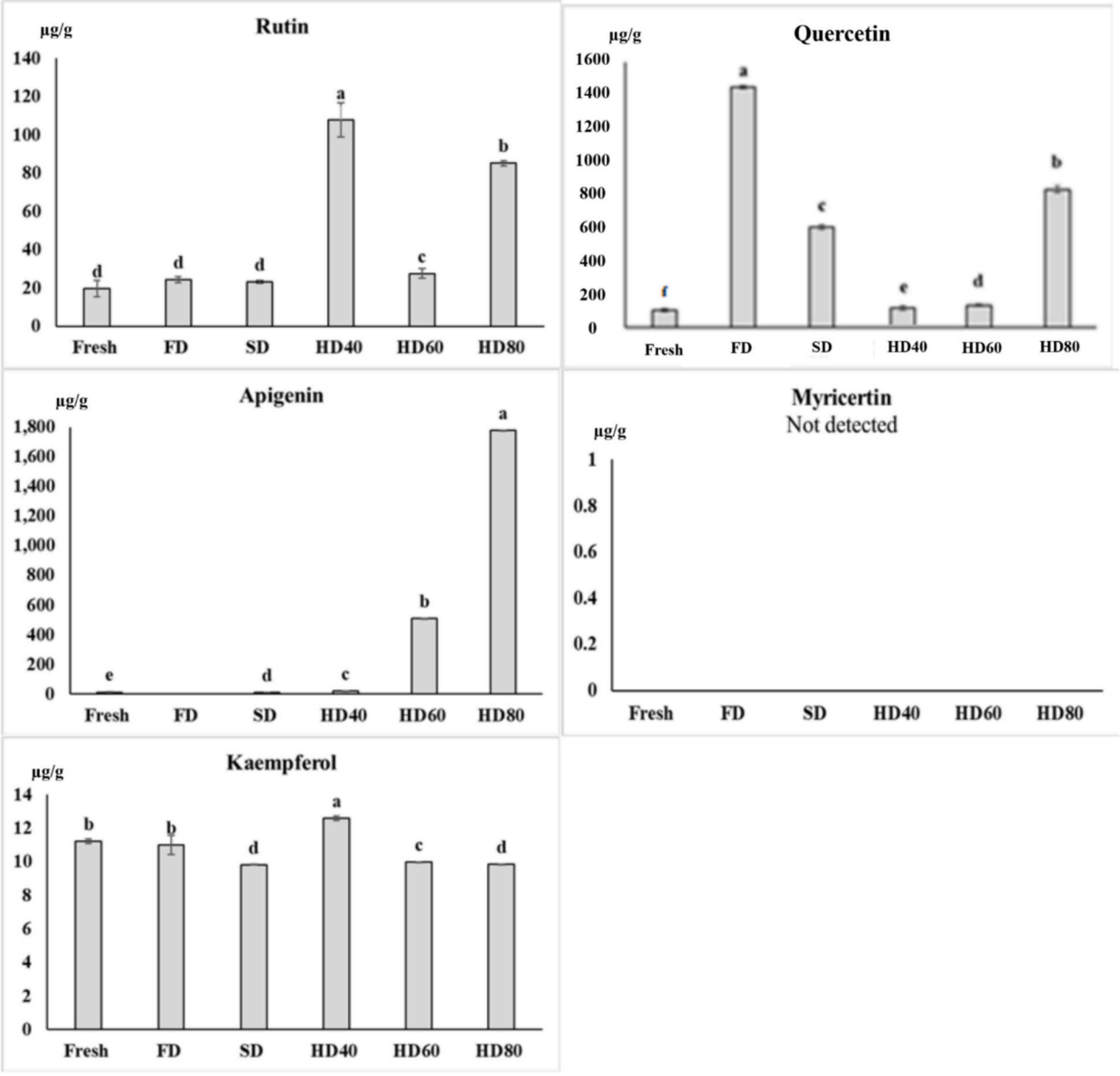

Fig. 3. Effect of different drying methods on flavonoids of shallots. Different letters significantly different at $p < 0.05$ within each sample.

these compounds, FD and specific HD methods ensure that the nutritional quality of dried shallots remains high. This translates to better health benefits for consumers, including potential anti-inflammatory, angiogenesis pathways (Ruksiriwanich et al., 2022) and anti-carcinogenic properties (Ravindranath et al., 2023). Enhanced preservation of flavonoids also means that dried shallots can have a longer shelf life while retaining their beneficial properties (Sharma et al., 2015). Both the industry and consumers will benefit together in terms of increasing the value of shallots and meeting the increasing consumer demand for health-promoting, beneficial foods. The findings suggest that specific drying methods can be optimized to target the preservation of particular flavonoids. For instance, using hot air drying at 40 °C can maximize rutin content, while freeze drying is ideal for preserving quercetin. This allows producers to tailor their processing techniques to create products that meet specific health-oriented market demands. In conclusion, the study demonstrates that the choice of drying method significantly impacts the preservation and enhancement of flavonoids in shallots. FD is particularly effective for preserving quercetin, while HD at specific temperatures (40 °C and 80 °C) significantly enhances rutin and apigenin content. These insights are crucial for the food industry, providing guidelines for optimizing drying processes to produce dried shallot products with enhanced health benefits. The practical benefits of these drying methods are clear, making them valuable tools for developing superior dried food products that meet the increasing consumer demand for nutritious and functional foods. In our present study, quercetin is a flavonoid found in shallot that is stable to processing. Food processing has both positive and negative effects on food samples. It also affects their physical and chemical properties, bioactive compounds and antioxidant activity. The effects of heat processing or trimming on fruit and vegetable samples are that they release oxidizing and hydrolytic enzymes, such as PPO (polyphenol oxidase), which have the ability to destroy phenolic compounds, resulting in a decrease in the amount of phenolics (Lv et al., 2017). However, processing with the ability to disrupt cellular constituents, such as the plant cell wall, results in the release of various substances from the cells, such as antioxidants, and heat can also disrupt the unstable covalent bonds of some bioactive compounds, transforming them into derivatives with the same antioxidant activity (Lv et al., 2017).

3.6. Amino acids

This study provides a comprehensive analysis of the effects of different drying methods on the amino acid contents of shallot (*Allium ascalonicum* L.). As shown in Table 1, it was found that proline was the amino acid with the highest amount in fresh shallot samples, while cysteine was not found in both fresh and processed shallot samples. The group of amino acids found higher in the fresh sample than in the processed sample included asparagine, glutamine, glutamic acid, histidine, isoleucine, leucine, lysine, phenylalanine, serine, threonine, tryptophan, and tyrosine. These amino acids decreased in quantity after drying. Some amino acid groups were increased in the dried samples, such as arginine, which was increased in the sun-dried shallot samples. The amount of alanine increased by 34 % when dried in hot air at 40 °C. By 32 %, methionine was found to increase when dried with hot air at 60 °C. The amount increased by 29 %, and proline was found to increase the content of shallots dried in SD. The amount increased by 24 %, while the amount of glycine was found to increase by 107 %. The amount increased when dried in hot air at 40 °C. However, glycine, though present in higher amounts, was among the least common amino acids in shallot samples (1.76 mg/100 g db). In contrast, proline was the most abundant amino acid in both fresh (2178 mg/100 g db) and dried shallots (2708 mg/100 g db). The decrease in amino acid content may be due to the Maillard reaction, in which reducing sugars and amino acids are reactants and result in the formation of brown products (Tang et al., 2023), in which the reduction of this amino acid was found in other studies have shown a reduction in lysine (43 %) and histidine (41 %) in HD and FD turmeric samples (Chumroenphat et al., 2021) and in HD colored rice (Ratseewo et al., 2020). Various Thai rice found that drying with HD60 decreased the amount of all amino acids, especially methionine, by 89 % (Ratseewo et al., 2020). The study by Chumroenphat et al. (2021) demonstrated that freeze-drying and low-temperature drying methods affected the degradation of all amino acids. HD40 significantly increased the levels of alanine and glycine, highlighting its potential for enhancing these amino acids. SD and HD80 were particularly effective in retaining high levels of arginine and proline, crucial for nutritional and functional properties. Different drying methods significantly impact the amino acid content of shallots. HD at specific temperatures can enhance or preserve certain amino acids. Certain amino acids may be concentrated due to reduced water content after drying. The reduction in certain amino acids, such as lysine and alanine, is consistent with the Maillard reaction observed in other studies, where amino acids and reducing sugars form brown products (Chumroenphat et al., 2021; Ratseewo et al., 2020). The findings indicate that FD, SD, and HD significantly impact the amino acid content, with specific methods favoring the retention or enhancement of certain amino acids. This aligns with the findings in this study, where FD maintained higher levels of certain amino acids compared to HD and SD.

3.7. Polysaccharide

Polysaccharide content for a major portion of the dried shallots by different drying methods namely glucose, fructose, sucrose, and fructooligosaccharides was presented in Fig. 4. In this study, fructose (2199 mg/g db) was found to be the highest, followed by sucrose (1077 mg/g db), glucose (999 mg/g db), stachyose (829 mg/g db) and fructooligosacharide (688 mg/g db), respectively, while mannose, xylitol and raffinose were not found in fresh shallot samples. The fructooligosacharide content was the highest in fresh shallot samples, followed by FD and HD80 samples. After drying, the fructooligosacharide content was significantly reduced, with FD (78.51 %), followed by HD80 (83.9 %), while SD was the most reduced at 89.53 %. Stachyose was the highest in fresh samples. After drying, stachyose content was significantly reduced in all treatments. Among the drying methods, FD resulted in the highest retention of stachyose, with a reduction of approximately 64.64 %, followed by HD80 with a 75.29 % reduction. The greatest decrease in stachyose was observed in SD and hot air HD60, with reductions of about 86 %. Sucrose followed a similar pattern to fructooligosaccharides and stachyose, with the highest concentration found in fresh samples, followed by FD and HD80. The FD method retained the highest sucrose content, showing a reduction of around 53.84 %, whereas the HD60 method resulted in the greatest sucrose loss at approximately 82.85 %. Monosaccharides, including fructose and glucose, also exhibited similar trends. Fresh samples contained the highest amounts, followed by FD, while HD60 resulted in the lowest concentrations. Glucose content in FD and HD80 samples decreased by 53.36 % and 76.45 %, respectively, while fructose content decreased by 64.11 % in FD samples and 72.46 % in HD80 samples. However, mannose, xylitol and raffinose were not found in the shallot samples in both fresh and dried samples. The results of this study indicated that the

Table 1
Effect of different drying methods on amino acids of shallots.

Amino acid Content	Sample name					
(mg/100 g db)	Fresh	FD	SD	HD40	HD60	HD80
Alanine	34.66 ± 1.78^{c}	11.34 ± 0.19^{f}	37.77 ± 0.27^{b}	45.60 ± 0.93^{a}	21.15 ± 0.72^{e}	24.76 ± 0.19^{d}
Arginine	547.18 ± 7.45^{d}	493.68 ± 10.39^{e}	755.27 ± 20.36^{a}	244.20 ± 7.92^{f}	628.50 ± 12.23^{c}	691.21 ± 18.69^{b}
asparagine	121.75 ± 3.84^{a}	51.54 ± 2.81^{c}	60.38 ± 3.19^{b}	48.08 ± 2.84^{d}	38.35 ± 3.82	49.48 ± 2.92^{d}
aspartic acid	87.22 ± 1.77^{a}	12.26 ± 0.63^{d}	13.87 ± 0.77^{c}	15.08 ± 0.65^{b}	8.81 ± 1.15^{f}	10.84 ± 0.53^{e}
Cysteine	ND	ND	ND	ND	ND	ND
glutamine	592.07 ± 9.03^{a}	173.29 ± 2.01^{c}	205.31 ± 2.56^{b}	163.04 ± 2.93^{d}	133.73 ± 2.22^{e}	72.75 ± 1.56^{f}
glutamic acid	253.21 ± 6.23^{a}	33.49 ± 1.30^{d}	51.07 ± 1.39^{c}	55.28 ± 1.98^{b}	35.78 ± 2.14^{d}	27.45 ± 0.28^{e}
Glycine	0.85 ± 0.01^{c}	0.12 ± 0.00^{e}	1.71 ± 0.02^{b}	1.76 ± 0.02^{a}	0.16 ± 0.00^{d}	0.11 ± 0.00^{f}
Histidine	108.43 ± 3.33^{a}	42.79 ± 0.69^{d}	41.16 ± 1.33^{d}	49.28 ± 1.23^{b}	46.92 ± 1.48^{c}	27.54 ± 1.67^{e}
isoleucine	339.94 ± 5.34^{a}	284.81 ± 2.65^{b}	227.38 ± 8.35^{d}	235.94 ± 4.40^{d}	251.25 ± 10.20^{c}	217.64 ± 3.24^{e}
Leucine	291.95 ± 6.04^{a}	244.72 ± 8.34^{b}	193.34 ± 6.48^{de}	199.36 ± 4.22^{d}	214.13 ± 7.73^{c}	185.62 ± 3.11^{e}
Lysine	567.96 ± 7.07^{a}	167.42 ± 3.95^{c}	189.03 ± 1.76^{b}	152.48 ± 2.72^{d}	113.48 ± 2.23^{e}	66.24 ± 1.49^{f}
methionine	184.70 ± 6.17^{b}	175.27 ± 2.91^{c}	134.46 ± 5.41^{e}	109.86 ± 2.75^{f}	239.95 ± 5.38^{a}	151.96 ± 3.84^{d}
phenylalanine	652.28 ± 7.68^{a}	609.45 ± 14.28^{b}	404.11 ± 9.91^{d}	396.75 ± 9.85^{d}	468.84 ± 10.08^{c}	413.07 ± 7.61^{d}
Proline	2178.05 ± 44.61^{d}	2104.18 ± 15.26^{e}	2791.35 ± 54.33^{a}	2497.11 ± 65.87^{c}	2553.38 ± 33.06^{b}	2708.67 ± 54.39^{a}
Serine	98.86 ± 0.63^{a}	26.56 ± 0.25^{d}	37.13 ± 0.42^{b}	31.35 ± 0.37^{c}	24.17 ± 1.38^{e}	30.19 ± 0.94^{c}
threonine	68.18 ± 1.41^{a}	36.72 ± 0.78^{d}	41.75 ± 1.16^{b}	37.13 ± 0.90^{d}	41.55 ± 1.88^{b}	39.55 ± 0.43^{c}
tryptophan	764.68 ± 17.21^{a}	641.71 ± 18.41^{b}	387.44 ± 13.02^{d}	438.51 ± 7.34^{c}	372.46 ± 18.12^{d}	321.77 ± 8.17^{e}
Tyrosine	486.93 ± 9.41^{a}	372.87 ± 6.97^{b}	251.29 ± 1.95^{e}	289.73 ± 8.34^{c}	267.68 ± 11.58^{d}	268.57 ± 5.31^{d}
Valine	118.49 ± 0.99^{b}	127.83 ± 2.75^{a}	97.93 ± 3.36^{d}	103.37 ± 5.92^{c}	120.98 ± 8.74^{a}	94.61 ± 2.64^{d}

Means±SD values on the same row within each variable following by different letters differed significantly ($p < 0.05$).
ND=Not detected.

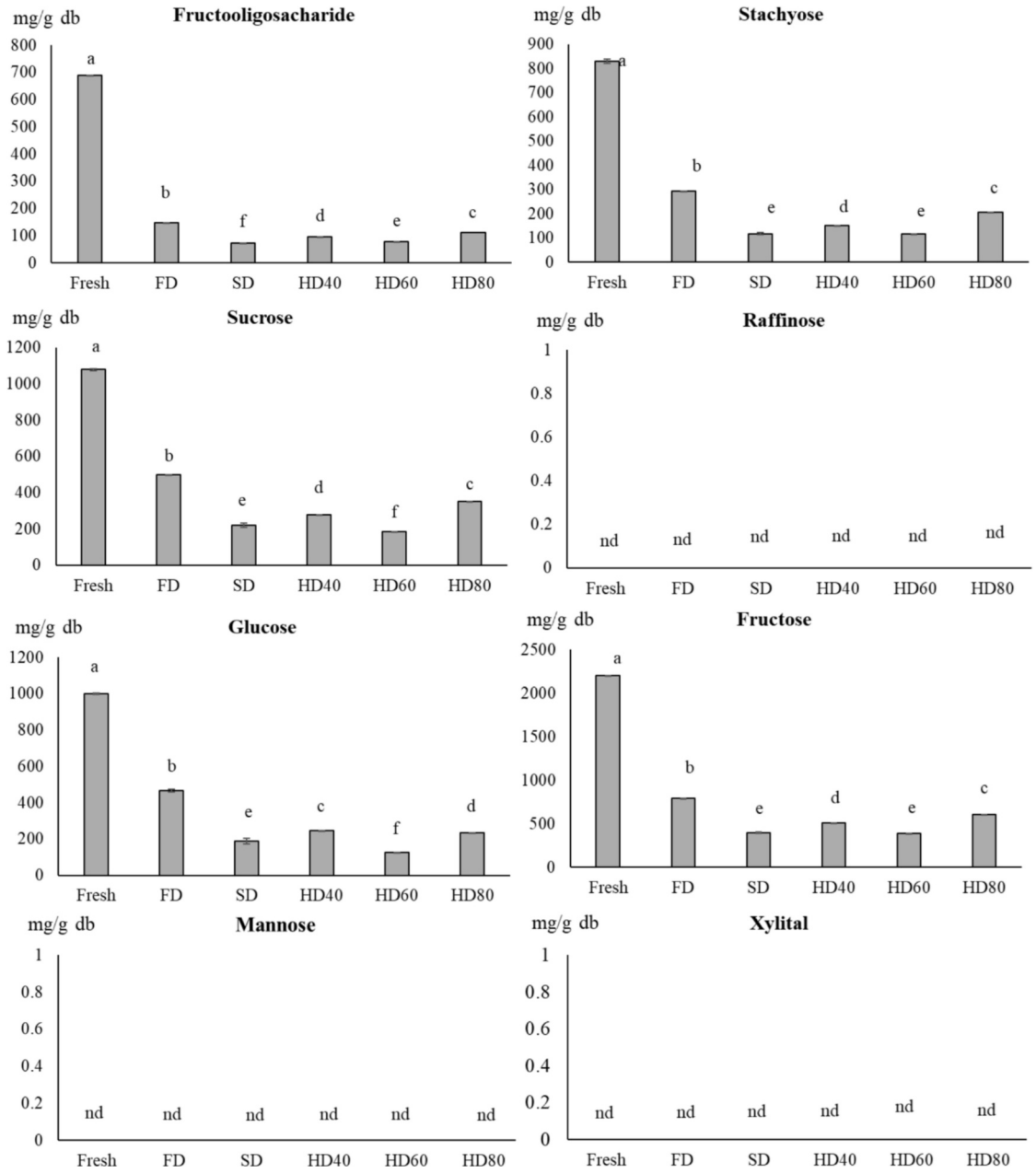

Fig. 4. Effect of different drying methods on sugars of shallots. Different letters significantly different at $p < 0.05$ within each sample.

different drying methods affected the amount of sugar compounds in shallots, with a statistically significant decrease compared to the fresh sample.

The sugar content of shallots was highest in FD samples, indicating that cold drying had the least effect on sugar content reduction compared to other heat drying methods. Heat drying had the least effect on sugar content reduction, with the sample with the highest remaining sugar content being the sample with 80 °C drying temperature. As the heating temperature decreased, a similar trend was observed in all the studied samples (Fig. 4). The polysaccharide content of different plant samples after drying has been studied. The results of this study were in the same direction as that of onion drying where drying resulted in a decrease in the polysaccharide content of onion. Drying of red onion at high temperature revealed a loss of glucose and fructose as high as 74 % and 64 %, respectively Sharma et al. (2015). Earlier studies have indicated that during heating, fructose initially reduces at a quicker rate than glucose, but over time, glucose loss exceeds that of fructose (Reyes-Alvarez et al., 2022). This difference may arise from variations in heating techniques and temperatures. Furthermore, the decline in sugar content is often linked to Maillard reactions, where sugars interact with amino acids and proteins (Martins et al., 2000). For example, in roasted foods like cocoa beans, glucose is completely depleted due to its reaction with amino acids, forming new compounds (Redgwell et al., 2003). The present findings suggest that both heat and cold drying impact the polysaccharide content of shallots, with cold techniques such as freeze drying being more effective at preserving these compounds compared to heat drying. The drying method significantly affects the retention of polysaccharides in shallots. Heat-based drying methods can cause significant losses, largely due to reactions like the Maillard reaction, which reduce sugars such as glucose. On the other hand, cold drying methods, including freeze drying, better preserve polysaccharides by reducing the degradation of heat-sensitive compounds, resulting in higher nutritional value in the dried shallots. This understanding is crucial for the food industry as it seeks to maintain both the health benefits and the quality

of shallot-based products.

3.8. Volatile compounds

The component of volatile compounds in shallot dried by different methods namely sun drying, freeze drying and hot-air drying (HD40, HD60 and HD80) were analyzed using GC–MS and the results are presented in Table 2. The chromatograms of the volatile compounds in dried shallots are shown in Supplementary data (Fig. S1). For instance, eucalyptol was identified as the most abundant volatile compound in fresh shallots, accounting for 61.53 %. Eucalyptol is the organic compound class of monoterpenoids with an ether ring, having a chemical formula of $C_{10}H_{18}O$. It is a colorless to pale yellow liquid with a spicy aroma, insoluble in water but miscible with ethanol, ethers, and chloroform. It is widely used in the food industry, beverages, confectionery, and cosmetics. Additionally, it serves as an insecticide and repellent in medicine and is included in mouthwash, cough medicine, and muscle relaxants (Sfara et al., 2009). Other volatile compounds identified in fresh shallots in descending order include β-pinene (19.96 %), an isomer of pinene with exocyclic double bonds. It acts as a plant metabolite found in essential oils of various plants. In addition, this compound has antimicrobial potential, inhibiting phospholipase and esterase activities, which were evaluated and obtained the best inhibitory results against *Cryptococcus neoformans* (Silva et al., 2012). The compound (1*S*,4*R*)-1,3,3-trimethyl-2-bicyclo [2.2.1] heptanyl] acetate or bornyl acetate was detected at 4.16 %, along with L-fenchone at 1.14 % and fenchol at 0.56 %. Fenchone also has antimicrobial activity, antifungal activity and antioxidant (Singh et al., 2020). In addition to having medicinal properties, these volatile compounds are classified as flavor and fragrance compounds, commonly used in enhancing the aroma and taste of food prepared with shallots or large-headed shallots. Additionally, amino acids and glutamic acids, which enhance food taste, were found. (+)-2-Bornanone or d-camphor at 0.37 %, an isomer of pinene, was also identified, serving as a gum-like substance used in the extraction and production of cough or cold medicines. The results of this study indicate that fresh shallots are a source of essential oils that have both medicinal properties and are flavor enhancers for foods.

The drying process influenced the concentration of certain volatile compounds depending on the method used. Among the drying methods, freeze drying (FD) preserved the highest amount of eucalyptol when compared to all drying methods. Remarkedly, retention of eucalyptol was greater in samples dried at higher temperatures (HD80) compared to those dried at lower temperatures (SD, HD60, and HD40), as shown in Table 2. In this study revealed the increase amount of some compounds after drying in died shallots. β-Pinene was identified in both fresh and dried shallot samples, belonging to the volatile compound group in food oils. It was increased in samples HD40 (34.82 %) and HD60 (25.35 %). Several volatile compounds were not detected in fresh shallot samples, while were detected in dried shallot samples. For example, 2-propanone, 1-hydroxy or dihydroxyacetone phosphate, 2,3-pentanedione and caryophyllene and were increased after treated by thermal drying methods. This study indicates that some drying methods affect the changes of volatile compounds in shallots and result in different flavor characteristics from fresh shallots. The concentration changes of volatile compounds during processing are influenced by various factors, including the cooking method and specific parameters related to the food being cooked (Venskutonis, 1997). In some instances, drying has led to an increase in certain components typical of a spice (Bartley & Jacobs, 2000), or the generation of new compounds after drying, likely due to oxidation reactions, hydrolysis of glycosylated forms, or the release of substances from broken cell walls (Huopalahti et al., 1985). Furthermore, Tian et al. (2021) analyzed the volatile compound changes in fried shallot oil at various frying temperatures, emphasizing the influence of thermal processing on flavor profiles.

3.9. Fourier transform infrared spectroscopy (FTIR) analysis

FTIR was used to investigate the functional groups present in fresh and dried shallots, as shown in Fig. 5. FTIR is widely employed across different industries including medical research and biological studies, particularly for identifying the identity of drugs, raw materials, or synthesized substances. Each peak comes from the absorption of functional groups in shallots. The result was presented peaks at 3290 cm^{-1} (N—H stretching of proteins and O—H stretching of carbohydrates and water) and 2930 cm^{-1} (CH_2 antisymmetric stretch of methyl groups from lipids) indicate higher moisture content in fresh and low-temperature dried shallots compared to high-temperature dried samples (Lu et al., 2011; Lu & Rasco, 2012). Phenolic compounds were presented the distinctive peak at 1634 cm^{-1} is attributed to the ring C—C stretch of phenyl, which is high in polyphenolic components typical in samples. Peaks at 1404 cm^{-1} (CH_3 asymmetric deformation) and 1325 cm^{-1} (in-plane C—O stretching vibration combined with ring stretch of phenyl) were observed. Drying increased some compounds while decreasing phenolics, as indicated by higher peaks in dried shallots compared to fresh samples. Peaks at 813 cm^{-1} (ring CH deformation) reflect structural information about polyphenols. These spectra were observed in all samples studied. This result is present at the peak of fresh shallot and low-temperature dried shallots, which have shown a higher peak due to their higher moisture content than high-temperature dried shallots. The distinguished peak observed at 1634 cm^{-1} corresponds to the C—C ring stretch of phenyl (Lu et al., 2011; Schulz & Baranska, 2007), which is found in high concentrations within the polyphenolic compounds of Allium plants. The band at 1404 cm^{-1} is attributed to the asymmetric deformation of CH_3 (Agarwal et al., 2006). Additionally, the peak at 1325 cm^{-1} arises from the in-plane C—O stretching vibration combined with the phenyl ring stretch (Lu et al., 2011; Schulz & Baranska, 2007). The bands observed at wavenumbers 1103 and 1018 cm^{-1} are associated with the stretching vibrations of hydrogen-bonding C-OH groups (Wang et al., 1997), as well as the C-OH stretching band of oligosaccharides (Yoshida et al., 1997). These bands also represent C—O stretching coupled with C—O bending of carbohydrate C-OH groups (Wang et al., 1997). This finding reinforces earlier research linking the presence of phenolic compounds and hydroxyl functional groups to antioxidant activity. Overall, FT-IR can effectively estimate the antioxidant capacity of shallots with accuracy comparable to traditional chemical assays. This approach is straightforward and quick once the tissue extracts are ready. The band at 813 cm^{-1} is attributed to ring CH deformation (Schulz & Baranska, 2007), which may also provide structural insights into polyphenols. The result was observed that peak of dried shallot has higher than fresh sample. It may be caused effect of drying on shallot decreased some high-molecule of phenolics and then increases some compounds after drying methods. This is supported by the association of phenolic compounds and hydroxyl functional groups with antioxidant capacity.

Polysaccharides are considered to be important components of plants as they are components of cell walls and tissues of plant cells. Carbohydrates and polysaccharides were presented the peaks at 1103 cm^{-1} are related to hydrogen-bonding C-OH groups, oligosaccharide C-OH stretching, and C—O stretching coupled with C—O bending of carbohydrates (Andrus, 2006; Lu et al., 2011), while the bands at 1018 and 985 cm^{-1} (Mordechai et al., 2001) are due to vibrational frequency of -CH_2OH groups of carbohydrates. Functional groups of polysaccharides appeared in the spectral range of 900–1200 cm^{-1}in both fresh and dried samples. Amino Acids and Proteins were explored the spectral range of 1300–1700 cm^{-1} is related to lipids and proteins. Both fresh and dried shallots showed peaks corresponding to amino acids. A minor band at 1242–1265 cm^{-1} is associated with amide III (random coil) for protein, suggesting a reduction of amino acids due to the Maillard reaction during heat drying. Peaks at 1243–1265 cm^{-1} (alleles of ethers) were more prominent in fresh samples. The result was observed that the fresh samples had larger and smaller peaks during drying. This suggests the

Table 2
Effect of different drying methods on volatile compounds of shallots.

No	Volatile compounds	RI	*Area (%)*					
			Fresh	FD	SD	HD40	HD60	HD80
1	Acetic acid	679	ND	ND	1.11	ND	ND	1.5
2	Allyl ethyl ether	681	ND	ND	ND	3.32	ND	ND
3	Butanal, 2-methyl-	682	ND	ND	3.82	ND	5.19	4.83
4	2-Propanone, 1-hydroxy	689	ND	ND	5.27	7.95	14.31	7.85
5	2,3-Pentanedione	704	ND	ND	2.45	2.50	4.67	2.41
6	Tricyclo[2.2.1.0(2,6)]heptane, 1,7,7-trimethyl	921	0.06	0.09	0.14	ND	ND	ND
7	Hexanal	805	ND	ND	1.81	ND	ND	ND
8	Bicyclo[3.1.0]hex-2-ene, 2-methyl-5-(1-methylethyl)-	927	0.23	0.21	0.52	0.41	ND	0.42
9	α-Pinene	933	8.60	7.65	9.30	13.50	13.4	8.39
10	Camphene	948	2.24	2.37	3.54	3.46	2.16	2.76
11	Bicyclo[3.1.0]hex-2-ene, 4-methylene-1-(1-methylethyl)-	954	ND	0.24	0.33	0.20	ND	0.27
12	Bicyclo[3.1.1]heptane, 6,6-dimethyl-2-methylene-, (1*S*)-	976	19.96	17.91	17.95	26.91	25.02	17.31
13	α-Phellandrene	1005	ND	ND	0.41	ND	ND	ND
14	Cyclopentene, 3-isopropenyl-5,5-dimethyl-	1010	ND	ND	ND	0.21	ND	ND
15	3-Carene	1010	ND	ND	0.19	ND	0.32	ND
16	Cyclohexene, 1-methyl-4-(1-methylethylidene)-	1017	ND	ND	ND	ND	0.17	ND
17	(+)-4-Carene	1017	ND	0.30	1.68	ND	ND	ND
18	2-Carene	1018	ND	ND	ND	0.67	ND	ND
19	(+)-4-Carene	1018	ND	ND	ND	ND	ND	0.48
20	o-Cymene	1026	ND	0.49	2.08	ND	1.15	0.86
21	Eucalyptol	1032	61.53	55.07	21.87	16.82	17.26	33.18
22	γ-Terpinene	1061	0.11	0.31	1.37	0.90	ND	ND
23	6-Nonynoic acid, methyl ester	1074	ND	0.13	ND	ND	ND	ND
24	Ethyl 2-(5-methyl-5-vinyltetrahydrofuran-2-yl)propan-2-yl carbonate	1075	ND	ND	ND	ND	0.51	ND
25	3-Cyclohexene-1-methanol, 2-hydroxy	1075	ND	ND	0.45	ND	ND	ND
26	L-Fenchone	1090	1.14	0.99	2.41	2.22	1.95	1.78
27	Benzene, 1-methyl-3- (1-methylethenyl)-	1096	ND	ND	1.35	ND	ND	ND
28	Fenchol	1115	0.56	0.41	1.07	0.62	0.34	0.45
29	Bicyclo[3.1.1]heptan-3-ol, 6,6-dimethyl-2-methylene-, [1S-(1α,3α,5α)]-	1140	0.17	1.03	1.41	1.12	0.76	ND
30	Isopinocarveol	1140	ND	ND	ND	ND	ND	0.89
31	(+)-2-Bornanone	1146	0.37	0.56	0.82	0.83	0.47	0.66
32	Bicyclo[2.2.1]heptan-2-ol, 2,3,3-trimethyl-	1150	ND	0.11	ND	ND	ND	ND
33	Pinocarvone	1165	ND	0.52	0.59	0.48	0.35	ND
34	*endo*-Borneol	1169	0.47	0.94	0.99	0.73	0.51	0.57
35	Myrtenal	1201	ND	1.61	0.81	ND	0.73	ND
36	Bicyclo[3.1.1]hept-3-en-2-one, 4,6,6-trimethyl-	1214	ND	0.14	ND	ND	ND	ND
37	β-pinene	1222	4.16	7.33	12.52	10.50	4.48	ND
39	2-Oxabicyclo[2.2.2]octan-6-ol, 1,3,3-trimethyl-	1230	0.33	ND	0.09	ND	ND	ND
40	Fenchyl acetate	1237	ND	ND	0.32	0.60	0.31	ND
41	Bicyclo[2.2.1]heptan-2-ol, 1,7,7-trimethyl-, acetate, (1S-endo)-	1289	ND	ND	ND	0.26	ND	ND
42	Bornyl acetate	1289	0.07	0.14	ND	ND	ND	0.16
43	Isobornyl acetate	1289	ND	ND	0.25	ND	ND	ND
44	(2,4,6-Trimethylcyclohexyl) methanol	1318	ND	ND	0.18	ND	ND	ND
45	Copaene	1380	ND	ND	ND	0.27	0.36	ND
46	Caryophyllene	1425	ND	0.37	0.64	1.51	1.08	0.67
47	γ-Muurolene	1482	ND	ND	ND	0.00	0.16	ND
48	2-Isopropenyl-4a,8-dimethyl-1,2,3,4,4a,5,6,7-octahydronaphthalene	1489	ND	ND	ND	ND	ND	0.21
49	4a,8-Dimethyl-2-(prop-1-en-2-yl)-1,2,3,4,4a,5,6,7-octahydronaphthalene	1490	ND	ND	ND	0.23	ND	ND
50	(4aS,5S,8aS)-5-Isopentyl-1,1,4a-trimethyl-6-methylenedec	1497	ND	0.21	ND	0.32	ND	ND
51	4-Hexen-1-ol, 6-(2,6,6-trimethyl-1-cyclohexenyl)-4-methy-, (*E*)-	1497	ND	ND	ND	ND	0.26	ND
52	2,4a,8,8-Tetramethyldecahydrocyclopropa[*d*]naphthalene	1497	ND	ND	ND	ND	ND	0.37
53	Caparratriene	1497	ND	ND	0.44	ND	ND	ND
54	β-copaene	1520	ND	0.16	ND	ND	ND	ND
55	Germacrene D	1521	ND	ND	ND	0.50	ND	ND
56	γ-Muurolene	1521	ND	ND	ND	ND	0.26	ND
57	Naphthalene, 1,2,3,5,6,8a-hexahydro-4,7-dimethyl-1-(1-methylethyl)-, (1S-cis)-	1529	ND	0.11	ND	ND	ND	ND
58	Tau-Cadinol acetate	1530	ND	ND	ND	0.32	ND	ND
59	Naphthalene, 1,2,3,5,6,8a-hexahydro-4,7-dimethyl-1-(1-methylethyl)-, (1S-cis)-	1530	ND	ND	ND	ND	0.09	ND
60	2(1H)-Naphthalenone, octahydro-4a,7,7-trimethyl-, cis-	1536	ND	ND	0.55	0.35	ND	ND
61	3,7,11,15-Tetramethyl-2-hexadecen-1-ol	1536	ND	ND	ND	ND	0.29	ND
62	Carotol	1536	ND	0.32	ND	ND	ND	ND
63	6-epi-shyobunol	1537	ND	ND	ND	ND	ND	0.41
64	Neoisolongifolene, 8-bromo-	1609	ND	0.28	ND	ND	ND	ND
65	1,3-Dimethyl-5-propyl-7-(propene-1-yl) adamantane	1610	ND	ND	ND	0.20	ND	ND
66	But-3-enal, 2-methyl-4-(2,6,6-trimethyl-1-cyclohexenyl)-	1642	ND	ND	ND	0.63	0.42	0.53
67	1,4-Methanoazulene, 7-bromodecahydro-4,8,8-trimethyl	1649	ND	ND	ND	ND	0.61	ND
68	9,11-Dimethyltetracyclo [7.3.1.0(2.7)0.1(7.11)] tetradecan	1649	ND	ND	ND	ND	ND	0.61
69	Longifolenaldehyde	1685	ND	ND	ND	ND	0.44	ND
70	Ambrial	1815	ND	ND	ND	ND	1.97	2.2
	Total		100	100	100	100	100	100

RI = Retention index.
ND=Not detected.

precursor reduction of amino acids in the Maillard reaction induced by heat drying. Especially in the smallest samples dried at 80 °C and SD as may be evident the results of the amino acid content analysis. The peaks found at 1243–1265 cm^{-1} were alleles of ethers, which were partly found in the structural formula of Eucalyptol, which were more characteristic of the clear peaks in fresh and dried samples. Lu et al. (2011) reported that FTIR can predict the antioxidant capacity of shallots with precision similar to conventional chemical assays. Overall, the FTIR analysis provided detailed insights into the changes in functional groups and chemical compositions of shallots due to different drying methods. The method proved to be simple and rapid for predicting antioxidant capacity and other biochemical properties of shallots.

3.10. Microstructural analysis by scanning electron microscope (SEM)

The five dried samples by three drying methods produced distinct structural changes in shallots, as revealed by scanning electron microscopy (SEM) (Fig. 6). Freeze-dried tissues closely resembled fresh samples, with the cell walls of fresh shallots being better maintained in comparison to all the dried samples. Hot-air drying (HD) resulted in a denser structure and notable cell wall damage. Different hot-air drying temperatures caused varying degrees of disruption, with the most significant structural alterations occurring at 80 °C. The sun-dried (SD) samples displayed similar structural traits to HD40, likely due to the comparable temperatures used, leading to similar post-drying structures. Fig. 6 also highlights how HD (HD60 and HD80) and SD samples developed distinct structural differences, driven by thermal and moisture gradients that led to cell wall deformation, folding, and damage during drying. The HD process caused more severe distortion than the SD method, which, despite being more gradual, showed inconsistency due to fluctuating sunlight temperatures. Freeze drying functions by transferring water from the frozen material to the surface via sublimation (An et al., 2016). Recent studies reinforce these findings. For example, Yang et al. (2024) study on apricot slices revealed that vacuum freeze-drying (VFD) resulted in better microstructural integrity and higher nutrient retention compared to hot air drying (HAD). Another study on apricot highlighted that freeze drying preserved the microstructure of sample (Li et al., 2024). These studies emphasize the advantages of freeze drying in maintaining the structural and nutritional quality of dried plant tissues. Drying methods significantly impact the surface morphology of shallots. Researchers observed that freeze-drying caused the almost complete disappearance of the extracellular polymeric substances (EPS) matrix. In a study by Lee and Chow (2012), cells on porous hydroxyapatite (HA) were prepared using freeze-drying for SEM examination. The results suggested that freeze-drying could effectively dehydrate cells on 3D scaffolds for SEM analysis. Furthermore, Chumroenphat et al. (2021) noted that the microstructure of freeze-dried tissue resembled that of fresh samples. In contrast, the hot air-dried (HD) samples exhibited a denser structure, with significant damage to the cell parenchyma and dispersed starch grains throughout the tissue, along with the absence of oil cells. In this present study, the reason to maintain the moisture content of final product at approximately 3 % is based on the local practice as the utilization of dried shallot product is normally for frying, rehydrating or even making the powder before further cooking or consuming. Although using hot air drying to research 3 % MC effectively enhances shelf life and antioxidant activity, it also negatively impacts microstructure, nutrient retention, and flavor quality. The authors suggest that future studies should optimize drying conditions to minimize these drawbacks while maintaining product quality. This study used freezing at −50 °C for 12 h to preserve the quality of shallots, including bioactive and aromatic compounds as well as structural integrity. As shown in Fig. 6, freeze-dried (FD) shallots exhibited minimal structural damage. Slight discoloration was observed, but the shallots retained their purple-red hue, similar to fresh samples without browning. Thus, any chilling injury appears negligible, with little to no impact on the final product. Sun-dried turmeric displayed a similar microstructure, although it was less damaged compared to the HD samples (Chumroenphat et al., 2021). Additionally, the use of a food dehydrator for drying sliced shallots has been shown to cause changes in color and bioactive compounds due to heat-induced damage, influencing the surface characteristics of the dried shallots. Therefore, the choice of drying method plays a crucial role in determining the surface morphology, overall quality and bioactive compounds of shallots. Overall, the literature indicates that the drying method used in SEM sample preparation can significantly impact the morphology and structure of biological samples.

4. Conclusions

This study highlights the significant influence of drying methods on the chemical composition, bioactive compounds, and structural integrity of shallots. Among the techniques evaluated, FD was most effective in preserving the microstructure and key volatile compounds, particularly eucalyptol, while enhancing the retention of phenolic acids such as syringic, *p*-coumaric, protocatechuic, vanillic, and ferulic acids. Conversely, HA80 resulted in the highest total phenolic and flavonoid contents, along with the greatest antioxidant activity as measured by the FRAP assay, and an increased concentration of apigenin. Despite these benefits, all drying methods caused reductions in fructooligosaccharides, sucrose, and stachyose, though proline remained the predominant amino acid, with arginine levels increasing in SD.

Based on these findings, HA80 appears to be the most suitable drying method for shallots, as it significantly enhances bioactive compounds while maintaining antioxidant properties. This method offers a balance between bioactive compound retention and drying efficiency, making it a practical and cost-effective approach for industrial applications, particularly in the functional food and nutraceutical sectors. Future studies should explore the sensory attributes, storage stability, and potential interactions of bioactive compounds in dried shallots over time. Additionally, investigations into optimizing drying conditions for preserving specific functional components, as well as scaling up production for commercial exploitation, would be beneficial. The incorporation of dried shallots into functional food formulations, dietary supplements, and natural flavoring agents also warrants further research to maximize their health benefits and market potential.

CRediT authorship contribution statement

Jiranan Ratseewo: Writing – review & editing, Writing – original draft, Investigation, Formal analysis, Data curation, Conceptualization. **Theeraphan Chumroenphat:** Writing – review & editing, Investigation, Formal analysis, Data curation. **Hua Li:** Writing – review & editing, Investigation, Data curation, Conceptualization. **Sirithon Siriamornpun:** Writing – review & editing, Writing – original draft, Investigation, Funding acquisition, Data curation, Conceptualization.

Declaration of generative AI and AI-assisted technologies in the writing process

During the preparation of this work, the author(s) used QuillBot in order to grammar-check and paraphrase. After using this tool/service, the author(s) reviewed and edited the content as needed and take(s) full responsibility for the content of the publication.

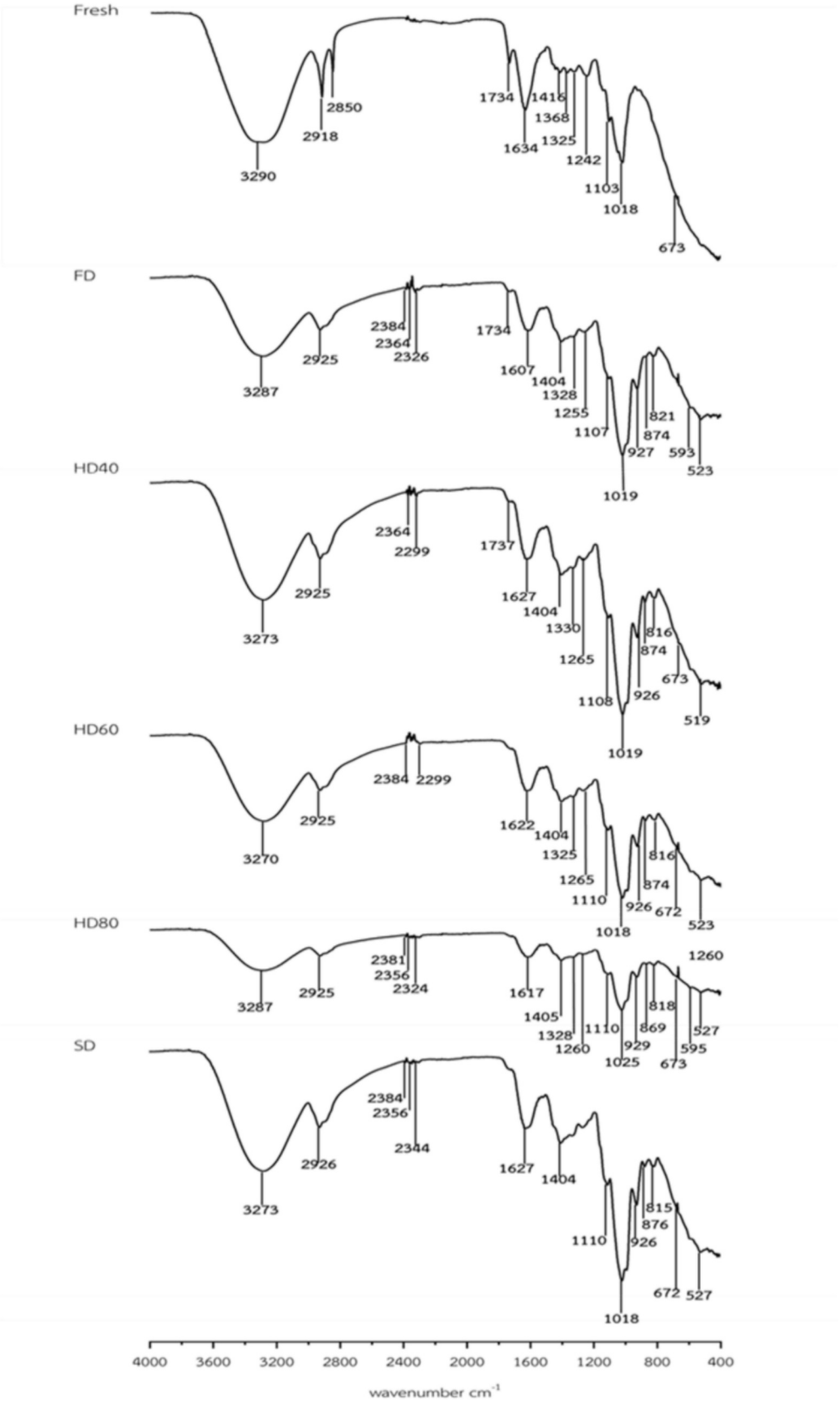

Fig. 5. Effect of different drying methods on FTIR of shallots.

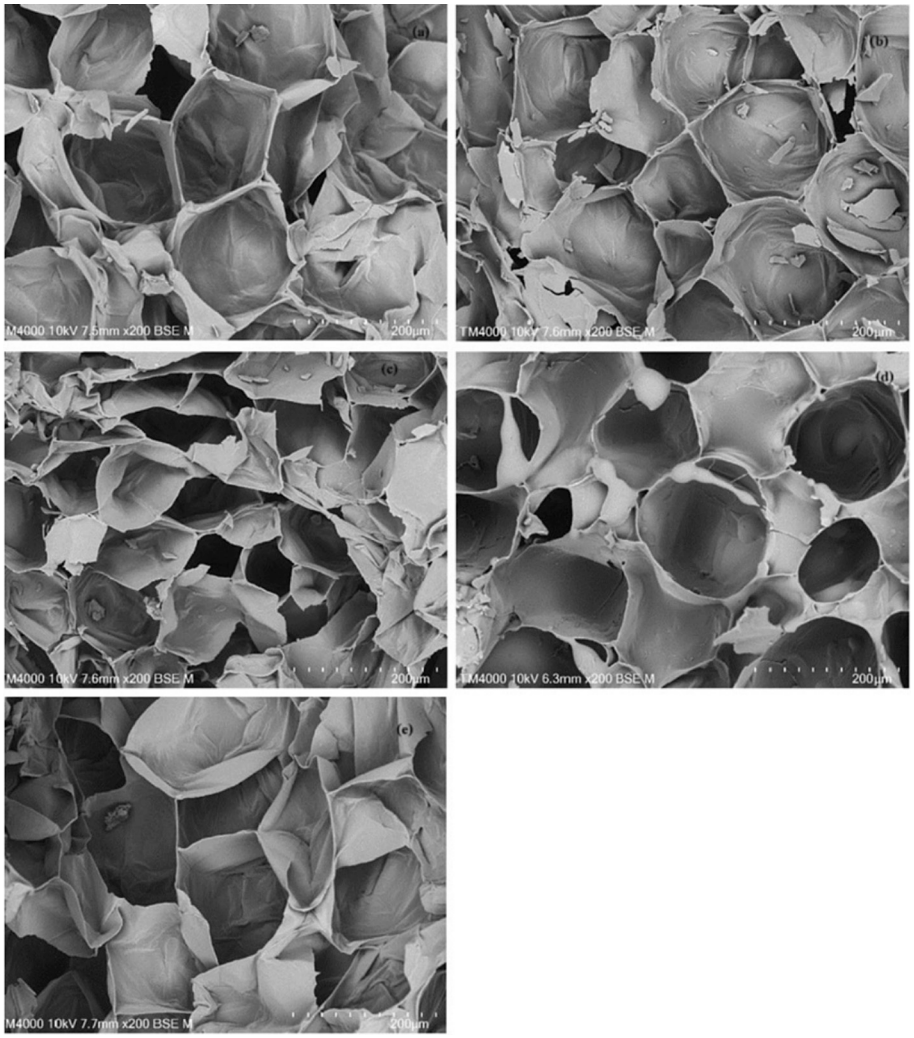

Fig. 6. Scanning electron micrographs of different drying methods on shallots. a: HD40 (×200); b: HD60 (×200); c: HD80 (×200); d: FD (×200); e: SD (×200).

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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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.fochx.2025.102419.

Data availability

Data will be made available on request.

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