

EXTRACTION EFFICIENCY OF SOLVENTS AND POSTHARVEST EFFECTS ON
MELON (*Cucumis melo* L.) HEALTH-PROMOTING COMPOUNDS MEASURED
USING HPLC AND HS-SPME-GC-MS

A Thesis

by

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ABSTRACT

Melons (*Cucumis melo*) have abundant health-promoting phytochemicals; understanding how these change during postharvest storage provides important information for preserving these phytochemicals. Here, we tested three cantaloupe cultivars (Western Shipper, Infinite Gold, and Da Vinci) and three honeydew varieties (Orange Casaba, HD150, and HD252) grown in Uvalde, Texas. Fruits were stored at 10 °C for 20 days, processed and analyzed at 5-day intervals for carotenoids, amino acids and ascorbic acid using HPLC. Da Vinci showed high levels of β -carotene ($24.27 \pm 0.88 \text{ mg kg}^{-1}$) on day 0 of storage. Total ascorbic acid was highest in Western Shipper and Da Vinci melons on day 0 ($112.82 \pm 13.96 \text{ mg kg}^{-1}$, $90.39 \pm 14.43 \text{ mg kg}^{-1}$) and decreased at day 20 ($17.67 \pm 1.88 \text{ mg kg}^{-1}$, $31.33 \pm 3.88 \text{ mg kg}^{-1}$). Honeydew variety HD252 showed the highest total ascorbic acid at day 5 ($70.94 \pm 3.50 \text{ mg kg}^{-1}$) and this decreased ($31.53 \pm 3.54 \text{ mg kg}^{-1}$) at the end of storage (day 20). The important neurotransmitter GABA was highest in Da Vinci cantaloupes at day 0 ($2985.04 \pm 79.17 \text{ } \mu\text{g g}^{-1}$) and decreased ($2426.89 \pm 102.57 \text{ } \mu\text{g g}^{-1}$) at day 20. The total amino acid levels changed during storage, showing an overall increase at day 20. Da Vinci and Infinite Gold varieties showed high total amino acid contents on day 0 ($8050.27 \pm 390.64 \text{ mg kg}^{-1}$, $7809.86 \pm 399.82 \text{ mg kg}^{-1}$), which increased on day 20 ($9495.72 \pm 640.85 \text{ mg kg}^{-1}$, $8473.21 \pm 358.24 \text{ mg kg}^{-1}$). Interestingly, the biogenic amines putrescine and spermidine were observed during storage. Total phenolics content (TPC) showed different trends in each variety; for example, on day 0 da Vinci showed high TPC ($21.81 \pm 0.70 \text{ g L}^{-1}$), Western Shipper showed high TPC on day 5 ($19.32 \pm 1.21 \text{ g L}^{-1}$), HD150 showed high TPC on day 15 ($19.53 \pm 0.85 \text{ g L}^{-1}$) and HD252 showed high

TPC ($18.87 \pm 1.20 \text{ g L}^{-1}$) on day 20. The aroma profile of each variety was analyzed using HS-SPME-GC-MS and identified compounds were classified as alcohols, aldehydes, esters, monoterpenoids (limonene, α -terpineol, 1,8 cineole, citronellal), and norisoprenoid (β -ionone). During postharvest storage, melon fruits showed significant variation in phytonutrients, free radical scavenging activity, and aroma profiles. These results indicate that the postharvest changes in bioactive compounds are influenced by melon variety and storage duration. This study compared the effect of 22 solvent combinations using different ratios of methanol, ethanol, acetone, water, and formic acid on TPC and free radical scavenging activity in melons obtained from College Station, Texas. TPC was determined using the Folin-Ciocalteu (F-C) assay and an optimized Fast Blue (FB) assay. The FB assay showed that water extracted the highest TPC ($94.82 \pm 8.02 \text{ mg kg}^{-1}$ gallic acid equivalents, GAE). The F-C assay showed that methanol extracted the highest TPC ($137.99 \pm 18.29 \text{ mg kg}^{-1}$ GAE), indicating that the FB assay was more sensitive for water extracts. Free radical scavenging activity was determined by 2,2-diphenyl-1-picrylhydrazyl (DPPH) and 2,2-azino-bis (3-ethylbenzothiazoline-6-sulfonic acid) (ABTS) assays. The DPPH scavenging activities were high in methanol (100%) ($38.49 \pm 0.36 \text{ mg kg}^{-1}$ ascorbic acid equivalents, AAE) and 80% methanol extracts ($38.99 \pm 0.44 \text{ mg kg}^{-1}$ AAE). Similarly, the ABTS scavenging activities were high in methanol (100%) ($315.11 \pm 10.38 \text{ mg kg}^{-1}$ AAE) and 80% methanol extracts ($297.39 \pm 14.98 \text{ mg kg}^{-1}$ AAE). Generally, the results indicated that the solvent used affected TPC and free radical scavenging activities. Moreover, we successfully detected six phenolic compounds in melon extracts by liquid chromatography coupled with high resolution quadrupole time-

of-flight mass spectrometry. Our results suggest that the polarity of the solvent used to extract the melon samples influenced the recovery of phenolic compounds and free radical scavenging activity.

DEDICATION

I would like to dedicate this thesis to my dear mother Amirtha and my loving father Ravindranath who taught me never to give up and keep going no matter what, for believing in me, supporting me, always encouraging and motivating me to do my best in all walks of life.

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CONTRIBUTORS AND FINDING SOURCES

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This work was supervised by a thesis committee consisting of Professor Bhimanagouda S. Patil, the committee chair of the Department of Nutrition and Food Science, Professor G.K. Jayaprakasha and Dr. Kevin M Crosby of the Department of Horticultural Sciences.

All the work conducted for the thesis was completed by the student independently.

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CHAPTER I

INTRODUCTION

Cucumis melo L. (Reticulatus group), commonly called as cantaloupe or muskmelon, is a member of the Cucurbitaceae family [1]. Consumer preference for melons is primarily determined by sweetness, flavor or aroma, texture and phytonutrients [2, 3]. Another subgroup under melons is the non-netted inodorous variety commonly known as honeydew (*Cucumis melo* var. *inodorous*) with smooth peel and larger size. Melons, as good sources of polyphenols and antioxidants, provide significant health benefits. Furthermore, melons, being rich in water, are categorized as diuretics. Studies also demonstrated that cantaloupe pulp extract possesses high antioxidant and anti-inflammatory properties due to the higher levels of vitamin A, vitamin C, potassium, and magnesium [4].

In the first study, the extraction efficiency of solvents was evaluated. Secondary metabolites, such as polyphenols or phenolics, include simple flavonoids, phenolic acids, complex flavonoids and colored anthocyanins. The various factors, such as physical and chemical properties of samples, type of solvent, extraction time, temperature and sample-to-solvent ratio influence the extraction yield of the compounds. Therefore, solvents and their role in extracting phenolic compounds determine the levels of total phenolics and antioxidant activity in samples. Previous studies have shown that the most suitable solvent for the recovery of polyphenolic compounds is an aqueous mixture containing acetone,

ethanol, methanol, and ethyl acetate [5]. The extraction of such phenolic compounds is influenced by the nature of the chemical structure and the polarity of solvents used for extraction. This study aimed to determine the phenolic content and antioxidant activities of melon extracts using different solvent combinations.

In the second study, postharvest storage of melon fruits was evaluated for changes in bioactive compounds. Melon consumption has remained high for a variety of reasons, as consumer preference is primarily determined by melons' sweetness, flavor, aroma, texture, and more recently for melons as a rich source of phytonutrients [6, 7]. Therefore it is important to preserve these health-promoting compounds in fruits and vegetables and consider the effects of pre-harvest and post-harvest factors [8]. Postharvest storage can influence fruit quality and shelf life, and contribute to nutrient composition changes. Studies of chemical treatments, a variation of storage temperatures, application of chitosan coating, the ethylene inhibitor 1-methylcyclopropene (1-MCP), and aroma profiling after treatment and analysis of fruit storage have helped improve fruit consumption and storage. However, there are few studies on the effects of postharvest storage on the bioactive compound profile of melons without chemical treatment. Therefore, this study aimed to evaluate the changes in metabolites, antioxidant properties, and aroma profiles of six melon varieties during postharvest storage at a uniform temperature.

Objectives

- 1) To study the effect of postharvest storage on the health-promoting compounds and antioxidant activities in six melon varieties
- 2) To evaluate the extraction efficiency of solvent combinations on antioxidant activity and optimize fast blue assay to measure the total phenolic content in melon and juice samples

CHAPTER II

LITERATURE REVIEW

Melon (*Cucumis melo* L.) is a commercially important horticultural crop worldwide that exhibits extensive phenotypic and genetic variation [9]. Postharvest procedures adopted to maintain Cucurbitaceae crops such as melons mainly involve refrigeration, hydro-cooling, edible coating and use of the ethylene inhibitor 1-Methylcyclopropene(1-MCP) [10-12]. Indeed, 1-MCP application, along with refrigeration, successfully reduced softening, maintained firmness, delayed decay, and ameliorated chilling injuries in summer squash (*Cucurbita pepo*) [13]. Another study evaluated Jiashi melons (*Cucumis melo*) treated with 3, 4, and 5% 1-MCP prior to postharvest storage; after 60 days of storage, this treatment delayed decay and maintained firmness of fruits [14]. The role of ethylene in ripening and postharvest characteristics was demonstrated in inodorous melon varieties stored at chilling and non-chilling temperatures [15]. Enzyme activity and gene expression changed during the storage of Gold Queen Hami melons at different temperatures and humidity for 36 days [16]. These studies have shown that postharvest treatments can effectively enhance storage and reduce the loss of fruit quality. The changes in the composition of bioactive compounds that occur in fruits during postharvest storage without any chemical treatment may require quantifying the levels of phytonutrients in fruits. Volatile compounds of cantaloupe and honeydew are influenced by factors such as harvesting time, developmental stages, shelf life, and varietal differences [17-23]. These compounds responsible for the characteristic flavor and aroma

of fruits are produced via metabolic pathways and depend on various factors, including species, variety, and treatment [24].

Among the phytochemical substances, phenolic compounds, including phenolic acids and flavonoids, are the major groups of natural components in plants that have received increasing interest due to their free radical scavenging properties. Consumption of fruits rich in phenolic compounds can limit oxidative stress due to antioxidant properties and reduce the risk of degenerative diseases such as cancer, cardiovascular diseases and diabetes [25]. Similarly, consumption of polyphenol-rich beverages resulted in a pronounced reduction in oxidized DNA bases in blood leukocytes [26]. These beneficial potentials are attributed to the presence of vitamins, minerals, phenolic metabolites, flavonoids, and alkaloids in plants; these act as free radical scavengers within human bodies [27]. Hence, selecting the best solvent is a key factor that impacts the quality and quantity of extracted phenolic compounds [28-30]. So far, the recovery of melon phenolic compounds by solvent combinations has not been explored. This study aims to examine the various solvents used for the extraction of melon samples to determine the phenolic content and antioxidant activity of the extracts.

CHAPTER III

POSTHARVEST STORAGE EFFECTS ON THE PHYTOCHEMICAL LEVELS, FREE RADICAL SCAVENGING ACTIVITY, AMINO ACIDS, AND VOLATILE COMPOSITION OF MELON VARIETIES

Introduction

Fruits and vegetables remain physiologically active after harvest and their metabolic processes continue during postharvest handling and storage. Abiotic stress can accelerate metabolic response during these processes, degradation of substrates and changes in bioactive compounds [31]. Postharvest storage of produce involves continuous changes in living tissues and causes a loss of nutrients that provide energy, which the plant tissues use to maintain their metabolism; this can lead to degradation of phytonutrients. Therefore, studies on postharvest treatments on fruits and vegetables is a crucial step to extend the shelf life of harvested produce [32].

Melon consumption has remained high for various reasons as consumer preference is determined largely by sweetness, flavor, aroma, texture, and more recently for melons as a rich source of phytonutrients [2, 3]. Therefore, it is important to preserve these health-promoting compounds in fruits and vegetables and consider the effects of preharvest and postharvest factors [33]. Postharvest storage can influence fruit quality, shelf life and the nutrient composition changes. Studies of chemical treatments, variation of storage temperatures, application of chitosan coating, 1-MCP [10-12], and aroma profiling after treatment and analysis of fruit storage have helped to improve fruit consumption and

storage. However, there are few studies on the effects of postharvest storage on the bioactive compound profile of melons without chemical treatment. Therefore, this study aimed to evaluate the changes in metabolites, antioxidant properties, and aroma profiles of six melon varieties during postharvest storage at uniform temperature.

Another important aspect of melon fruit after harvest is development of aromatic compounds. A previous study examined aroma volatiles showed that shelf life determines the aroma of medium and long shelf-life Charentais melons harvested at different maturity stages [23]. A similar study on Oriental sweet melons suggested that volatile compounds in melons are derived from conversion of amino acids and phytonutrients, thus influencing the melon's flavor profile [34]. Another study on Makino melon (*Cucumis melo* var. *makuwa*) stored at room temperature showed the effects of postharvest treatments on the quality of fruit found that 3-lipoxygenase genes are involved in fruit ripening and production of volatile compounds (aldehydes and alcohols) [35]. External factors such as storage and temperature along with biochemical factors such as variety of fruit, bioactive compounds and ripening behaviors affect the postharvest changes in the aromatic profile of melons.

However, there are few studies on the effects of postharvest storage on the bioactive compound profile of melons without chemical treatment. Therefore, this study aimed to evaluate the changes in metabolites during storage, measure levels of bioactive compounds (carotenoids, ascorbic acid, amino acids, bioamines and volatile aromatic compounds), evaluate the antioxidant properties from day 0 to day 20 of selected melon varieties during postharvest storage.

Materials And Methods

Chemicals

ACS grade solvents were used for extraction and high-performance liquid chromatography (HPLC) grade solvents acetonitrile and methanol were used for quantitative analysis. Meta phosphoric acid, TCEP (tris(2-carboxyethyl) phosphine), dansyl chloride, phosphoric acid, formic acid, triethylamine, sodium carbonate, 2,2-diphenyl-1-(2,4,6-trinitrophenyl) hydrazyl (DPPH), Folin Ciocalteu (FC) reagent, 2, 2'-azinobis (3-ethylbenzothiazoline-6-sulphonic acid) diammonium salt (ABTS), analytical grade R(+)-limonene, 1,8-cineole, 2-pentylfuran, ethyl hexanoate, (E)-2-heptenal, 6-methylhept-5-en-2-one, ethyl heptanoate, 1-hexanol, dimethyl trisulfide, (Z)-3-hexan-1-ol, ethyl caprylate, 1-octen-3-ol, decanal, benzaldehyde, (E)-2-nonenal, (E,Z)-2,6-nonadienal, β -cyclocitral, ethyl decanoate, ethyl benzoate, 1-nonanol, 1-decanol, (E)-carveol, Geranylacetone, β -ionone, benzothiazole, β -ionol, thymol, farnesyl acetone, nootkatone, sodium chloride, n-alkane (C6-C24) were used as standard and purchased from Sigma Aldrich (St. Louis, MO). SPME fibers and amino acid standards were also purchased from Sigma Aldrich (St. Louis, MO).

Melons And Storage Study

Three cantaloupe melon varieties, Western Shipper, Infinite Gold (Harper type), Da Vinci (Sakata), and three honeydew varieties, Orange Casaba, HD150 and HD252 were selected for this study. The fruits were cultivated and harvested from Uvalde AgriLife Center, TX. Fruits were harvested during July 2019 and transported immediately

to the Vegetable and Fruit Improvement center (Texas A&M University, College Station, TX). The fruits were segregated according to variety and stored at 10 °C throughout the study. Fruits were collected at 0, 5, 10, 15, and 20 days of storage for processing (Fig. 1). For each storage period, three fruits were collected from each variety. The collected melons were evaluated for shelf quality and bioactive compounds were quantified at different storage intervals.

Physicochemical assessment

The melon fruits were assessed for total soluble solids contents using a refractometer (Reichert, NY, U.S.A.). Total soluble solids were taken from the blended melon juice, calculated as the average of three measurements, and expressed as °Brix. Fruit firmness was assessed using Penetrometer (Agtec fruit firmness tester, FHP 803) with a 5-mm probe and expressed in Newtons (N). Color of melon flesh of cantaloupe and honeydew varieties was assessed using a colorimeter, Lightness (L*) was used to evaluate the brightness of orange (cantaloupe) and green (honeydew) melons; a* (hue of colors green to red), b* (hue of colors blue to yellow), and C (Chroma) were measured using a Minolta CR-200 Chroma Meter (Minolta, Osaka, Japan). The instrument was calibrated with a white tile standard and triplicate readings were recorded for each parameter.

Sample preparation

Melon fruits were washed under running hot water for 30 sec and then cleaned with paper towels. Melons were cut into two halves, deseeded and peeled. The flesh was cut into small cubes and blended using an Oster blender (Model 6647,US). Blended samples were transferred into storage containers and stored at -20 °C for further analysis.

Total phenolics and radical scavenging activity

Analysis of phenolics

Extraction was carried out as per previously described protocol [36] with slight modification. Briefly, melon sample 10 g of each were extracted twice with 10 mL of methanol. The samples were vortexed, homogenized for 1 min and sonicated (Cole-Parmer Ultrasonic cleaner 8893) in ice-cold water for 30 min. Extracted samples were centrifuged at 1814 x g for 1 min to obtain a clear supernatant and residue was re-extracted using 5 mL of methanol. The extracts were pooled and stored in clean tubes at -20 °C. The total phenolics content in the different extracts was determined by Folin-Ciocalteu assay [37]. The absorbance was monitored at 760 nm with a microplate reader (Bio Tek Instruments, Winooski, VT). Total phenolics were expressed as mg kg⁻¹ of standard (gallic acid equivalents, GAE).

DPPH radical scavenging activity

Free radical scavenging activity of extracts were measured as per our previously published protocol [38]. Briefly, samples were pipetted into different wells in triplicate of a 96-well microplate and the absorbance was recorded at 515 nm using a microplate reader. Results were expressed as mg kg⁻¹ of ascorbic acid equivalents.

ABTS radical scavenging activity

ABTS activity was measured by according to the previously published protocol [37]. Briefly, the 2,2'-azino-bis-(3-ethylbenzothiazoline-6-sulphonic acid) (ABTS) reagent was prepared by mixing 0.165 g of potassium persulfate in 200 mL NanoPure water and in a separate beaker, 0.193 g of ABTS was measured and dissolved in 200 mL

NanoPure water. The mixture was further diluted with 100 mL of NanoPure water to obtain 500 mL of solution. An aqua green color developed after overnight incubation in the dark, and the resulting ABTS stock solution was used for the assay. Fresh ascorbic acid standard (0.05 mg mL^{-1}) was prepared in 3% metaphosphoric acid (MPA). The samples were analyzed for their antioxidant capacity at different storage intervals.

Quantification Of Carotenoids

Melon samples (5 g) were extracted in the dark with 10 mL of chloroform: acetone (1:3), vortexed for 1 minute, homogenized (850 Homogenizer, Fisher Scientific, Waltham, Massachusetts, USA) for 1 min at $1814 \times g$ and sonicated (Cole-Parmer Ultrasonic cleaner 8893) in ice cold water for 30 min. Sample tubes were centrifuged using at $4480 \times g$ for 10 min (Beckman Model TJ-6, Ramsey, Minnesota, USA). The organic layers obtained were collected and used for HPLC analysis. A Waters 1525 HPLC system (Milford, MA, USA), equipped with a Waters 2996 PDA detector and Waters 717 Plus autosampler was used for quantification. Carotenoids were separated with a YMC carotenoid C30 (150 x 4.6 mm) column and a mobile phase of (A) methanol and (B) tert-butyl methyl ether with a flow rate of 0.60 mL min^{-1} . The gradient method with 25 min run time started with 25–75% B (14 min), 75–25% B (8 min) and 25–75% B (3 min). Samples of 50 μL of melon extracts were injected into the HPLC. The carotenoids' chromatogram was recorded at four different wavelengths such as phytoene at 286 nm, phytofluene at 300 nm, ζ -carotene isomers at 400 nm and α -carotene, β -cryptoxanthin, β -carotene and β -carotene isomers at 450 nm.

Quantification Of Ascorbic Acid (AA) And Dehydroascorbic Acid (DHA)

Melon samples (5 g) were mixed with 5 mL of 3% MPA solvent, vortexed for 1 min, homogenized (850 Homogenizer, Fisher Scientific) for 1 min at 1098 x g, sonicated (Cole-Parmer Ultrasonic cleaner 8893) for 30 min in ice cold water and then centrifuged at 16128 x g for 10 min (Beckman Model TJ-6, Ramsey, Minnesota , USA). The supernatant was filtered, collected, and stored at -80 °C for HPLC analysis. Dehydroascorbic acid (DHA) analysis was carried out by mixing 350 µL of extract with 350 µL of tris-(2-carboxyethyl) phosphine (TCEP). AA and DHA were quantified as per the previously published method [39] using an Agilent 1220 Infinity series HPLC system (Waldbronn Germany, Oxfordshire, U.K.) with a photodiode array detector, autosampler and a multistage pump. The solvent system consisted of (A) 0.03 M aqueous phosphoric acid and (B) methanol as a mobile phase with 16-min run time at flow rate of 400 µL min⁻¹. Separation of AA and DHA was achieved on an Eclipse Plus C18 column (250×4.6 mm, 5 µm). HPLC method included 100% A for 9 min, 100–70% B for 4 min and 100% A for 3 min. The AA and DHA peaks were recorded at 244 nm and levels were quantified by the regression equation and dilution factor using ascorbic acid standard.

Quantity of sample required for ascorbic acid analysis

Fresh melon fruits were purchased in H-E-B supermarket (College Station, TX). Melon fruit was cleaned, peeled, flesh was cut into cubes and blended to obtain juice. Sample was weighed 50, 100, 250, and 500 mg were extracted with 1.5 mL of 3% metaphosphoric acid. Samples were vortexed, homogenized for 1 min and sonicated for 30 min. Sample tubes were then then centrifuged at 16128 x g for 10 min (Beckman Model TJ-6, Ramsey,

Minnesota, USA). Supernatant was used for HPLC analysis of ascorbic acid (AA), TCEP was added for analysis of dehydroascorbic acid (DHA) and total ascorbic acid was calculated based on the values of AA and DHA.

Derivatization And Quantification Of Amino Acids And Bioamines

Derivatization of methanolic extracts with dansyl chloride was carried out to quantify amino acids [40]. Briefly, 350 μ L of methanol extract was mixed with 125 μ L dansyl chloride followed by 300 μ L sodium borate buffer (pH 9.4) and 50 μ L of diamino heptane standard in the dark. Later, the tubes were placed in water bath shaker maintained at 60 °C for 30 min. To stop further derivatization and stabilize the sample mixture, 60 μ L of 2 N acetic acid was added to sample and allowed cool at room temperature. The tubes were centrifuged, and the samples were transferred to vials for HPLC-FLD analysis. Amino acids were analyzed as per a previously reported method [41] with minor modifications. Briefly, our setup used a Perkin Elmer Series 200 HPLC system with binary pump and autosampler (Shelton, Connecticut, USA), a Gastorr TG-14 inline HPLC mobile phase degasser (FLOM USA, San Diego, CA, USA) and an Eppendorf TC-50 controller with CH-30 column heater (Eppendorf, Westbury, NY, USA). Detection of amino acids was achieved by using 1260 Infinity fluorescence detector controlled by Instant Pilot model G4208A (Agilent Technologies, Santa Clara, CA, USA). The system was supported by an interface (PE Nelson 900) and a Link box (PE Nelson 600). For the separation, Zorbax Eclipse XDB-C8 (4.6 x 150 mm, 5 μ m) was used with guard cartridge. The mobile phase consisted of 1% formic acid as solvent A and acetonitrile: formic acid: TEA (98:1:1,

v/v) as solvent B. The gradient programming included 15% B (4 min), 15% A and 20% B (12 min), a gradient increase to 45% B (2 min), remain isocratic for 2 min at 45% B, then 45% to 50% B (2 min), followed by an increase to 100% B (2 min), isocratic for 5 min, then a gradient reduction to 15% B (2 min) and isocratic for 2 min. Flow rate was set at 0.6 mL min^{-1} , injection volume was $5 \mu\text{L}$. The excitation and emission of the fluorescence detector were set at 293 nm and 492 nm, respectively for monitoring the derivatized amino acids. Perkin Elmer TotalChrom version 6.3.2. software was used to process the data.

HS-SPME-GC-MS Analysis Of Volatile Aroma Compounds

Volatile compounds were identified using a Thermo Finnigan GC-MS (Thermo Fisher Scientific, Inc., San Jose, CA, USA) equipped with an electron ionization source with a Dual-Stage Quadrupole (DSQ II) mass spectrometer (Thermo Scientific, Austin, TX, USA). For analysis, approximately 1 g of melon juice was weighed in a 20-mL GCMS vial containing 1 mL 30% NaCl and, $5 \mu\text{L}$ internal standard (nootkatone). The GC-MS sequence was set up and the method started with the vials being placed into a thermostatic stirrer for 30 min maintained at $80 \text{ }^\circ\text{C}$. A 2-cm SPME fiber coated with 50/30 μm divinylbenzene/carboxen/polydimethylsiloxane (DVB/CAR/PDMS) was used for extraction. Separation of compounds was achieved on a Restek Rtx-Wax column (30 m x 0.25 mm ID with $0.25 \mu\text{m}$ film thickness; Restek Corp., Bellefonte, PA, USA). Helium was used as the carrier gas at a constant flow rate of 1 mL min^{-1} in splitless mode and the inlet temperature was maintained at $225 \text{ }^\circ\text{C}$. The initial oven temperature was maintained at $40 \text{ }^\circ\text{C}$ for 2 min, and then increased to $210 \text{ }^\circ\text{C}$ at a rate of $5 \text{ }^\circ\text{C/min}$ with a holding time

of 1 min. The analysis time was 37 min. The ion source temperature and mass transfer line temperature were maintained at 285 and 280 °C. The ionization voltage was 70 eV, the mass range was 30–300 amu and the scan rate was 11.7 scans per second.

Calibration of standard, identification and quantification of volatile compounds

Six different concentrations (7.8 to 250 µg mL⁻¹) of standard nootkatone in 2 mL 30% NaCl were mixed, extracted, and injected into GC-MS by SPME in triplicate to obtain peak areas. Data were processed using Xcalibur software (v. 2.0.7., Thermo-Fisher Scientific, San Jose, CA, USA). Kovat's Indices (KI) values were calculated by the retention time of a mixture of n-alkane standards (C₆–C₂₄) analyzed under the same conditions as the sample. Identification of volatile compounds was achieved by comparing retention time, Kovat's Index (KI) and the mass spectrum from the National Institute of Standards and Technology library (NIST MS Search 2.0). The regression equation of nootkatone was used to calculate the concentration of volatile compounds and results were expressed as µg/kg of sample equivalence to nootkatone.

Statistical Analysis

All results were expressed as means ± standard error (SE). Three cantaloupe samples were analyzed from each variety, and duplicate measurements ($n = 3 \times 2$) were performed for each sample and used to quantify and analyze phytonutrients. The data were subjected to one-way analysis of variance (ANOVA) using Student's *t*-test for mean comparison at $P \leq 0.05$ confidence interval. Results of amino acid data were subjected to

multivariate statistical analysis (principal component analysis PCA) constructed using XLSTAT software (Addinsoft, Paris, France).

Results And Discussion

Influence Of Storage On Physiochemical Characteristics

Fruit firmness

In the present study, the firmness was measured in Newtons (N) as an average of triplicate readings (Table 1). Firmness decreased towards the end of the 20-day storage period. Cantaloupe variety Western Shipper showed maximum loss of fruit firmness from day 0 (22.35 ± 0.52 N) to day 20 (17.78 ± 0.25 N) while Infinite Gold also showed a similar reduction from day 0 (24.4 ± 0.22 N) compared to day 20 (20.28 ± 0.29 N). By contrast, the Orange Casaba and HD252 varieties retained firmness but HD150 lost firmness (24.45 ± 0.3 N at day 0 to 20.6 ± 0.22 N on day 20).

The firmness of fruit flesh is an important quality indicator and our results suggest that postharvest storage leads to noticeable loss of firmness at the end of storage among all melon varieties used in the study. A study showed that dipping melons in hot water protected the cell wall and denatured the enzymes, which helped to maintain rigidity and firmness of fruit flesh. This study evaluated the suppressing activities of enzymes and accumulation of suberin and cellulose, thereby explaining the changes in firmness, which might be due to changes in cell wall composition [42]. Fruit firmness is closely associated with cell wall structure and composition, particularly with cell wall changes during

ripening. The cell wall provides rigidity and strength, and the osmotic pressure of the protoplast exerts force and provides turgor, thereby maintaining fruit firmness. Primary cell walls are extensible, somewhat elastic, and are capable of being loosened to allow growth and the loss of fruit firmness may involve a loosening of cells during fruit storage [43]

Table 1. Firmness of fruits during storage period of all melon varieties. Values represented in as Mean \pm SE (N) Values with different letters between storage days indicate significant differences according to Student's *t*- test ($P < 0.05$)

Firmness (N)					
Variety	0 Days	5 Days	10 Days	15 Days	20 Days
Western Shipper	22.35 \pm 0.52 ^a	23.38 \pm 0.71 ^a	20.41 \pm 0.8 ^b	20.03 \pm 0.26 ^b	17.78 \pm 0.25 ^c
Da Vinci	25.02 \pm 0.43 ^a	24.52 \pm 0.34 ^a	23.31 \pm 0.4 ^b	20.65 \pm 0.24 ^b	22.4 \pm 0.52 ^c
Infinite Gold	24.4 \pm 0.22 ^a	23.13 \pm 0.45 ^{bc}	23.83 \pm 0.09 ^{ab}	22.51 \pm 0.31 ^c	20.28 \pm 0.29 ^d
Orange Casaba	23.7 \pm 0.54 ^a	23.96 \pm 1.07 ^b	23.96 \pm 0.24 ^a	24.03 \pm 0.44 ^a	22.33 \pm 0.2 ^{ab}
HD150	24.45 \pm 0.3 ^a	22.05 \pm 0.36 ^b	22.21 \pm 0.83 ^b	21.31 \pm 0.24 ^{bc}	20.6 \pm 0.22 ^c
HD252	23.33 \pm 0.27 ^a	23.4 \pm 0.24 ^a	22.88 \pm 0.3 ^a	23.42 \pm 0.2 ^a	21.18 \pm 0.6 ^b

Total soluble solids (TSS)

The total soluble solids increase as the storage period progressed and then decreased after 15 days. Honeydew varieties had higher TSS compared to cantaloupe varieties and among the six varieties, we measured the highest TSS in HD150 on day 5 (11.3 \pm 0.2%) and HD252 on day 15 (13.15 \pm 0.95%). These results agree with a previous study of Makdimon melon fruit stored for 48 days, which evaluated the accumulation of sugars in melon fruits as a result of ripening [44]. Progress in ripening during storage might be due to the increase in TSS contents in melon varieties (Table 2).

Table 2. Total soluble solids of melon varieties measured during storage period. Values represented as Mean \pm SE of brix %. Values with different letters between storage days indicate significant differences according to Student's *t*- test ($P < 0.05$)

TSS (%)					
Variety	0 Days	5 Days	10 Days	15 Days	20 Days
Western Shipper	8.85 \pm 0.15 ^a	9.3 \pm 0.2 ^a	7.85 \pm 0.45 ^a	8.9 \pm 0.3 ^a	8.55 \pm 1.05 ^a
Da Vinci	7.93 \pm 0.23 ^a	8.66 \pm 1.06 ^a	7.86 \pm 0.14 ^a	8.3 \pm 0.51 ^a	6.5 \pm 0.9 ^a
Infinite Gold	8.8 \pm 0.1 ^a	9.35 \pm 0.94 ^a	9 \pm 0.8 ^a	8.75 \pm 0.55 ^a	8.65 \pm 0.94 ^a
Orange casaba	9.4 \pm 0.09 ^b	10.85 \pm 0.04 ^a	10.4 \pm 0.09 ^{ab}	10.1 \pm 0.7 ^{ab}	9.6 \pm 0 ^{ab}
HD150	9.6 \pm 0.09 ^{ab}	11.3 \pm 0.2 ^a	9.25 \pm 1.15 ^{ab}	9.9 \pm 0.09 ^b	9.4 \pm 0.09 ^{ab}
HD252	8.85 \pm 0.15 ^{ab}	9.6 \pm 0.3 ^{bc}	10.15 \pm 0.15 ^{abc}	13.15 \pm 0.95 ^{ab}	11.55 \pm 1.45 ^a

Color assessment

The color was assessed by measuring L*, C and hue values. A larger L* value indicates that the surface of the sample is brighter and a* indicates red-green difference (negative value indicates green, positive value indicates red). The larger the absolute value of a*, the darker the red-green or green; b* indicates yellow-blue difference. H* represents the hue angle and a*/b* represents the hue of the color [45]. In cantaloupe varieties, the brightness of flesh increased gradually, and a*, b* values for cantaloupe melon suggested the ripening and accumulation of carotenoids during storage (Table 3). At day 20, hue values calculated based on red-green and blue-yellow ranges decreased indicating a change in fruit flesh composition and quality. In honeydew varieties, green-fleshed fruit maintained a uniform color throughout the storage period with slight changes in a*/b* values (Table 4). Previous studies measured the changes in color of fresh cut cubes and the effect of treatment on the cut pieces when treated with different concentrations of

ascorbic acid, ethylenediaminetetraacetic acid (EDTA), and manganese chloride results demonstrate the color changes were caused due to oxidation of carotenoids in cut pieces [46, 47]. In the present study, fruits measured at each storage interval showed changes in color of which indicates changes in the composition of the fruit flesh during the storage.

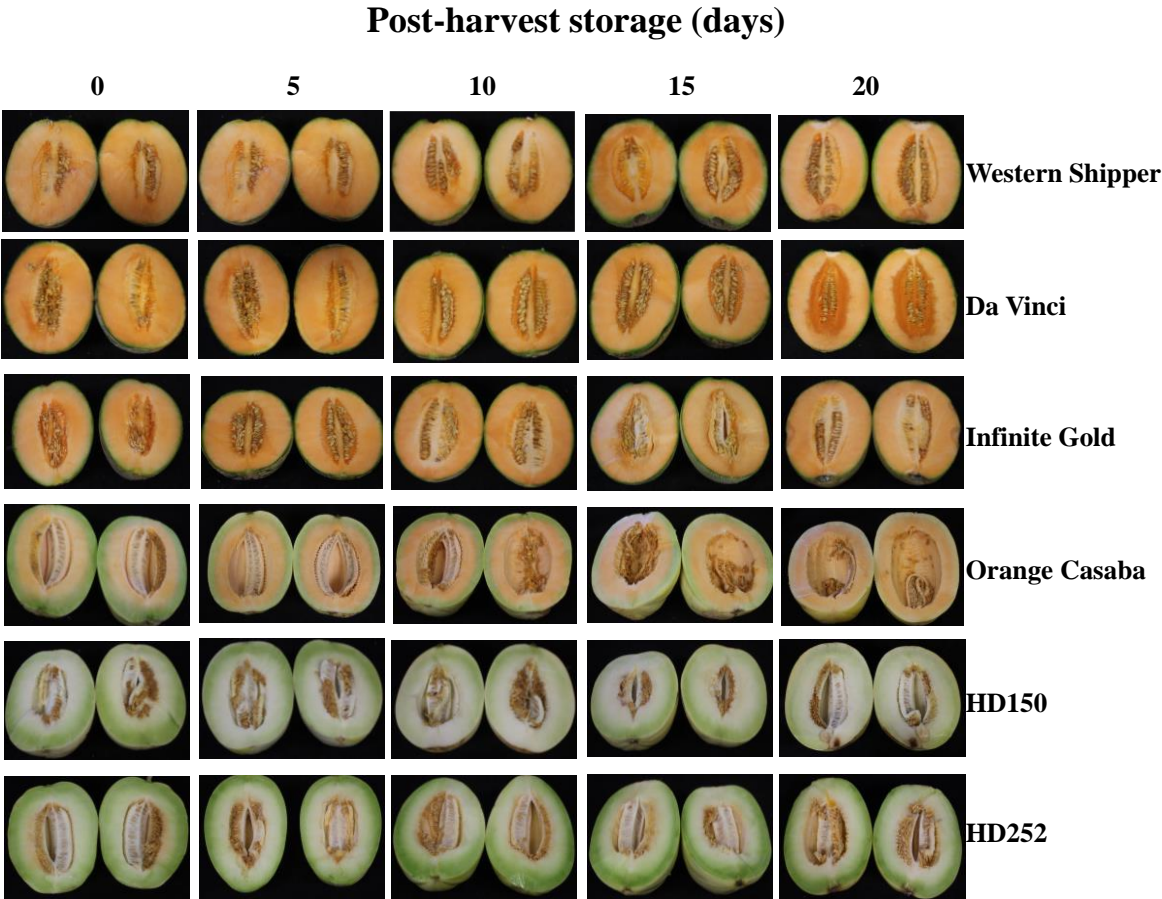


Figure 1. Pictures of typical cut melons used for the study arranged according to variety and storage days

Table 3. Color assessment of cantaloupe melon varieties on each storage day. Each value represents the mean \pm standard error. Values with different letters between storage days indicate significant differences according to Student's *t*- test ($P < 0.05$)

Variety	Storage days	L	a*	b*	C*	Hue
Western Shipper	0	75.378 \pm 0.59 ^a	18.671 \pm 0.622 ^a	45.855 \pm 0.381 ^{ab}	49.523 \pm 0.557 ^{ab}	1.184 \pm 0.009 ^a
	5	73.96 \pm 0.607 ^a	19.036 \pm 0.956 ^a	47.643 \pm 0.681 ^a	51.278 \pm 0.767 ^{ab}	1.191 \pm 0.017 ^a
	10	74.251 \pm 1.857 ^a	17.618 \pm 0.876 ^a	44.211 \pm 0.515 ^b	47.623 \pm 0.655 ^b	1.192 \pm 0.016 ^a
	15	74.68 \pm 0.743 ^a	19.731 \pm 1.178 ^a	47.468 \pm 0.853 ^a	51.443 \pm 1.179 ^a	1.178 \pm 0.016 ^a
	20	75.725 \pm 0.66 ^a	17.331 \pm 0.509 ^a	45.061 \pm 0.675 ^b	47.813 \pm 0.883 ^b	1.203 \pm 0.008 ^a
Infinite Gold	0	78.365 \pm 0.657 ^{ab}	17.08 \pm 0.66 ^{ab}	42.111 \pm 1.735 ^a	45.418 \pm 1.858 ^{ab}	1.184 \pm 0.008 ^b
	5	75.031 \pm 1.529 ^c	15.101 \pm 0.526 ^{bc}	39.505 \pm 1.54 ^a	42.296 \pm 1.607 ^b	1.205 \pm 0.005 ^{ab}
	10	75.59 \pm 0.684 ^{bc}	18.79 \pm 1.219 ^a	40.703 \pm 5.276 ^a	49.343 \pm 1.482 ^a	1.104 \pm 0.066 ^b
	15	74.841 \pm 1.601 ^c	16.765 \pm 0.521 ^{ab}	39.87 \pm 1.458 ^a	43.311 \pm 1.567 ^b	1.172 \pm 0.003 ^b
	20	79.64 \pm 0.582 ^a	12.31 \pm 1.895 ^c	41.638 \pm 1.46 ^a	43.923 \pm 1.497 ^b	1.288 \pm 0.041 ^a
Da Vinci	0	73.072 \pm 0.779 ^b	21.551 \pm 0.49 ^a	48.158 \pm 0.353 ^a	52.775 \pm 0.388 ^a	1.15 \pm 0.008 ^{bc}
	5	76.777 \pm 0.602 ^a	17.751 \pm 0.866 ^b	46.274 \pm 0.683 ^{bc}	49.496 \pm 0.812 ^a	1.205 \pm 0.014 ^a
	10	74.575 \pm 0.903 ^{ab}	17.385 \pm 1.476 ^b	47.178 \pm 0.812 ^{ab}	50.685 \pm 0.934 ^a	1.219 \pm 0.027 ^a
	15	75.488 \pm 0.489 ^{ab}	17.795 \pm 0.586 ^b	44.702 \pm 0.588 ^c	48.127 \pm 0.727 ^a	1.192 \pm 0.008 ^{ab}
	20	68.757 \pm 1.559 ^c	19.193 \pm 0.617 ^{ab}	41.661 \pm 0.549 ^d	47.395 \pm 1.568 ^a	1.138 \pm 0.015 ^c

Table 4. Color assessment of honeydew melon varieties on each storage day. Each value represents the mean \pm standard error. Values with different letters between storage days indicate significant differences according to Student's *t*- test ($P < 0.05$)

Variety	Storage days	L	a*	b*	C*	Hue
Orange Casaba	0	80.098 \pm 0.772 ^a	5.266 \pm 2.367 ^b	35.166 \pm 0.902 ^a	35.913 \pm 1.047 ^a	0.381 \pm 0.599 ^b
	5	80.828 \pm 1.208 ^a	10.406 \pm 0.529 ^a	34.675 \pm 0.987 ^a	36.21 \pm 1.07 ^a	1.279 \pm 0.009 ^a
	10	79.365 \pm 1.164 ^a	10.475 \pm 1.076 ^a	36.085 \pm 1.193 ^a	37.673 \pm 1.063 ^a	1.286 \pm 0.031 ^a
	15	79.863 \pm 0.715 ^a	8.443 \pm 1.555 ^{ab}	36.333 \pm 0.606 ^a	37.44 \pm 0.833 ^a	1.346 \pm 0.039 ^a
	20	80.593 \pm 0.876 ^a	6.94 \pm 1.878 ^{ab}	35.406 \pm 0.782 ^a	36.153 \pm 1.165 ^a	1.38 \pm 0.045 ^{ab}
HD150	0	86.136 \pm 0.309 ^a	-8.486 \pm 0.265 ^{ab}	24.921 \pm 0.209 ^b	25.846 \pm 0.578 ^b	-1.242 \pm 0.01 ^a
	5	83.573 \pm 1.311 ^{ab}	-6.381 \pm 1.999 ^a	25.77 \pm 2.23 ^b	26.831 \pm 2.44 ^b	-0.807 \pm 0.472 ^a
	10	82.071 \pm 0.892 ^b	-10.571 \pm 1.406 ^b	32.233 \pm 1.043 ^a	34.09 \pm 0.986 ^a	-1.253 \pm 0.041 ^a
	15	77.801 \pm 1.336 ^c	-11.821 \pm 1.414 ^b	31.416 \pm 1.227 ^a	33.633 \pm 1.621 ^a	-1.217 \pm 0.028 ^a
	20	80.64 \pm 0.992 ^{bc}	-9.788 \pm 1.172 ^{ab}	31.961 \pm 0.741 ^a	33.5 \pm 0.978 ^a	-1.277 \pm 0.03 ^a
HD252	0	85.263 \pm 0.926 ^a	-8.788 \pm 1.251 ^a	24.336 \pm 1.544 ^a	25.918 \pm 1.868 ^a	-1.232 \pm 0.026 ^a
	5	85.647 \pm 0.806 ^a	-10.67 \pm 0.598 ^a	26.555 \pm 0.66 ^a	28.638 \pm 0.808 ^a	-1.19 \pm 0.013 ^a
	10	84.57 \pm 0.594 ^a	-10.306 \pm 0.699 ^a	26.782 \pm 0.721 ^a	28.728 \pm 0.888 ^a	-1.206 \pm 0.017 ^a
	15	83.724 \pm 0.703 ^a	-10.227 \pm 0.892 ^a	26.107 \pm 1.207 ^a	28.078 \pm 1.418 ^a	-1.204 \pm 0.019 ^a
	20	79.7 \pm 5.305 ^a	-9.992 \pm 0.914 ^a	26.941 \pm 1.064 ^a	28.783 \pm 1.292 ^a	-1.22 \pm 0.018 ^a

Analysis Of Total Phenolic Contents And Antioxidant Activities

Total Phenolic Contents

Postharvest abiotic stresses may affect the levels of secondary metabolites in crop tissues by affecting the pathways involved in the biosynthesis of secondary metabolites, mainly phenolic compounds [48]. Results of total phenolic contents (TPC) are represented as gallic acid equivalents (GAE). On day 0 storage, Da Vinci (201.68 ± 6.31 mg kg⁻¹ GAE) and Western Shipper (166.48 ± 8.22 mg kg⁻¹ GAE) had high TPC (Fig. 2A). After day 5, Western Shipper showed higher TPC (169.80 ± 9.59 mg kg⁻¹ GAE) than other cantaloupe varieties while HD150 (162.05 ± 4.69 mg kg⁻¹ GAE) had higher TPC than other honeydew varieties. The variation in TPC in melons was observed in different storage periods may due to degradation or breakdown of phenolic compounds [49] Another study reported similar trend in TPC measured on day 0 (243.8 ± 50.4 mg kg⁻¹ GAE) compared to day 9 (232.1 ± 29.4 mg kg⁻¹ GAE), which shows reduced levels during storage in cantaloupe [50]. However, lower TPC in cantaloupe juice (95.35 ± 9.23 mg kg⁻¹ GAE) and pulp (101.90 ± 14.99 mg kg⁻¹ GAE) were also reported [51].

TPC was also influenced by melon cultivar, harvesting location, and growing conditions. In a recent study, different melon varieties grown in multiple locations in the US showed variation in TPC. Western Shipper and Da Vinci melons harvested from Indiana (489.76 ± 15.49 mg kg⁻¹ GAE) and North Carolina (381.91 ± 33.82 mg kg⁻¹ GAE) had higher TPC compared to other growing locations [40]. Similarly, in our results, Western Shipper and Da Vinci showed higher TPC compared to other varieties harvested from Uvalde, TX.

Results of the DPPH and ABTS assay are represented as ascorbic acid equivalents. The DPPH assay demonstrated that cantaloupe had high antioxidant activity after 5 days of storage (Fig 2 B). On day 5, Western Shipper ($56.38 \pm 1.04 \text{ mg kg}^{-1}$) and Da Vinci ($44.50 \pm 1.09 \text{ mg kg}^{-1}$) showed higher activity while in honeydew, higher activity was observed in HD252 ($67.05 \pm 1.23 \text{ mg kg}^{-1}$) followed by HD150 ($63.40 \pm 1.2 \text{ mg kg}^{-1}$) (Fig 2 B). The ABTS assay showed high activity in Western Shipper ($208.97 \pm 11.83 \text{ mg kg}^{-1}$) followed by Da Vinci ($177.78 \pm 10.40 \text{ mg kg}^{-1}$) at day 0 while HD252 ($168.12 \pm 10.65 \text{ mg kg}^{-1}$) was highest among analyzed honeydews. After day 10, ABTS activity decreased as the storage period increased (Fig 2 C).

The radical scavenging patterns observed in the present study suggested that fluctuation in antioxidants activity occurred in most fruits during postharvest storage [52]. A previous study on antioxidant activity of six different cantaloupe varieties shows similar DPPH and ABTS activities in Western Shipper variety harvested from AZ and TX had the highest DPPH ($115.46 \pm 10.6 \text{ mg kg}^{-1}$) and ABTS activity ($231.01 \pm 56.85 \text{ mg kg}^{-1}$), respectively [40].

The difference in DPPH and ABTS may reflect the discrepancies of reaction kinetic mechanisms between these assays [53]. The reactivity of the compound is dependent on the position of functional groups [49] and in melon extracts this may be one of the factors influencing the results. In the DPPH assay, low antioxidant capacity may result from weak reactions and certain compounds such as ascorbic acid and phenolics may autoxidize oxygen to O_2 which reacts rapidly with ABTS radicals resulting in increased activity [54].

The levels of antioxidant activity in fruits may be influenced by storage temperature. The continuous process of ripening during storage resulting in fluctuations of antioxidant levels in fruits [55]. This change in phenolic contents could be attributed to greater accumulation of products from the oxidation of phenolics and additional release due to stress factors [56]. Similar storage study suggested that there were large fluctuations in antioxidant activities measured by DPPH and ABTS method due to the accumulation of phenolic compounds in fruits during the storage period [57]. From the results obtained, it can be assumed that the nature of antioxidants present in fruits determines the levels of antioxidant potential.

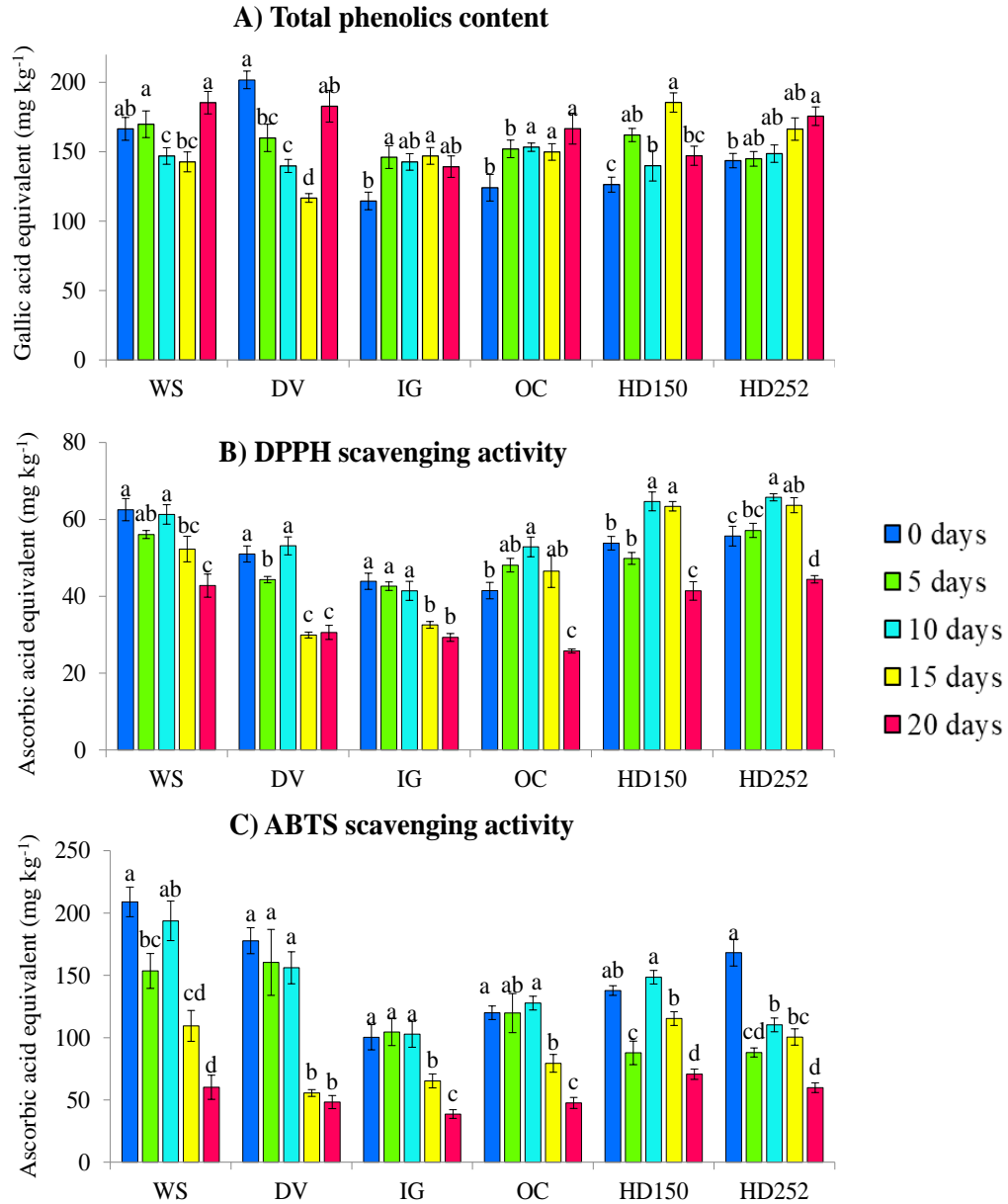


Figure 2. A) Total phenolic contents of melon varieties during storage. Results (mean \pm SE) are expressed as mg gallic acid equivalent kg^{-1} . B) DPPH scavenging activity and C) ABTS scavenging activity. Results (mean \pm SE) are expressed as μg ascorbic acid equivalents

Quantification Of Carotenoids

Eleven carotenoids were successfully quantified at each storage interval in all varieties. Earlier, studies focused on total carotenoids, β -carotene, and lutein; this is the first report of detection and quantification of 11 carotenoids during postharvest storage of cantaloupe and honeydew varieties. In the present study, detection and quantification of the colorless carotenoids, phytoene and phytofluene, were carried out at 286 nm and 350 nm, respectively (Fig.3). Phytoene and phytofluene serve as precursors for ζ -carotene and lycopene during the biosynthesis of carotenoid molecules. Among cantaloupe varieties, the highest content of phytoene (0.69 ± 0.06 mg kg⁻¹) and phytofluene (0.93 ± 0.04 mg kg⁻¹) was recorded in Da Vinci followed by Western Shipper (0.32 ± 0.07 mg kg⁻¹ and 0.67 ± 0.07 mg kg⁻¹) (Fig. 3 A & B). Orange Casaba showed phytoene (0.08 ± 0.02 mg kg⁻¹) and phytofluene (0.26 ± 0.05 mg kg⁻¹) content at day 0; this increased to 0.16 ± 0.03 mg kg⁻¹ and 0.24 ± 0.05 mg kg⁻¹, respectively after 15 days. Phytoene and phytofluene were not detected in HD150 and HD252.

Quantification of β -cryptoxanthin and β -carotene was carried out at 450 nm using their reference compounds. As storage progressed, a gradual accumulation of β -cryptoxanthin was observed in all cantaloupe types. On day 20, it was highest in Western Shipper (0.40 ± 0.01 mg kg⁻¹), followed by Infinite Gold (0.35 ± 0.01 mg kg⁻¹) and Da Vinci (0.33 ± 0.07 mg kg⁻¹). Orange Casaba showed high levels of β -cryptoxanthin (0.28 ± 0.06 mg kg⁻¹) on day 10 (Fig 3C). Levels of β -carotene were highest in Da Vinci (24.27 ± 0.88 mg kg⁻¹), followed by Western Shipper (17.60 ± 0.43 mg kg⁻¹) at 0 day storage and later decreased (Fig 3G). It is noteworthy that levels of β -carotene and β -cryptoxanthin showed

an inverse trend as storage progressed. This might be due to the conversion of β -carotene to β -cryptoxanthin, which generally occurred during postharvest storage [58, 59].

Quantification of ζ -carotene isomers was carried out at 400 nm as per the reference compound, ζ -carotene. Among the four isomers, the levels of isomer 1 and 3 gradually increase as storage progressed. The highest content of isomer 1 was in Western Shipper on day 0 ($4.39 \pm 0.12 \text{ mg kg}^{-1}$ to $6.85 \pm 0.18 \text{ mg kg}^{-1}$) and the highest isomer 3 levels were observed in Da Vinci on day 0 ($5.52 \pm 0.41 \text{ mg kg}^{-1}$ to $10.19 \pm 0.54 \text{ mg kg}^{-1}$) followed by Western Shipper ($4.49 \pm 0.36 \text{ mg kg}^{-1}$ to $8.35 \pm 0.65 \text{ mg kg}^{-1}$ on day 20) (Fig. 3 D & H). Interestingly, isomer 2 and 4 (Fig. 3 E & I) increased up to 15 days in Western Shipper ($1.55 \pm 0.22 \text{ mg kg}^{-1}$) and isomer 4 was highest in Da Vinci ($0.59 \pm 0.07 \text{ mg kg}^{-1}$). The levels later decreased towards the end of storage. The elevated levels of α -carotene after day 15 suggested that isomer 2 and 4 might be converted to α -carotene.

The ζ - carotene isomers 1, 2, 3, and 4 detected in the melon varieties indicate their role in forming lycopene isomers or their further conversion to α -carotene. Lycopene isomer levels were highest in Infinite Gold and Da Vinci varieties on day 0 ($0.33 \pm 0.02 \text{ mg kg}^{-1}$) and ($0.32 \pm 0.02 \text{ mg kg}^{-1}$) increased at day 15 ($0.37 \pm 0.01 \text{ mg kg}^{-1}$ and $0.04 \pm 0.03 \text{ mg kg}^{-1}$) and decreased at day 20 (Fig.3 J & K). These results suggested that the changes in the carotenoid biosynthetic pathway involves the conversion of phytoene to lycopene [58].

Melons are rich sources of β -carotene; for example, a cantaloupe melon of unknown variety obtained from Guatemala had high β -carotene (38.6 mg kg^{-1} FW [fresh weight]) but low quantities of ζ -carotene and β -cryptoxanthin [60]. Another study reported

the presence of phytoene (0.38 mg kg^{-1}) and phytofluene (0.44 mg kg^{-1}) in cantaloupe [61]. Our results suggest that the changes in carotenoids take place during the storage period of 20 days, the biochemical changes may be influenced by various factors such as melon cultivar and genotype, storage temperature and storage duration. However, published studies on postharvest storage of grapefruits stored at $11 \text{ }^{\circ}\text{C}$ for 12 weeks showed similar trends in results β -carotene contents. β -carotene was initially present at lower levels and then increased during storage [62]. Carotenoid biosynthesis in citrus fruits is temperature dependent, with temperatures of $5\text{--}25^{\circ}\text{C}$ allowing for the most carotenoid production [59]. The results obtained from the present study also suggest that the occurrence of different carotenoids in melon varieties is expected during postharvest storage.

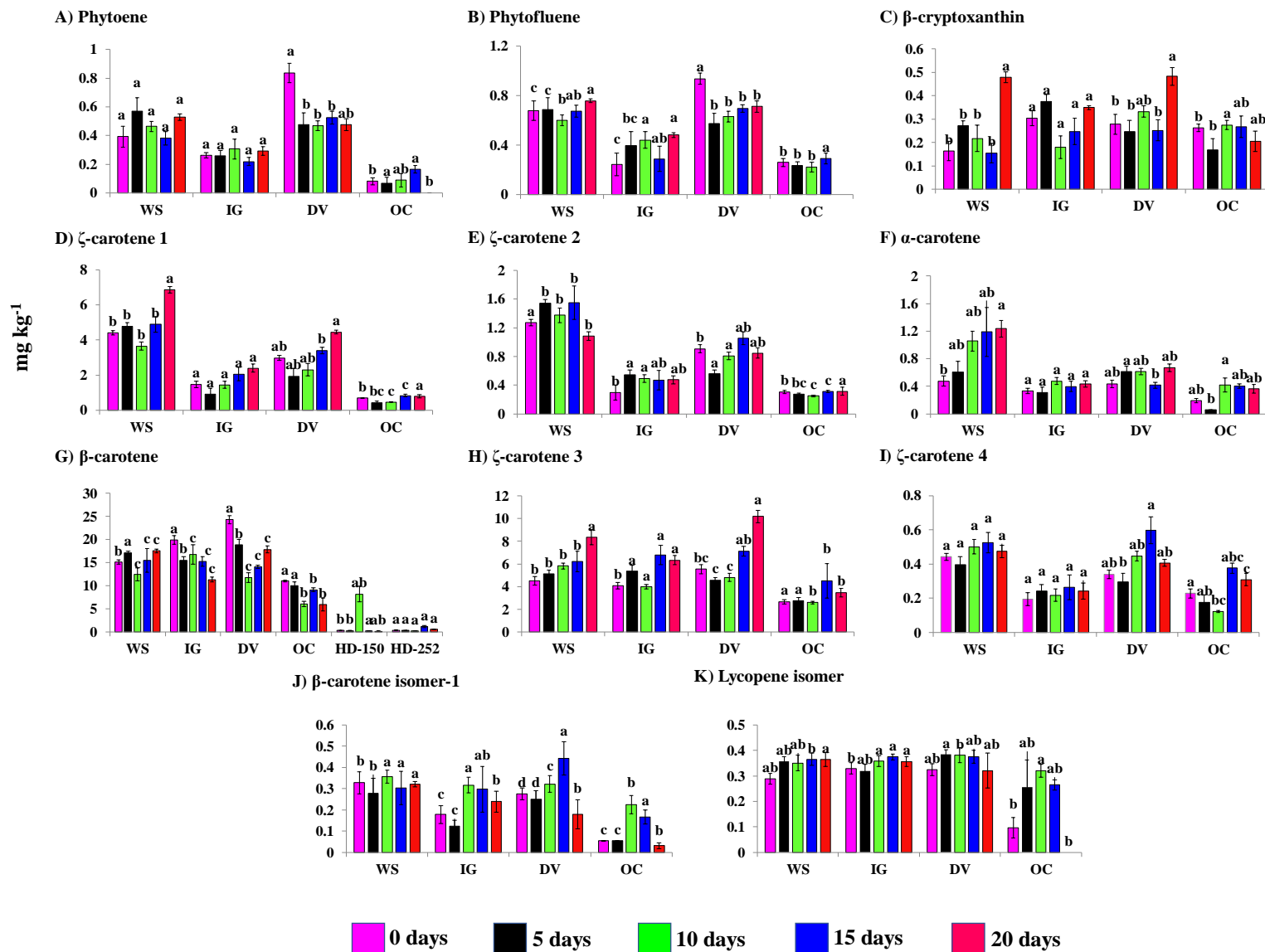


Figure 3. Carotenoid content in different melon varieties Western Shipper (WS), Da Vinci (DV), Infinite Gold (IG), Orange Casaba (OC), Honeydew 150 (HD150), and Honeydew 252 (HD252) stored for 20 days postharvest. Changes quantified during postharvest storage A) phytoene, B) phytofluene, C) β -cryptoxanthin, D) ζ - carotene-1, E) ζ - carotene-2, F) α -carotene, G) β -carotene, H) ζ - carotene-3, I) ζ - carotene-4 , J) β -carotene isomer-1, and K) Lycopene isomer. Phytofluene and Phytoene were quantified using standard phytoene, β -cryptoxanthin quantified based on standard β -cryptoxanthin. ζ - carotene isomers were quantified based on standard β -carotene measured at 400 nm. β -carotene, β -carotene isomer -1 and α -carotene were quantified based on standard β -carotene at 450 nm. Lycopene isomer was quantified based on standard Lycopene. Results are presented as mean \pm SE obtained from values of triplicate samples replications with from each fruit. For each compound, different letters indicate significant differences ($P \leq 0.05$) among storage period

Evaluation Of Ascorbic Acid Levels During Storage

The total ascorbic acid content was calculated by summing up the values of ascorbic acid (AA) and dehydroascorbic acid (DHA) (Fig. 4a(A-C)). AA is readily oxidized under physiological conditions in plant and animal tissue to form DHA. The reducing agent TCEP (tris(2-carboxyethyl)phosphine) is used to reduce AA to DHA and then the levels are used to determine the levels of total ascorbic acid [63]. The AA and DHA concentrations were highest in Western Shipper and Da Vinci on day 0, when the AA concentration was $54.98 \pm 3.12 \text{ mg kg}^{-1}$ and $43.67 \pm 6.68 \text{ mg kg}^{-1}$, respectively, and the DHA concentration was $57.32 \pm 15.07 \text{ mg kg}^{-1}$ and $63.66 \pm 15.73 \text{ mg kg}^{-1}$, respectively (Fig.4 B). HD150 and HD252 did not show significant levels of AA and a low concentration of DHA was observed on day 0. On day 5 AA increased significantly in all varieties except HD150 and HD252, while DHA highest in HD150 with $41.27 \pm 9.70 \text{ mg kg}^{-1}$ and HD252 with $74.19 \pm 5.53 \text{ mg kg}^{-1}$ compared with other varieties AA and DHA diminished noticeably at day 10 interval and further declined after day 10 storage in all varieties. Interestingly, the levels of DHA were typically higher after 20 days of storage compared to other storage days. DHA is the oxidation product of AA. The higher level of DHA observed after 20 days of storage may be in response to higher stress during prolonged cold storage. A detailed study of the degradation of AA explains the possible pathway that leads to apoplastic H_2O_2 , which is proposed to loosen the cell wall and enhance cell expansion and fruit softening [64].

Evaluation of the amount of sample required for AA, DHA, and total ascorbic acid (TAA) analysis is shown in (Fig. 4b (A-C)). Results of the HPLC analysis indicate the

trend in AA values. Among all measurements, 50 mg showed low ascorbic acid ($12.28 \pm 0.87 \text{ mg kg}^{-1}$) and dehydroascorbic acid ($46.65 \pm 3.17 \text{ mg kg}^{-1}$). The values increased when 250 mg was analyzed for AA ($48.03 \pm 0.87 \text{ mg kg}^{-1}$) and DHA ($203.60 \pm 6.00 \text{ mg kg}^{-1}$). However, increasing the amount of sample from 250 mg to 500 mg showed no significant differences in the values of AA and DHA; for example the AA content in the 500-mg sample ascorbic acid ($49.60 \pm 1.24 \text{ mg kg}^{-1}$) and DHA ($225.46 \pm 6.38 \text{ mg kg}^{-1}$) was very similar to that in the 250-mg sample ($48.03 \pm 1.24 \text{ mg kg}^{-1}$ and DHA ($203.60 \pm 6.00 \text{ mg kg}^{-1}$). From this analysis, the minimum quantity that can be used for ascorbic acid analysis is 250 mg, with 1.5 to 2.0 mL solvent for extraction.

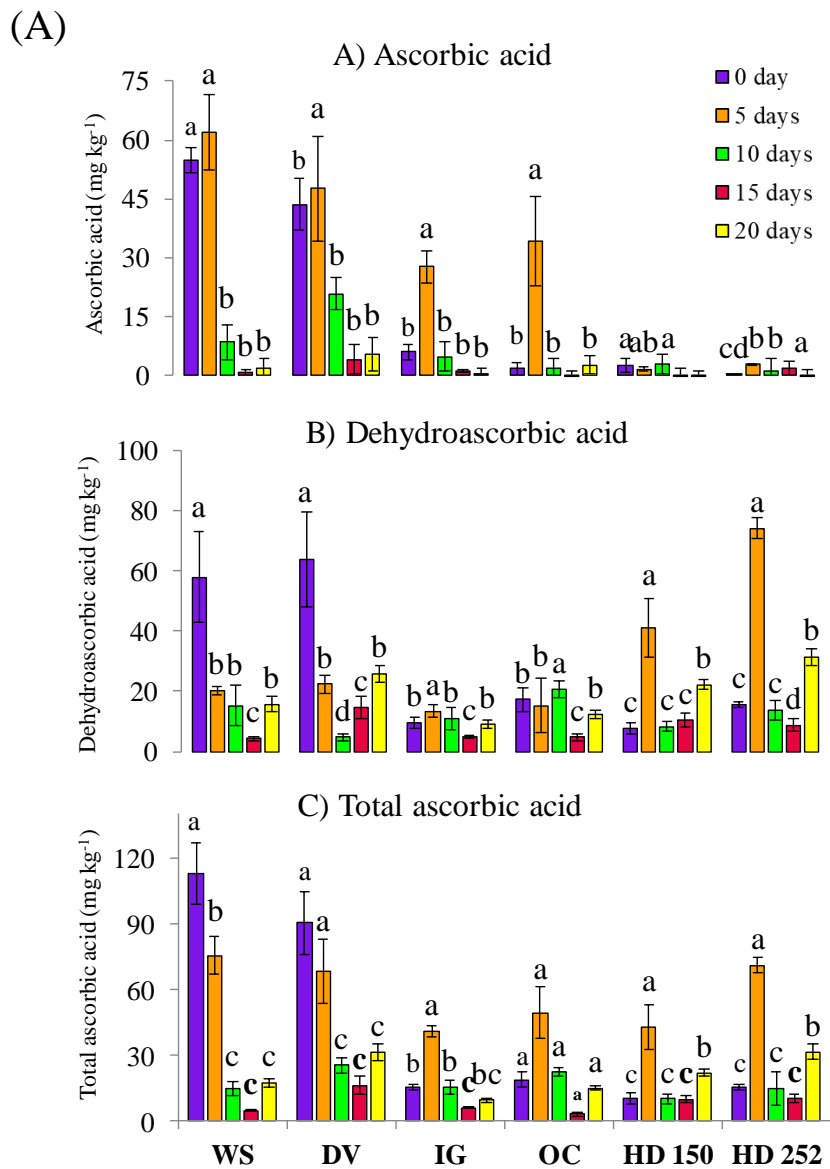
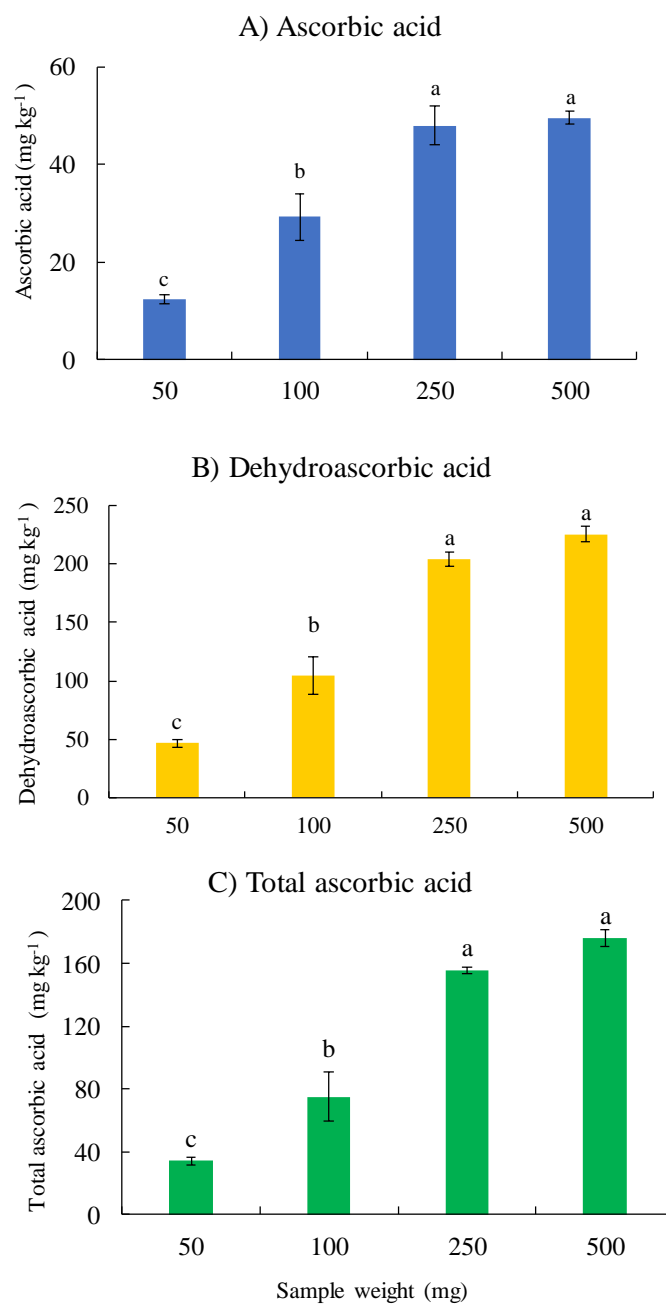


Figure 4. (A) Effect of postharvest storage days on A) ascorbic acid, B) dehydroascorbic acid and C) total ascorbic acid in melon varieties harvested from Uvalde, TX. Results are presented as mean \pm SE from three replications. For each compound, different letters indicate significant differences ($P \leq 0.05$) among storage periods. (B) Melon samples analysed for A) ascorbic acid, (B) dehydroascorbic acid and C) total ascorbic acid from low sample quantity. Results are presented as mean \pm SE from two replications. For each sample weight, different letters indicate significant differences ($P \leq 0.05$) among samples

Figure 4 Continued

(B)



Effect Of Storage On The Concentration Of Amino Acids

Amino acid metabolism is an important biochemical adaptation to environmental stress and temperature of the environment during the storage of fruits. The postharvest accumulation of amino acids observed in crops is a common metabolic response to abiotic stress after harvest [65, 66]. It was reported that various factors lead to changes in concentrations and are assumed to participate in biosynthetic pathways such as the citric acid cycle and upregulation of the urea cycle in some cases [67]. A report on the metabolic profile of Arabidopsis plants speculated that the response to temperature stress, cold acclimation, and biochemical activity were associated with the accumulation of amino acids, due to upregulation of the urea cycle [68]. To understand the trend of the postharvest changes, quantification of amino acids was carried out in each variety. The results demonstrated that glutamine, aspartic acid, and γ -aminobutyric acid (GABA) concentrations changed throughout the storage period compared with freshly harvested fruit and decreased towards the end of storage period. The amino acids content in different cantaloupe and honeydew varieties are presented in the APPENDIX (Table A-1 and Table A-2).

Among the varieties, glutamine was highest in Infinite Gold on day 0 (1219 ± 37.04 mg kg⁻¹) but decreased (103 ± 48.10 mg kg⁻¹) after 20 days of storage. Similarly, honeydew variety HD252 had high glutamine content (1422.90 ± 35.43 mg kg⁻¹) on 0 day and this decreased to 323.58 ± 136.79 mg kg⁻¹ after 20 days. The aspartic acid content in Da Vinci was 1092.84 ± 105.79 mg kg⁻¹ on 0 day, which increased (2402.02 ± 176.63 mg kg⁻¹) at the end of the storage period (20 days). However, in HD252, the level of aspartic acid

decreased during the storage period from 0 days ($1370.83 \pm 261.60 \text{ mg kg}^{-1}$) to 20 days ($1253.05 \pm 89.35 \text{ mg kg}^{-1}$). The level of GABA also decreased in all studied varieties. In Da Vinci, GABA content was $2985.04 \pm 79.17 \text{ mg kg}^{-1}$ at day 0, and decreased to $2426.89 \pm 102.57 \text{ mg kg}^{-1}$ at day 20. Similarly, in HD252, GABA content was $2352.20 \pm 96.34 \text{ mg kg}^{-1}$ at day 0 and $1624.14 \pm 143.16 \text{ mg kg}^{-1}$ after 20 days. Each variety showed a different accumulation pattern of amino acids and this may be due to the influence of variety and sensitivity to storage stress. It was reported that in Arabidopsis seeds, the metabolic pathway involving enzymes and reactions in the GABA shunt was particularly active during abiotic stress [69]. The total amino acid contents described below predominantly reflected the high GABA levels, showing the importance of GABA and stress signaling during cold storage.

The sum of 22 free amino acids corresponding to each variety and storage days were represented as total amino acids (TAA) content (Table 5). At the initial storage period high TAA levels were observed on day 0 in Da Vinci ($7168.17 \pm 349.85 \text{ mg kg}^{-1}$) which increased at day 20 ($8771.44 \pm 626.01 \text{ mg kg}^{-1}$). Among the honeydew varieties, HD252 showed high TAA on day 0 ($6840.07 \pm 611.15 \text{ mg kg}^{-1}$) and reduced to $6273.39 \pm 553.02 \text{ mg kg}^{-1}$ after 20 days. The changes during postharvest storage in melon may result from the breakdown of proteins and used to form other metabolites or participate in biosynthetic pathways. As per our knowledge, this is the first report on amino acid analysis during the postharvest storage of melons.

Table 5. Total amino acid levels of melon varieties during storage period represented as mean \pm SE

Variety	0 Days	5 Days	10 Days	15 Days	20 Days
Western Shipper	6516.71 \pm 428. 76	8290.22 \pm 450. 29	4910.8 \pm 323.7 3	6129.58 \pm 535. 28	6948.86 \pm 469. 29
Infinite Gold	6843.37 \pm 339. 8	6438.35 \pm 605. 2	6088.53 \pm 362. 3	6054.01 \pm 774. 27	7399.53 \pm 329. 84
Da Vinci	7168.17 \pm 349. 85	7743.05 \pm 480. 04	6404.5 \pm 198.9 9	7455.49 \pm 456. 31	8771.44 \pm 626. 01
Orange Casaba	6152.54 \pm 546. 57	7992.98 \pm 590. 67	5913.85 \pm 249. 15	8290.09 \pm 720. 2	7465.38 \pm 384. 66
HD150	5505.77 \pm 419. 6	6923.72 \pm 286. 88	4575.86 \pm 183. 63	6057.46 \pm 548. 38	7007.23 \pm 538. 26
HD252	6840.07 \pm 611. 15	6016.71 \pm 431. 11	5145.15 \pm 272. 09	5833.15 \pm 424. 37	6273.39 \pm 553. 02

Biogenic Amines During Storage

Two biogenic amines were identified in the melon samples. Amines are synthesized most commonly by the decarboxylation of amino acids and these compounds are present in a wide range of foods including fruits and vegetables [70]. The formation of putrescine is initiated by the breakdown of arginine via the arginine decarboxylase cycle and spermidine is formed [71] Putrescine and spermidine were present in all melon varieties. Fluctuations in the arginine level may be due to the conversion of arginine to putrescine, which is further converted to spermidine; indeed, the levels of putrescine and spermidine were inversely related (Fig 5). Among cantaloupe varieties, Infinite Gold showed the high concentration of putrescine on day 0 (1.10 \pm 0.44 mg kg⁻¹) and low concentration of spermidine (0.11 \pm 0.03 mg kg⁻¹). However, after 20 days, a decrease in the levels of putrescine (0.55 \pm 0.03 mg kg⁻¹) was recorded along with an increase in the levels of spermidine (0.44 \pm 0.03 mg kg⁻¹). Among honeydew varieties, HD150 showed the

highest putrescine levels on day 15 ($0.93\pm 0.03 \text{ mg kg}^{-1}$) and spermidine ($0.21\pm 0.01 \text{ mg kg}^{-1}$) these levels decreased at the end of storage.

A study on fermented vegetables reported the occurrence of putrescine [72]. Similarly, a study on biogenic amine profiles in red wine stored at different temperatures showed reduced levels of putrescine in wines stored at 4°C at the end of storage and another study on fruit juices showed the occurrence of biogenic amines due to processing or during storage [73]. This suggests that biogenic amines are ubiquitously present in fruits and storage influences biogenic amine levels in melon fruits. Analysis of biogenic amines indicates the quality of fruit and level of microbial degradation; the results obtained show low levels at the end of storage.

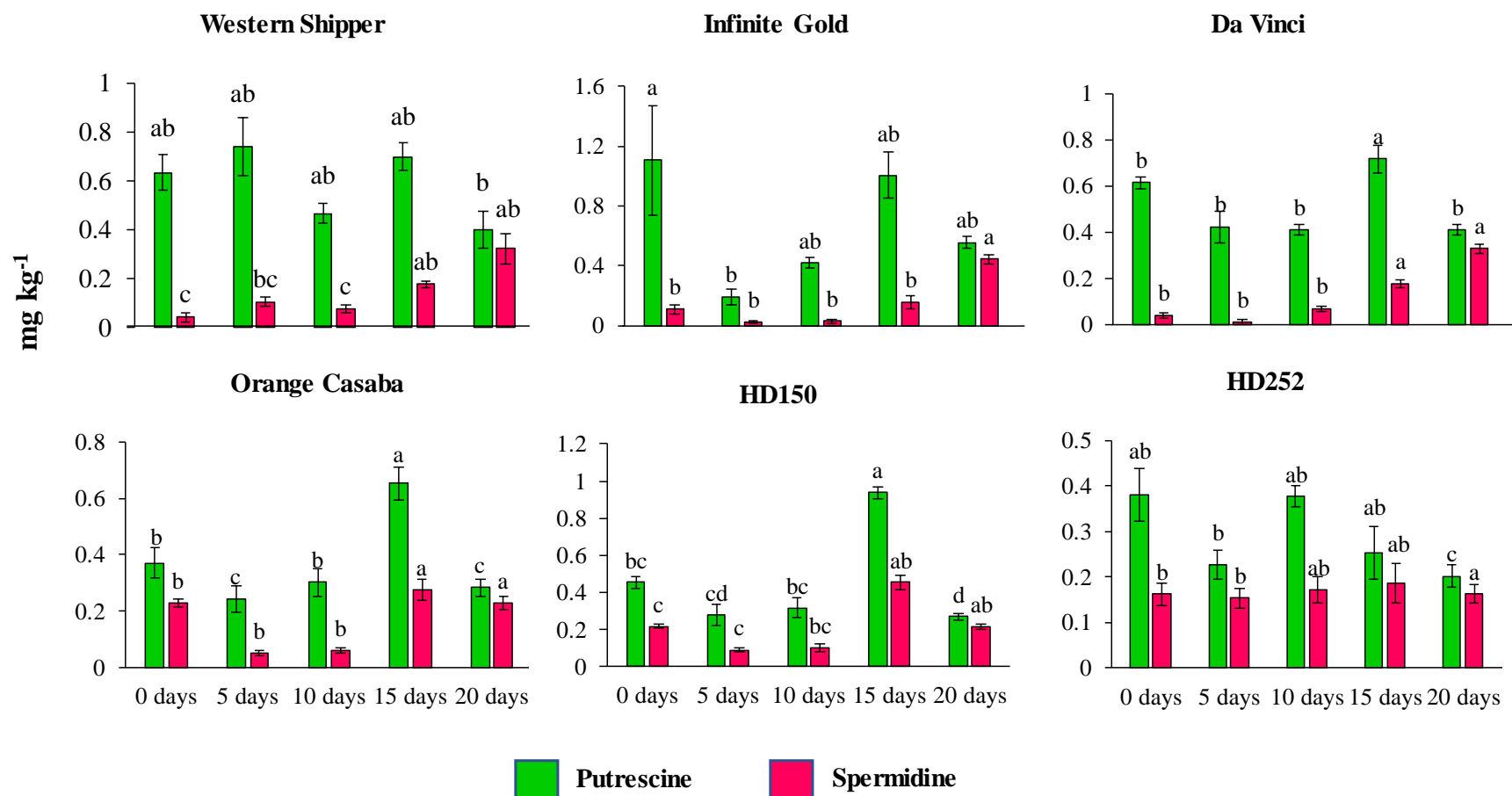


Figure 5. Level of biogenic amines in each variety during postharvest storage. Results are presented as mean \pm SD from three replications. For each compound, different letters indicate significant differences ($P \leq 0.05$) among storage periods

Principal Component Analysis Of Amino Acids

A principal component analysis (PCA) was performed to determine the difference in the amino acid profiles of three cantaloupe varieties (Fig. 6). The first two principal components described 50.11% of the total variance with F1 contributing 26.66% and F2 contributing 23.45%. The F1 axis separated 0, 5, and 10 days from 15 and 20 days. The F1 axis was defined primarily by hydroxyproline, β -alanine, and leucine on the positive side and glycine, valine, proline, and phenylalanine on the negative side. The F2 axis completely separated 20 days from 0, 5, 10, and 15 days which was primarily defined by arginine, serine, and methionine on the positive side with tryptophan and isoleucine on the negative side. Overall, the Western Shipper variety day 0 and day5 separation was not profound compared to other cantaloupe varieties; this was due to the increase in serine, β -alanine, and methionine. At 5 days and 0 days the separation observed in Western Shipper determined by high concentrations of valine and proline.

PCA was also performed to visualize the amino acid distribution of honeydew cultivars (Fig. 7). The first two principal components account for 62.91% of the total variance with F1 contributing 46.92% and F2 contributing 15.99%. F1 separated 5 and 15 days from 0, 10, and 20 days which was primarily defined by asparagine, threonine, methionine, valine, tryptophan, isoleucine, and leucine on the positive side and arginine, citrulline, hydroxyproline, beta alanine, alanine, and proline on the negative side. The F2 axis separates 5 and 20 days from 0, 10, and 15 days and is defined by aspartic acid on the positive side and glycine on negative side. However, due to high concentrations of aspartic acid in Orange Casaba at day 15 and low concentrations of glutamine and beta-alanine in

HD252 at day 5, these two cultivars/storage periods were not in proximity with other honeydews at day 15 and day 5, respectively. In conclusion, PCA shows that amino acid profiles changed through storage period and day 15 and day 20 are clearly separated, indicating the postharvest storage effect on amino acids in cantaloupe and honeydew melons.

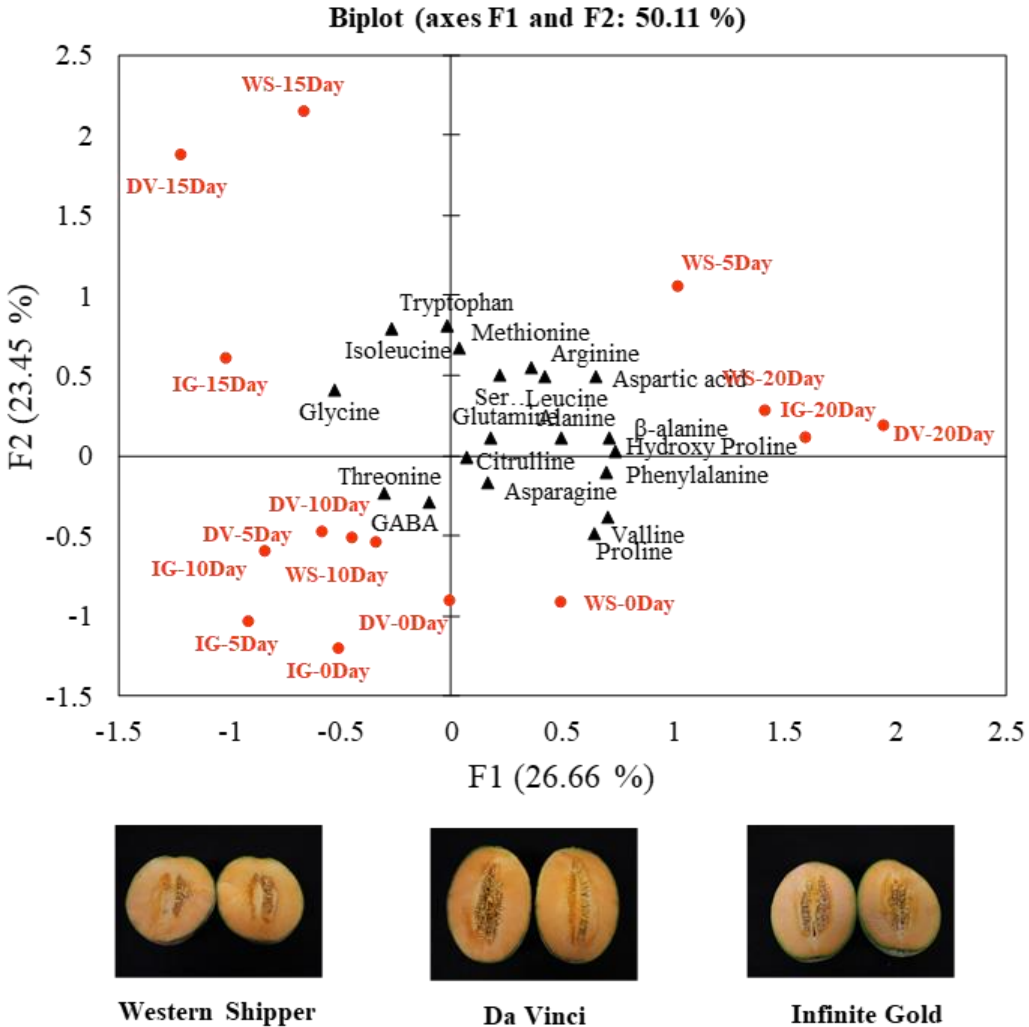


Figure 6. Principal component analysis of average amino acids scores of cantaloupe varieties Western Shipper (WS), Da Vinci (DV), and Infinite Gold (IG) stored 20 days. Fruits were collected at 0, 5, 10, 15, and 20 days of storage for analysis

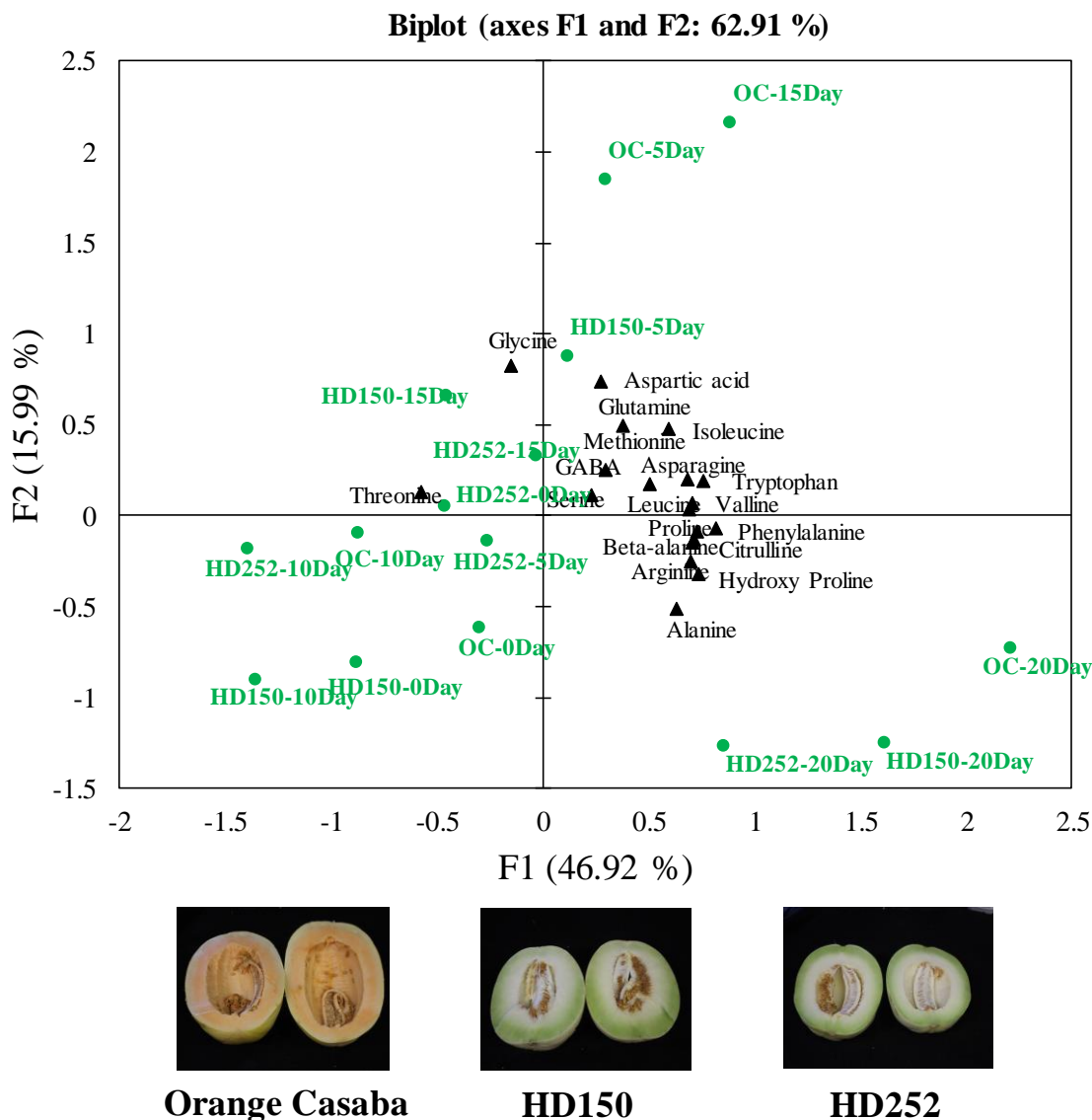


Figure 7. Principal component analysis model of average amino acids scores of honeydew varieties Orange Casaba (OC), Honeydew 150 (HD150), and Honeydew 252 (HD252) stored for 20 days postharvest. Fruits were collected at 0, 5, 10, 15, and 20 days of storage for analysis

Volatile Compounds During Postharvest Storage

The occurrence of various aromatic compounds during storage and the evaluation of melon samples suggests the changes in volatile compound concentrations occur towards the end of the storage period. A total of 79 volatile compounds in cantaloupe and 76 volatile compounds in honeydew varieties were identified and quantified. The volatile compounds were grouped into nine chemical groups: esters, alcohol, aldehydes, ketones, furans, acids, sulphurs, hydrocarbons, and others (Fig. 8).

The volatile composition of fruits includes a combination of esters, alcohols, acids, aldehydes, ketones, acetals, hydrocarbons, ethers, and heterocyclic compounds that have different biochemical pathways for aroma production [20, 74]. Esters contribute the characteristic odor of fruit and contribute to their overall quality perception; ester production in melon fruits is regulated by ethylene [44, 75]. In our study esters were observed in all cantaloupe varieties and lower amounts in honeydew varieties. The total ester content (Fig. 8) was defined by presence of ethyl 2-methylbutanoate, ethyl hexanoate, ethyl caprylate, ethyl 2,4-hexadienoate, methyl benzoate, ethyl decanoate, and fatty acid esters such as methyl hexadecanoate, ethyl hexadecanoate, and ethyl 9-hexadecenoate. Total esters increased as storage period increased in Western Shipper ($528.34 \mu\text{g kg}^{-1}$) and Infinite Gold ($125.13 \mu\text{g kg}^{-1}$), while Da Vinci increased until day 15 ($567.341 \mu\text{g kg}^{-1}$) and decreased at day 20. As suggested by Lamikanra et al., the reduced concentrations of esters observed at the end of storage suggests loss of freshness and reduced pleasant aroma in melon varieties. [76]. This agrees with the results of this

study, as the total esters were lower on day 20 of storage compared with day 0 in all melon varieties except Western Shipper and Infinite Gold.

Alcohol compounds such as 2-methylbutanol, benzyl alcohol, 1-octanol, *Z*-3-nonen-1-ol (green), (*E,Z*)-3,6-nonadien-1-ol (cucumber smell), (*E*)-2-octen-1-ol, 3-phenylpropanol (sweet floral), phenylethyl alcohol, and β -ionol were identified in cantaloupe and honeydew varieties. Overall, the total alcohol compounds tended to increase as storage progressed. However, increased alcohol production is often considered part of senescence in fruit [77]. Benzyl alcohol increased substantially from day 0 ($83.52 \pm 10.61 \mu\text{g kg}^{-1}$) to day 20 ($851.39 \pm 238.12 \mu\text{g kg}^{-1}$) in Da Vinci. Honeydew varieties showed high concentrations of C9 alcohol compounds such as (*Z*)-3-nonen-1-ol, (*Z*)-6-nonen-1-ol, and (*E,Z*)-3,6-nonadien-1-ol, but these alcohols were low in cantaloupe varieties. The results agree with other reports in the literature where the presence of C9 alcohol compounds contributes an undesirable green smell and is strongly associated with honeydew melon [23, 78].

The monoterpene alcohols α -terpineol, (*E*)-carveol, and the sesquiterpene alcohols elemol, α -cadinol, δ -cadinol, globulol, and α -eudesmol were also detected. Elemol (sweet notes) was detected in Western Shipper on day 0 ($2.83 \pm 0.54 \mu\text{g kg}^{-1}$) and increased at day 15 ($59.4 \pm 9.27 \mu\text{g kg}^{-1}$). Eugenol (clove-like scent) was identified only in the cantaloupe Infinite Gold on day 10 ($5.66 \pm 0.78 \mu\text{g kg}^{-1}$) and increased on day 20 ($15.59 \pm 5.16 \mu\text{g kg}^{-1}$). δ -Cadinol was detected at the highest levels in HD150 honeydew on day 20 ($77.38 \pm 25.95 \mu\text{g kg}^{-1}$). Western Shipper ($139.82 \pm 9.27 \mu\text{g kg}^{-1}$) and Da Vinci showed high concentrations of α -terpineol (floral, citrus-woody notes).

Aldehydes detected in melon varieties included benzaldehyde (pleasant sweet), nonanal (rose like, floral), β -cyclocitral (fruity, green, minty odor), and phenylacetaldehyde (honey like, sweet, green) contributing to melon aroma. Benzaldehyde was abundant in Western Shipper on day 0 ($62.53 \pm 9.61 \mu\text{g kg}^{-1}$) and increased on day 20 ($692.85 \pm 346.29 \mu\text{g kg}^{-1}$) while honeydew varieties had low concentrations (Fig 8). The C₉ aldehyde compounds (*Z*)-6-nonenal, (*E*)-2-nonenal, and (*E,Z*)-2,6-nonadienal and aromatic aldehydes benzaldehyde and phenylacetaldehyde were detected in high concentrations as the storage period continued, which indicates the development of off-flavors [22]. In honeydew, the C₉-aldehydes were high at day 0 as compared to cantaloupe and decreased as the storage period continued. The predominant aromatic ketone β -ionone (floral, rose, and woody notes), geranylacetone (floral fruity tropical), and farnesyl acetone were detected in all varieties. The total ketones were higher in cantaloupe varieties where β -ionone and geranylacetone were the most abundant. Geranylacetone and β -ionone inhibit microbial growth in the fruit and have characteristic aromas [76].

Sulphur compounds such as dimethyl trisulfide, ethyl 2-(methylthio) acetate, ethyl 3-(methylthio) propionate and ethyl 3-(methylthio)-(*E*)-2-propenoate were detected in all the varieties except HD150. The sulfur compounds were detected from day 10 to day 20 wherein the cantaloupes had high concentrations that contribute to the musky notes in cantaloupe aroma [79]. The aroma in melons is associated with ripening of the melon fruits [44, 75]. The changes observed in melon varieties are associated with factors such as the genetic makeup of the fruit, its maturity, environmental conditions during

production, postharvest handling, and storage [80]. The results obtained are in agreement with previously published studies showing that the aroma of melon varieties is influenced by storage and changes in volatile concentrations represent the influence of various factors such as ripening and the initiation of different metabolic pathways during postharvest storage.

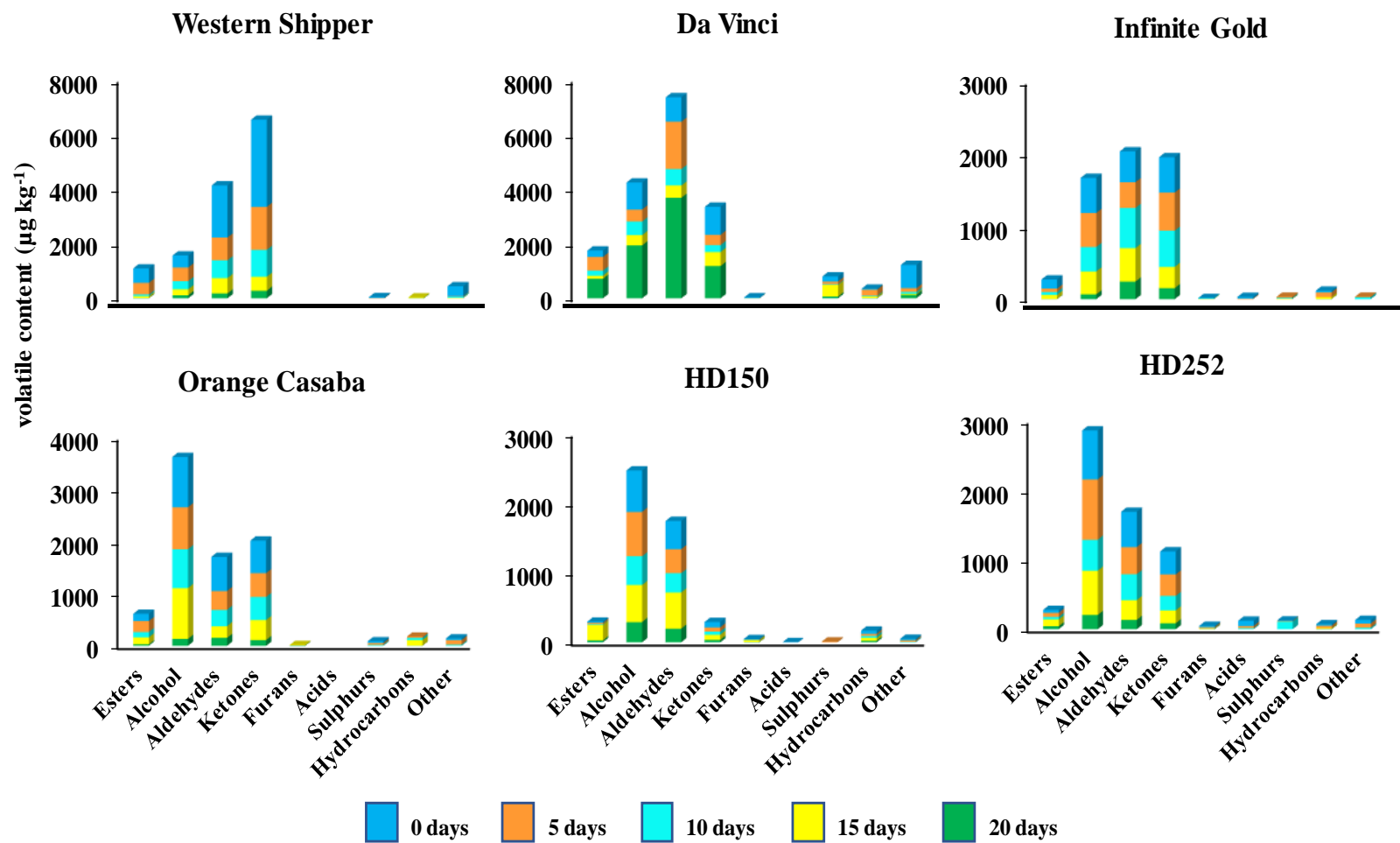


Figure 8. Volatile organic compounds (VOCs) in six melon varieties detected by HS-SPME-GCMS. Total of each class of compounds represented in each variety observed during storage

CHAPTER IV

OPTIMIZATION OF EXTRACTION SOLVENTS, FAST BLUE ASSAY FOR COMPARATIVE ANALYSIS OF ANTIOXIDANT PHENOLICS FROM *CUCUMIS MELO* AND THEIR IDENTIFICATION BY LC-HR-QTOF-MS

Introduction

The efficiency of extraction is affected by the extraction method, the sample matrix, and the acidity and polarity of the solvent [81]. Solid-to-liquid extraction is commonly used to recover natural antioxidants from plant materials using methanol, 80% methanol, or ethanol [4, 82]. For example, an analysis of phenolic compounds and antioxidant activities in melon fruits was carried out using methanol [4] and another study used 80% methanol to determine the free and bound phenolics in melon fruit pulp [82]. It was reported that dry melon peel extracts obtained from aqueous methanol show high total phenolic contents (TPC) compared to the aqueous ethanol extract [83]. A study evaluating the extraction efficiency of solvents was previously carried out using five solvent combinations (acetone, n-butanol, 80% ethanol, methanol, and deionized water) and showed that the 80% ethanol extract had the highest TPC and antioxidant activity in bitter melon (*Momordica charantia*) fruits [84].

The traditional method to measure TPC in natural products is the Folin-Ciocalteu (F-C) assay, but the F-C reagents interact and interfere with certain non-phenolic substances such as sugars, aromatic amines, sulphur dioxide, ascorbic acid, or organic

acids, which drastically affect the results. To counter this effect, several authors have proposed a single solid-phase extraction cleanup procedure. However, this cleanup is costly and time-consuming. The Fast Blue assay is based on the direct reaction of Fast Blue diazonium salts with the active hydroxyl groups present in the phenolic compounds. This method seems to be unaffected by the presence of sugars, organic acids, and ascorbic acid in samples, especially fruits and beverages that are high in vitamin C. The diazonium complex formation occurs in an alkaline medium in the presence of base and the reaction is based on the position of active -OH groups [85, 86].

Like TPC, there are multiple assays to measure antioxidant capacity, based on different principles of measuring radical scavenging. Antioxidant scavenging activity measured with 2,2-diphenyl-1-picrylhydrazyl (DPPH) and 2,2'-azino-bis (3-ethylbenzothiazoline-6-sulfonic acid) (ABTS) assays gives a wide range of values in different cultivars when comparing the type of production system or the time frame [87]. The ABTS radical cation is scavenged by hydrophilic and hydrophobic antioxidants, whereas most hydrophobic compounds scavenge DPPH in the presence of organic solvents [88]. Therefore, these two assays test different aspects of the antioxidant capacity of samples. Studies have shown that the antioxidant activity varied directly proportional to the TPC of a melon variety [89]. Factors such as cultivar, production time, and the type of production system influence the antioxidant levels of melons [90]. The health benefits of melon consumption are attributed to the presence of vitamins, minerals, phenolic metabolites, flavonoids, and alkaloids [27]. However, the recovery of melon phenolic compounds by different solvent combinations has not been explored.

Therefore, this study aimed to observe the effects of different extraction solvents on total phenolics content and antioxidant activities. In addition, Fast Blue (FB) assay was also optimized, and the method was validated on different melon varieties and commercial juices. The present study also involved the identification of phenolic compounds using liquid chromatography coupled with high-resolution quadrupole time-of-flight mass spectrometry (LC-HR-QTOF-MS).

Materials And Methods

Chemicals

ACS and LCMS grade solvents were used for extraction and liquid chromatography (LC) analysis. Methanol, acetonitrile, acetone, ethanol, formic acid, phosphoric acid, sodium carbonate, potassium hydroxide 2,2-diphenyl-1-(2,4,6-trinitrophenyl) hydrazyl (DPPH), Folin-Ciocalteu (F-C) reagent, 2, 2'-azinobis (3-ethylbenzothiazoline-6-sulphonic acid) diammonium salt (ABTS), Fast Blue B salt, quercetin, gallic acid, naringin, naringenin, and chlorogenic acid were obtained from Sigma (St. Louis, MO).

Plant Material And Extraction Solvents

Melons used for extraction were obtained from the H-E-B supermarket (College Station, TX, USA). For this experiment, two melon fruits were used, and duplicate samples was used for each solvent combination (n x 2). Each melon was cleaned in running water for 1 minute, dried, cut into two halves, deseeded, peeled, chopped into

small cubes and then blended (Oster 6684 12-Speed Blender) to obtain melon juice. The melon juice samples were combined and used for the analysis. Each sample was taken into clean 50-mL centrifuge tubes (VWR), and 10 g of juice was mixed with 10 mL of solvent. We used 22 solvent combinations and designated each with the letter S and a number: S1, water; S2, methanol: water: formic acid (50:45:5); S3, methanol: water: formic acid (50:48:2); S4, methanol: water: formic acid (80:15:5); S5, methanol: water: formic acid (80:18:2); S6, methanol: water (50:50); S7, methanol: water (80:20); S8, 100% methanol; S9, ethanol: water: formic acid (50:45:5); S10, ethanol: water: formic acid (50:48:2); S11, ethanol: water: formic acid (80:15:5); S12, ethanol: water: formic acid (80:18:2); S13, ethanol: water (50:50); S14, ethanol: water (80:20); S15, 100% ethanol; S16, acetone: water: formic acid (50:45:5); S17, acetone: water: formic acid (50:48:2); S18, acetone: water: formic acid (80:15:5); S19, acetone: water: formic acid (80:18:2); S20, acetone: water (50:50); S21, acetone: water (80:20); and S22, 100% acetone. Samples were vortexed for 30 sec, homogenized for 1 min at 9000 rpm (1540 x g) and sonicated for 1 h (Cole-Parmer Ultrasonic cleaner 8893). They were then centrifuged (VINTAGE Beckman J2-21 208V 30A 341735 Refrigerated Centrifuge) at 12000 rpm (59728 x g). The supernatant was then filtered using Whatman filter paper No. 1. In order to ensure complete extraction, the residue was re-extracted twice using 10 mL of the respective solvents. The extracts were then pooled, collected in clean tubes and stored at -20°C. These extracts were further used to measure TPC and radical scavenging activity.

Antioxidant Assays

DPPH radical scavenging activity

Melon extracts of different varieties were measured for scavenging activity using 2,2-diphenyl-1-picrylhydrazyl (DPPH) free radical. Samples were pipetted into different wells of a 96-well microplate in triplicate and the absorbance was recorded at 515 nm using a microplate reader (Bio Tek Instruments, Winooski, VT). Results were expressed in μg of ascorbic acid equivalents (AAE) per gram of sample. The DPPH free radical scavenging activity was measured according to a published protocol [91] and results were expressed as μg of AAE per mL of juice sample.

ABTS radical scavenging activity

Extracts were also analyzed for radical scavenging using 2,2'-azino-bis-(3-ethylbenzothiazoline-6-sulphonic acid) (ABTS) reagent. The reagent was prepared by mixing 0.165 g of potassium persulfate and 0.193 g of ABTS dissolved with NanoPure water. After overnight incubation in the dark, a green colored solution was obtained. Fresh ascorbic acid standard (0.05 mg mL^{-1}) was prepared in 3% metaphosphoric acid (MPA) and used for the assay. The ABTS radical cation eliminating activity was determined according to the published method [91]. The reaction was initiated by adding 180 μL ABTS stock solution into the mixture and the absorbance was recorded at a wavelength of 734 nm. The results were expressed as μg of AAE per mL of sample.

Folin-Ciocalteu Method To Measure Total Phenolic Contents

The TPC in the different melon extracts was determined by the F-C assay, as previously described, with slight modifications [36]. For each assay, 40 μL of extract was used and the absorbance was monitored at 760 nm with a microplate reader (Bio Tek Instruments, Winooski, VT). TPC was expressed as mg kg^{-1} gallic acid equivalents (GAE).

Optimization Of The Microplate Assay Using Fast Blue B Salt

The FB assay was previously developed for use with a spectrophotometer; in this study, the assay was carried out on a 96-well plate containing samples and a standard reference to measure TPC. This allowed 12 samples to be analyzed at the same time to reduce the amount of reagents and time required to analyze samples. To carry out this experiment, a protocol was developed based on modifications of previously published studies. NanoPure water was used as a dilution medium and the Fast Blue B salt (FBBS) solution was prepared daily by dissolving 10 mg FBBS in 100 mL NanoPure water to obtain a 0.01% solution. The optimization included selection of base, base concentration, standards, incubation time, volume of sample, and addition of water.

Optimization of base type and concentration

For the microplate analysis, solutions of bases at different normality were prepared using Nano Pure water. Three concentrations (0.5, 1, and 2 N) of NaOH, KOH, Na_2CO_3 , and K_2CO_3 were evaluated using $250 \mu\text{g mL}^{-1}$ of gallic acid standard. The resulting color was measured spectrophotometrically at 420 nm. Triplicate readings were taken, and

absorbance was measured at 0, 30, 60, and 90 min. The regression equations at each time period were compared to understand the effect of base at each time point.

Evaluation Of Standards Using The Optimized Fast Blue Assay

For the evaluation of reaction times for the formation of the azo complexes, different standards commonly found in fruits and vegetables were evaluated. Six standards (quercetin, chlorogenic acid, caffeic acid, catechin hydrate, naringenin, and gallic acid) were prepared at 250 ppm and used to determine the linearity of the standards at different incubation periods. Absorbance was measured at 0, 30, 60, and 90 min. The regression equation at each time point was compared to understand the sensitivity of standards.

Application Of The Optimized Fast Blue Assay To Measure TPC In Melon

To test the optimized method, another set of melon fruit samples was used for this experiment. Six melon varieties, Tarasco, T-Rex, Accolade, Mamut, Saurio, and Sweet Spring (seeds obtained from Syngenta seed company and grown and harvested in Uvalde, Texas). Based on the results obtained from TPC measured in melon using different solvents by the F-C and optimized FB methods showed that water gave the highest extraction efficiency and contained more phenolics than other solvent combinations. For sample extraction, fruits were cleaned, peeled, deseeded, then flesh was cut into cubes and blended to obtain melon juice. Melon samples (10 g) were extracted using NanoPure water and the residue was re-extracted to ensure complete extraction. The supernatant was

pooled and filtered using Whatman filter paper 1, and the collected extracts were stored at -20°C for further use. To perform the Fast Blue assay, 40 µL of extracts were plated into a clean 96-well microplate, followed by adding NanoPure water (160 µL), then 20 µL of freshly prepared FB solution (0.01%). After 10 min of incubation, 20 µL of 1 N KOH was added to the sample mixture in all wells. The plate was incubated for 120 min after which a 1 min reading measured the absorbance at 420 nm. The F-C assay according to previously published protocol [36] with slight modifications was carried out using melon extracts samples to compare results.

Application Of The Optimized Fast Blue Assay To Assess Total Phenolics In

Commercial Juices

The optimized assay was used to measure TPC in commercial juice samples to check the reproducibility of the assay. Pure juices of different brands available commercially in the H-E-B supermarket, College Station, Texas were purchased and extracted using methanol (100%) in the ratio of 2:1 methanol: juice according to a published method [92]. Juice samples and reagent concentration were adjusted according to the activity requirement. For example, white grape juices (J1 and J2) (Table 1) had lower TPC compared to the pomegranate juices (J15 and J16). Therefore, for FB assay of J1–J12, 40 µL of sample extract was used, followed by 160 µL of NanoPure water and FB solution (20 µL). Samples were incubated for 10 min and later 20 µL of 1 N KOH was added to the reaction mixture. For juices J13–J16, 10 µL of sample extract was used followed by 190 µL NanoPure water, FB solution (20 µL) and, after a 10 min incubation,

20 μL of 1 N KOH solution was added. The F-C assay was also performed on juice samples, the according to a published protocol [36].

Identification Of Phenolic Compounds By Liquid Chromatography/Electrospray Ionization High-Resolution Quadrupole Time-Of-flight Tandem Mass Spectrometry (LC/ESI-HR-QTOF-MS)

To identify phenolic compounds, melon samples were lyophilized to melon powder (0.250 g) and then extracted with 5 mL of various solvent combinations by vortexing for 1 min, homogenization for 1 min, sonication for 1 h and centrifugation (Beckman Model TJ-6) at $4480 \times g$ for 15 min. The sample extracts were analyzed for identification of phenolics using Agilent 1290 liquid chromatography (Santa Clara, CA, USA) equipped with a diode array detector and coupled to a maXis Impact high-resolution mass spectrometer (Bruker Daltonics, Billerica, MA, USA). The chromatographic separation was carried out on a Zorbax Eclipse Plus C18 column (100 mm \times 2.1 mm, 1.8 μm) at a flow rate of $100 \mu\text{L min}^{-1}$. The mobile phase was composed of (A) 0.1% formic acid in water and (B) 0.1% formic acid in acetonitrile. The solvent gradient was 80–20% B (0 min), 50–50% B (2 min), 38–62% B (3 min), 32–68% B (7 min), 28–72% B (3 min), 10–90% B (12 min), 80–20% B (14 min) and 80–20% B (16 min). Mass spectral analyses were performed in positive ionization mode according to our previous methodology [93, 94].

Statistical Analysis

For this study, two melon fruits were used, and pooled together to form one sample. Each solvent extract from the melon samples was prepared in duplicate and the results are given as (means \pm SE). For analysis of commercial juices, 16 commercial juice brands were selected and duplicate samples from each were used for the experiment. Analysis of variance and significant differences among means were tested by one-way ANOVA and Student's *t*-test was used to compare the means using JMP (version 14.0 for Windows SAS Inc., Cary, NC 27513).

Results And Discussion

Effect Of Extraction Solvent On Free Radical Scavenging Activities

The nature of the extraction solvent determines the amount of antioxidant activity measured for the plant materials. The antioxidant compounds present in the plant matrix may or may not be extracted with one particular solvent due to their varied chemical characteristics and polarities [95]. Therefore, the selection of a suitable solvent plays an important role in the maximum extraction of bioactive compounds and determining their free radical scavenging activities.

DPPH radical scavenging activity

The DPPH is scavenged by hydrophobic compounds in the presence of organic solvents and this causes a color change from purple to light yellow [88]. The results of DPPH scavenging activities of extracts from melon samples made with different solvents

are shown in Fig. 1 (A). In our study, samples extracted using methanol and combinations with water and acid showed higher DPPH activity compared to other solvent combinations. Solvent S8 (100% methanol) demonstrated the highest DPPH activity ($39.48 \pm 0.36 \text{ mg kg}^{-1}$) followed by S7 (methanol: water, 80:20) ($38.99 \pm 0.44 \text{ mg kg}^{-1}$) while samples extracted with acid combination S3 (methanol: water: formic acid, 50:48:2) showed higher activity ($33.46 \pm 0.92 \text{ mg kg}^{-1}$) than other methanol: water: acid combinations.

Samples extracted with ethanol showed lower scavenging activity than methanol combinations. Among the ethanol combinations, S15 (100% ethanol) demonstrated the highest scavenging activity ($37.37 \pm 1.78 \text{ mg kg}^{-1}$) followed by S11 ($29.01 \pm 0.30 \text{ mg kg}^{-1}$) and S14 ($29.27 \pm 1.50 \text{ mg kg}^{-1}$). Samples extracted with acetone showed the least DPPH scavenging activity. S16 (acetone: water: formic acid, 50:45:5) demonstrated higher activity of ($24.86 \pm 1.29 \text{ mg kg}^{-1}$) than the other acetone combinations. The 100% methanol extracts demonstrated the highest activity from the results obtained, followed by 100% ethanol extracts and then 100% acetone extracts. The water extract ($4.29 \pm 1.14 \text{ mg kg}^{-1}$) showed the lowest scavenging activity and was significantly different from that of the others ($p < 0.05$).

ABTS radical scavenging activity

The ABTS assay involves the generation of ABTS chromophores by oxidation of ABTS with potassium persulfate; antioxidant compounds prevent the generation of these chromophores. Unlike the DPPH assay, the ABTS assay detects both hydrophilic and lipophilic (hydrophobic) compounds [96]. Our results showed that scavenging activities

were higher when evaluated using ABTS assay than with the DPPH assay (Fig 1 A&B); this may be due to the type of reaction mechanisms, i.e. hydrogen atom transfer for the ABTS assay, and single-electron transfer for the DPPH assay [40]. Moreover, factors like stereo selectivity of radicals or solubility of extracts in different systems affect the extracts' capacity to react and quench the different radicals [97, 98] .

The results of ABTS assay (Fig. 1B) showed that extracts of 100% methanol S8 had the highest activity ($315.11 \pm 10.48 \text{ mg kg}^{-1}$) followed by methanol-water combination S7 ($297.39 \pm 14.98 \text{ mg kg}^{-1}$). Among the ethanol combinations, samples extracted using S14 (ethanol: water, 80:20) showed the highest activity ($276.61 \pm 13.62 \text{ mg kg}^{-1}$) followed by samples extracted using 100% ethanol S15 ($254.58 \pm 8.83 \text{ mg kg}^{-1}$). Acetone combination S21 ($275.83 \pm 18.81 \text{ mg kg}^{-1}$) showed the higher activity compared to other acetone combinations. Results of the ABTS assays demonstrated that the extracts obtained from solvents combined with water in the ratio of 80:20 (S7, S14, and S21) showed greater antioxidant activity. This may be due to change in solvent polarity on mixing with water. As a very polar solvent, water was observed to influence the extraction of polar phenolic compounds with high radical scavenging activity.

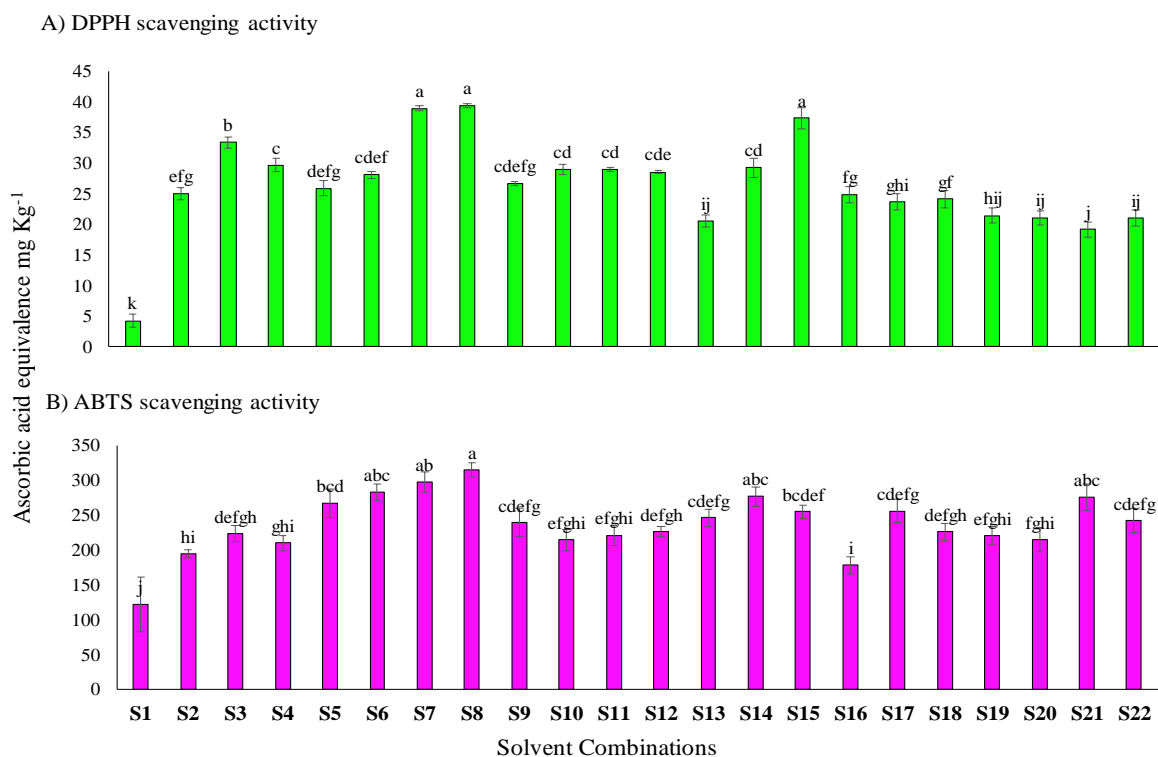


Figure 9. Free radical scavenging activity of cantaloupe extracts measured by A) DPPH assay (2,2-diphenyl-1-picrylhydrazyl) and B) ABTS assay (2,2'-azino-bis(3-ethylbenzothiazoline-6-sulfonic acid)). Solvent combinations used S1-water, S2-Methanol:water:formic acid (50:45:5), S3-Methanol:water:formic acid (50:48:2), S4-Methanol:water:formic acid (80:15:5), S5-Methanol:water:formic acid (80:18:2), S6-Methanol:water (50:50),S7-Methanol:water (80:20),S8-Methanol 100%, S9-ethanol:water: formic acid (50:45:5), S10-Ethanol:water:formic acid (50:48:2), S11-Ethanol:water:formic acid (80:15:5), S12-Ethanol:water:formic acid (80:18:2), S13-Ethanol:water (50:50), S14-Ethanol: water (80:20), S15-Ethanol 100%, S16-Acetone:water:formic acid (50:45:5), S17-Acetone:water:formic acid (50:48:2), S18-Acetone:water:formic acid (80:15:5), S19-Acetone:water:formic acid (80:18:2), S20-Acetone:water (50:50), S21- Acetone: water (80:20) , S22-Acetone 100%. For each solvent, different letters indicate significant differences ($P \leq 0.05$) among the solvent combinations

Optimized Fast Blue B Assay

The assay mixtures consisted of water (dilution medium), sample extracts, 20 μ L of FBBB salt solution (0.01%), and 20 μ L of KOH (1 N). For the standard reference, 250

ppm gallic acid was used to calculate regression at a reaction time of 120 min for each plate containing 12 samples. The quantity of the sample needs to be modified according to the presence of phenolic compounds. For analysis of melon extracts, 40 μL was used and for commercial juices such as pomegranate and prune juice, 10 μL was used as these juices are expected to be rich in phenolic compounds. Previous studies assessed beverage and juice samples by preparing the FBBB mixtures in borosilicate tubes and transferring the mixture to a microplate; the optimized assay described here does not require preparation in separate tubes. The optimization of the assay reduced the time required to analyze samples and reduced the steps required to carry out TPC analysis in fruit and beverage samples.

Optimization of base concentrations

The optimization parameters included selection of concentration of base and incubation time. For this study, bases (NaOH , KOH , Na_2CO_3 and K_2CO_3) at different concentrations (0.5, 1, and 2 N) were evaluated using 250 μg gallic acid as a standard. The results (Table 1 shows average regression of four replications) demonstrated that among the four bases evaluated, KOH at 0.5 N and 1 N showed faster reactions after 60 min of incubation. In the case of KOH (1 N), we observed a consistent regression ($r^2= 0.99$) when a reaction time of 120 min showed a linear response in all four replications. Interestingly, higher base concentrations did not favor the reactions and reaction time was not below 60 min. A previously published study compared 5% NaOH and 20% Na_2CO_3 , and showed that 5% NaOH had a faster completion of reaction compared to 20% Na_2CO_3 [99].

The use of lower amounts of chemical to prepare the base solution was preferred. Therefore, for the optimized assay 20 μ L of 1 N KOH was selected as base for a 120-min incubation. Spectrometric readings at 420 nm with a FBBB concentration of 0.1% prepared using NanoPure water were adopted for the analysis.

Table 6. Optimization of base concentrations for the Fast Blue assay. Results represented as average of regression equations obtained from four plate readings measured at 0, 30, 60, 90, and 120 minutes incubation

Base	Concentration (N)	0 min	30 min	60 min	90 min	120 min
NaOH	0.5	0.83	0.95	0.97	0.98	0.99
	1	0.85	0.96	0.98	0.99	0.99
	2	0.78	0.9	0.9	0.94	0.96
KOH	0.5	0.82	0.97	0.99	0.98	0.99
	1	0.79	0.92	0.94	0.96	0.99
	2	0.8	0.8	0.92	0.90	0.90
Na₂CO₃	0.5	0.89	0.95	0.98	0.99	0.99
	1	0.85	0.96	0.98	0.99	0.99
	2	0.83	0.98	0.99	0.97	0.97
K₂CO₃	0.5	0.82	0.85	0.98	0.97	0.97
	1	0.66	0.83	0.93	0.95	0.97
	2	0.73	0.87	0.94	0.96	0.97

Evaluation Of Standards Using Fast Blue Assay

The evaluation of standards using the optimized assay are represented at each time period (Fig. 4). The quality of phenolic compound plays an important role in the coupling reaction of the FBBB salt to the -OH groups. Previously, the proposed interactions with chlorogenic acid, caffeic acid, and flavonoids in NaOH buffer showed the formation of the azo complex in active ortho and para positions [99]. From the proposed mechanisms, it can be understood that the higher number of available reactive -OH groups in the compound that are present in ortho and para positions may influence the reaction times (incubation). Our results demonstrated that the quercetin standard had a linear reaction at 30 min, followed by chlorogenic acid, naringenin, and catechin hydrate at 60 min, and lastly gallic acid at 90 min while caffeic acid showed poor linearity. However, in the presence of KOH (1N), quercetin showed in the highest linear regression due to the presence of more reactive -OH groups at the ortho and para positions. Quercetin may be used as a standard reference to test TPC in fruit samples expected to have higher levels of flavonoid compounds. The results indicate that the standard references may be selected based on the structure and quality of the phenolic compounds present in the samples.

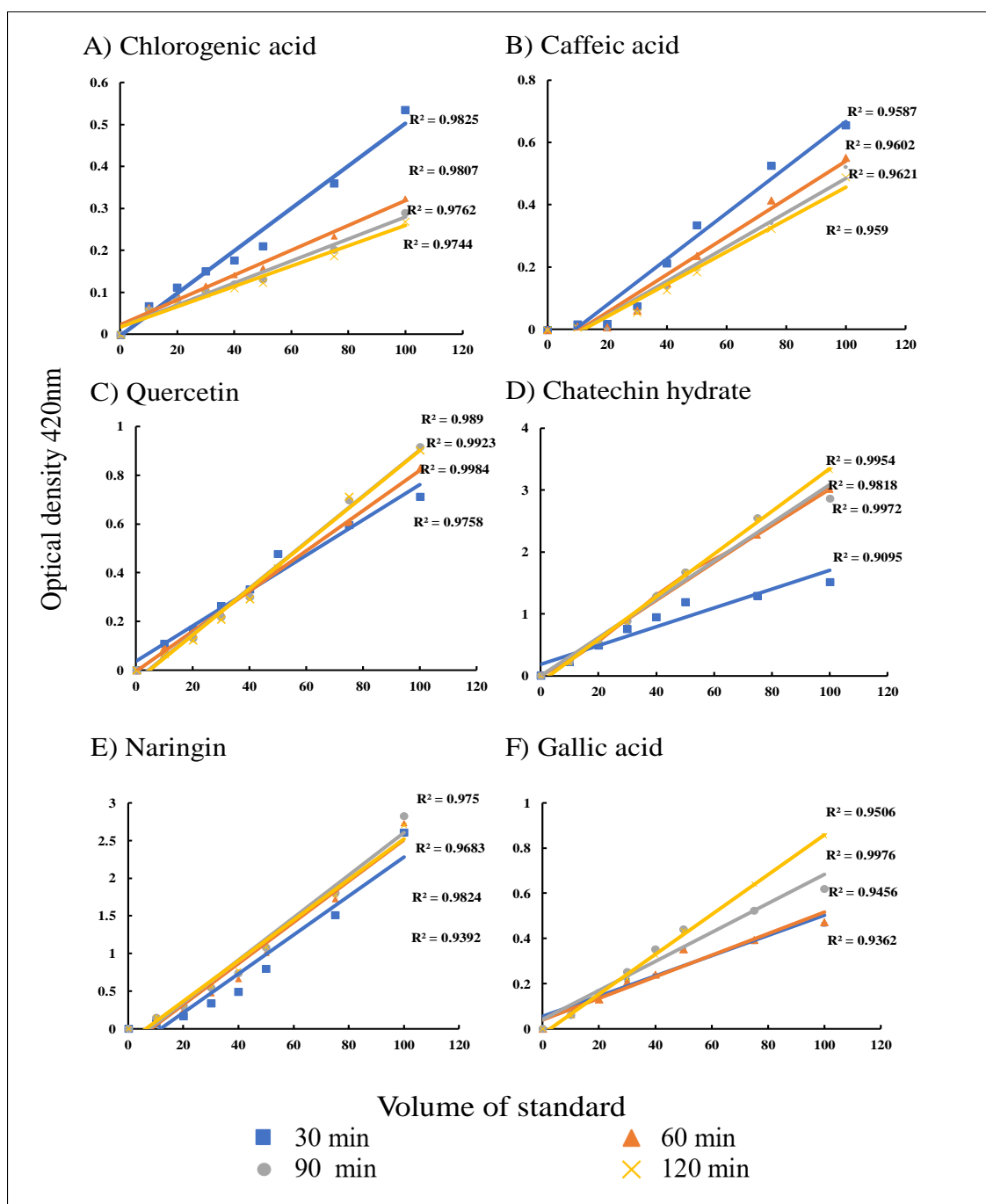


Figure 10. Reaction rate and incubation times evaluated using the optimized Fast Blue assay standards. A) Chlorogenic acid, B) caffeic acid, C) quercetin D) catechin hydrate, E) naringenin and F) gallic acid. Absorbance was read at 420 nm. All standards concentrations were $250 \mu\text{g mL}^{-1}$ for alkaline medium KOH (1N) used as base and 0.01% Fast Blue assay solution used for assay

Comparison Of Fast Blue B (FBBB) And Folin-Ciocalteu (F-C) Assays

The polarity of solvents affects the extraction of phenolic compounds which thereby influences the antioxidant activity of extracts. Our results showed that solvent combinations with higher polarity indices showed better activity compared to combinations with lower indices. To investigate the impact of the solvent type and the extraction efficiency of solvents, total phenolics content of melon samples extracted with the 22 solvent combinations were examined. The FB assay was previously developed to determine phenolic contents in food samples without interference of non-phenolic compounds [99, 100]. This assay uses the Fast Blue BB diazonium salt (FBBB) in which the diazonium group specifically couple with reactive phenolic hydroxyl (-OH) groups only in the presence of alkaline (base) to form stable azo complexes, which can be measured at 420 nm [101]. The azo-based assay (FBBB method) has higher GAE values than F-C for TPC. For example, when the samples with the addition of ascorbic acid and high-fructose corn syrup tested for TPC using the F-C assay reported under value of TPC compared to FBBB assay [99, 101]. To evaluate whether the previously designed spectrophotometric method can be scaled down to a microplate method, we optimized various factors to improve the sensitivity of the method and the use of less reagents.

The F-C method is simple and used commonly to measure TPC in extracts [102]. The assay measures all compounds readily oxidizable under the reaction conditions inclusive of monophenols and certain substances that are non-phenols or proteins. They also tend to react under these conditions [103], The F-C assay has for many years been used as a measure of TPC in natural products, and the basic mechanism is an oxidation-

reduction reaction. However, the reaction is slow at acid pH, and it lacks specificity [104]. The higher results obtained from the F-C assay in this study indicated the presence of non-phenolic compounds and higher values may be due to reaction with interfering substances present in the extract. Also, the acidity of the extracts evidently affected the reaction, as solvent combinations S4 and S16 recorded the least TPC probably due to acidity of the extracts which influenced the reaction. Extracts obtained from S1 also showed low TPC; this indicates that water extracts may contain impurities that affect the reaction and detection of phenolic compounds in the extract. However, combinations S8, S12, S15, and S20 recorded high TPC. Among all combinations used, 100% methanol and 100% ethanol combinations showed the highest recovery of phenolic compounds.

The TPC was measured using the F-C method and was compared with the gallic acid standard (mg kg^{-1}). The F-C method showed high TPC in samples obtained using ethanol extracts S15 ($139.49 \pm 4.77 \text{ mg kg}^{-1}$) and S12 ($138.72 \pm 10.72 \text{ mg kg}^{-1}$) followed by extracts obtained using methanol S8 ($137.99 \pm 8.07 \text{ mg kg}^{-1}$) and S6 ($134.14 \pm 7.10 \text{ mg kg}^{-1}$). Samples extracted using methanol-acid combinations, S2 and S4 showed in lower TPC, probably due to greater acidity of the solvent. Among acetone combinations, extracts S20 ($135.70 \pm 5.78 \text{ mg kg}^{-1}$) showed higher TPC compared to other acetone combinations.

To evaluate the accuracy of the conventional method and the optimized method, melon extracts were analyzed for TPC using both assays (Fig 2). The F-C assay showed higher values when compared with the FBAB assay, except for water extracts. Results of the FBAB assay demonstrated that extracts obtained using S1 ($94.82 \pm 18.39 \text{ mg kg}^{-1}$) showed the highest TPC, followed by extracts obtained using water and acid combination

(S17; $72.40 \pm 6.71 \text{ mg kg}^{-1}$). The recovery of phenolic compounds in the extracts showed that the highest TPC measured using FBBB assay was in S1 (water) and the lowest in S22 (acetone) among all the extracts ($P < 0.05$). Interestingly F-C assay results demonstrated that the extracts obtained using S15 ($139.49 \pm 4.77 \text{ mg kg}^{-1}$) and S8 ($137.99 \pm 8.02 \text{ mg kg}^{-1}$) showed higher TPC than other extracts, while S1 showed the lowest TPC.

The extracts analyzed with different solvents showed highest TPC in the order water > methanol > methanol + water > ethanol + water + acid > acetone + water + acid > acetone + water > acetone (Fig. 2). Previous studies also showed higher TPC in water extracts [105, 106]. However, higher extraction yield does not always indicate that the extract will have higher antioxidant activity, because the antioxidant activity also depends on the active antioxidant compounds present in the extract [106]. This agrees with our results, as water showed the highest TPC when measured using the Fast Blue assay but lowest anti-oxidant activity when measured using DPPH and ABTS antioxidant assays.

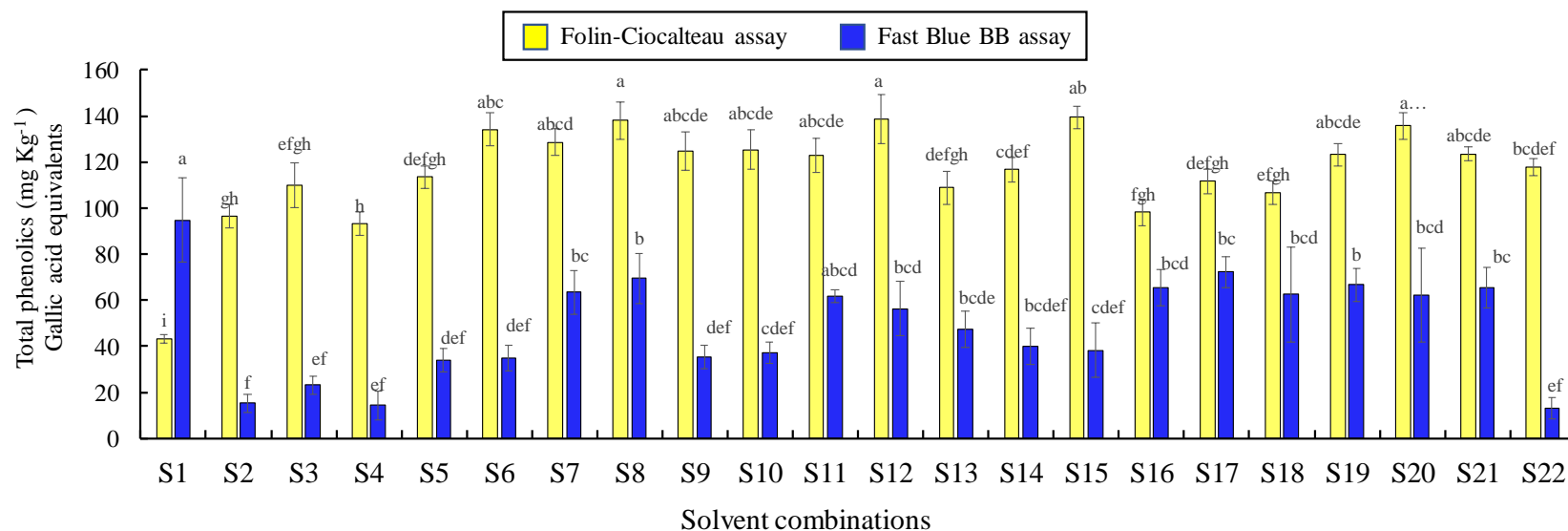


Figure 11. Total phenolic content measure by Folin-Ciocalteu and Fast Blue assays in cantaloupe melon extracts. Solvent combinations used S1-water, S2-Methanol:water:formic acid (50:45:5), S3-Methanol:water:formic acid (50:48:2), S4-Methanol:water:formic acid (80:15:5), S5-Methanol:water:formic acid (80:18:2), S6-Methanol:water (50:50), S7-Methanol:water (80:20), S8-Methanol 100%, S9-Ethanol:water:formic acid (50:45:5), S10-Ethanol:water:formic acid (50:48:2), S11-Ethanol:water:formic acid (80:15:5), S12-Ethanol:water:formic acid (80:18:2), S13-Ethanol:water (50:50), S14-Ethanol:water (80:20), S15-Ethanol 100%, S16-Acetone:water:formic acid (50:45:5), S17-Acetone:water:formic acid (50:48:2), S18-Acetone:water:formic acid (80:15:5), S19-Acetone:water:formic acid (80:18:2), S20-Acetone:water (50:50), S21- Acetone:water (80:20) , S22-Acetone 100%. For each solvent, different letters indicate significant differences ($P \leq 0.05$) among the solvent combinations

Application Of Optimized Fast Blue BB (FBBB) Assay On Melon Varieties

From the evaluation of 22 solvent combinations used to extract melon phenolics, it was observed that water extracted phenolics more efficiently than other combinations (Fig. 2) and therefore, water was used for the extraction of melon samples. To evaluate the TPC, six melon varieties (Syngenta) harvested from Uvalde, TX were examined using the optimized FBBB assay and compared with the F-C assay. The result showed that the F-C assay had higher TPC than the FBBB assay. Our results (Fig. 4) showed the Saurio variety had higher TPC measured by F-C assay (206.75 ± 8.79 mg kg⁻¹ GAE) compared to FBBB assay (196.88 ± 9.00 mg kg⁻¹ GAE), followed by Tarasco variety, which also showed higher TPC measured by the F-C assay (193.16 ± 4.21 mg kg⁻¹ GAE) than the FBBB assay (76.45 ± 4.06 mg kg⁻¹ GAE). These results indicate that the optimized assay can be applied to measure TPC in melon varieties accurately.

Some varieties showed comparatively lower TPC from the FBBB assay, which indicates that accuracy of the F-C method may be influenced by other compounds that interfere with detection of phenolic compounds (such as organic acids, sugars and ascorbic acid). An analysis of beverages and grains obtained similar results, showing higher F-C value results compared to FBBB [100].

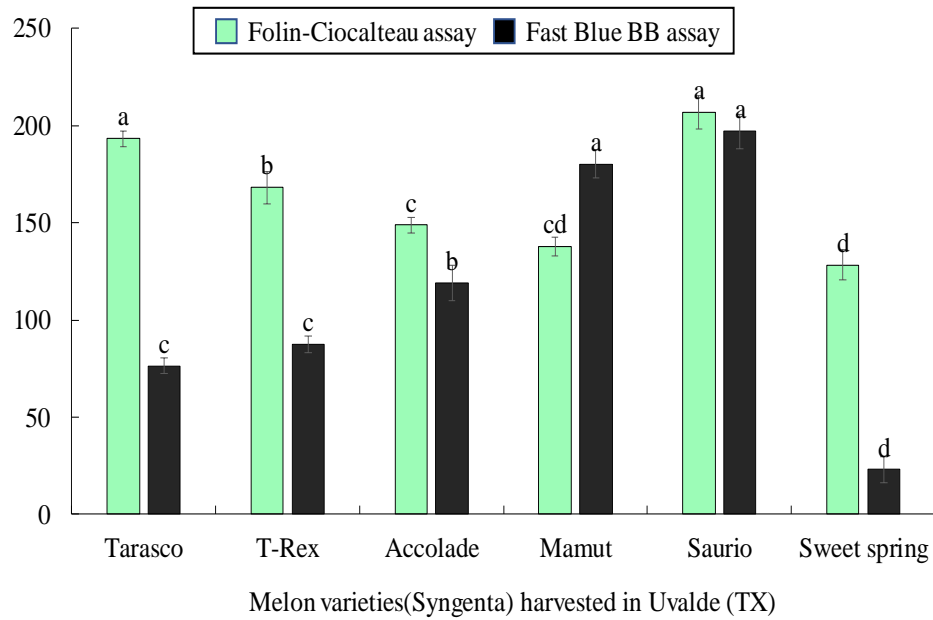


Figure 12. Application of optimized assay on melon samples (Syngenta) obtained from Uvalde, Texas. Total phenolic content measured using Fast Blue BB assay) and Folin-Ciocalteu (F-C) assay. Results are represented as mean±SE (mg kg⁻¹) gallic acid equivalents. For each variety, different letters indicate significant differences (P≤0.05) among the melon varieties

Application Of Optimized Fast Blue BB Assay (FBBB) On Commercial Juice Samples

Analysis of juices by the F-C assay and optimized FBBB assay showed that the TPC measured by FBBB method was higher compared to the values obtained from F-C method (Table 2). The pomegranate juices measured high TPC among all juices analyzed by FBBB and F-C methods. Results showed pomegranate juices J15 ($4564.08 \pm 114.9 \text{ mg kg}^{-1} \text{ GAE}$) and J16 ($5634.19 \pm 26.83 \text{ mg kg}^{-1} \text{ GAE}$) had higher TPC compared to the TPC measured by F-C method; J15 ($1649.39 \pm 40.88 \text{ mg kg}^{-1} \text{ GAE}$) and J16 ($1756.86 \pm 23.23 \text{ mg kg}^{-1} \text{ GAE}$). This suggested that there may be a high concentration of non-phenolic compounds in the sample extracts. Similarly, a study comparing the FBBB and F-C assays used fresh pomegranate extracts and showed higher TPC measured by FBBB method ($193 \text{ mg}/100 \text{ g GAE}$) compared to F-C method ($161 \text{ mg}/100 \text{ g GAE}$)[99]. This suggested that non-phenolic compounds and reducing sugars naturally present or added to juice mixes contribute to higher F-C values. The same study also showed that the FBBB method results were higher than F-C method, which agrees with our results.

Table 7. TPC of commercial juice extracts measured by the optimized Fast Blue (FBBB) assay and Folin Ciocalteu (F-C). Results represented as mg \pm SE total phenolics as Gallic acid equivalents (GAE). For each Juice, different letters indicate significant differences ($P \leq 0.05$) among the juices

Juice ID	Juice Samples	FBBB Assay (mg kg⁻¹ GAE)	F-C Assay (mg kg⁻¹ GAE)
J1	Welch's White Grape	301.18 \pm 8.33f ^g	347.01 \pm 9.24 ^{hi}
J2	H-E-B White Grape	41.42 \pm 1.88 ^h	127.31 \pm 12.39 ^k
J3	Old Orchard Apple Juice	93.56 \pm 7.49 ^{gh}	150.87 \pm 13.88 ^k
J4	Feeding America Apple Juice	321.7 \pm 12.81 ^f	379.82 \pm 18.7 ^{gh}
J5	H-E-B Pineapple Juice	135.31 \pm 9.3f ^{gh}	289.17 \pm 8.02 ^{ij}
J6	Central Market Pineapple Juice	267.77 \pm 28.62 ^{fgh}	298.07 \pm 19.13 ^{ij}
J7	Ocean Spray Grapefruit	676.84 \pm 30.29 ^e	287.5 \pm 17.23 ^{ij}
J8	H-E-B Grapefruit	950.11 \pm 10.41 ^{cd}	267.9 \pm 12.41 ^j
J9	H-E-B Organic Grape	738.28 \pm 22.13 ^{de}	567.89 \pm 15.34 ^f
J10	Central Market Concord Grape	1010.97 \pm 30.29 ^c	836.12 \pm 26.62 ^c
J11	Ocean Spray Cranberry	343.88 \pm 18.24 ^f	409.07 \pm 19.07 ^g
J12	Lakewood Organic Cranberry	594.3 \pm 19.93 ^e	435.57 \pm 11.54 ^g
J13	Sunsweet Amazin Prune Juice	716.92 \pm 11.44 ^e	798.65 \pm 20.32 ^d
J14	H-E-B 100% Prune Juice	730.77 \pm 13.64 ^e	659.77 \pm 21.1 ^e
J15	Langers Pomegranate Juice	4565.08 \pm 114.91 ^b	1649.39 \pm 40.88 ^b
J16	Central Market Pomegranate Juice	5634.19 \pm 26.83 ^a	1756.86 \pm 23.23 ^a

Identification Of Phenolic Compounds By LC/ESI-HR-QTOF-MS

The LC chromatograms of the methanolic extracts are shown in Fig 5(A). Previously, a study investigated the anti-inflammatory activity of melon phenolic compounds in melon peel and pulp and represented a metabolic profile of the various compounds detected in ethanol extracts using UPLC-DAD-MS/MS in negative ion mode [107]. Similarly, another study investigated three Spanish melon varieties and reported phenolic compounds, amino acid derivatives, and flavonoids in polar fractions acidified mobile phases A and B consisted of water (0.5% acetic acid, v/v) and acetonitrile were used. The gradient was programmed was : 0 min, 5% B;10 min,30% B; 12 min, 33% B; 16 min, 38% B; 19 min, 50% B; 22 min, 95% B;24 min 5% B, andfinally a conditioning cycle of 10 min with the initial conditions, flow rate was set at 0.80 ml/min throughout the gradient. In our study The identification of phenolics compounds in melon extracts was performed by LC-HR-ESI-QTOF-MS using positive ionization mode.Six phenolics derivatives were identified. The tandem mass spectra extracted ion chromatograms and mass spectra of identified compounds are presented in Fig 5 (B). A peak eluted at retention time (RT) 1.7 min showed an accurate mass spectrum at m/z 579.1906 $[M+H]^+$. The peak was identified as apigenin-7-O-rutinoside based on the mass spectra and the published literature [107]. Another peak that eluted at RT 3.7 min was identified as gentisic acid-hexoside isomer 2 (m/z 317.1207 $[M+H]^+$ reported previously in melon extracts obtained using methanol:water (80:20) [108]. Similarly, a peak eluted at 5.5 min at (RT) representing the molecular ion peak at m/z 611.1606 $[M+H]^+$ was recognized as rutin. Two peaks that eluted at RT 5.5 and 5.9 min were identified as isorhamnetin-3-O-glucoside and naringin,

having a molecular ion peak at m/z 479.1184 $[M+H]^+$ and m/z 581.1864 $[M+H]^+$, respectively. A peak at RT 7.4 min displayed molecular ion peak at m/z 273.0757 $[M+H]^+$ and was identified as naringenin and previously reported by Rodríguez-Pérez, C. et al., [107]. It has also been suggested that the chemical composition of melon and other fruits is strongly influenced by the physiological state of the plants and by the environmental parameters as well as by the genotype [108].

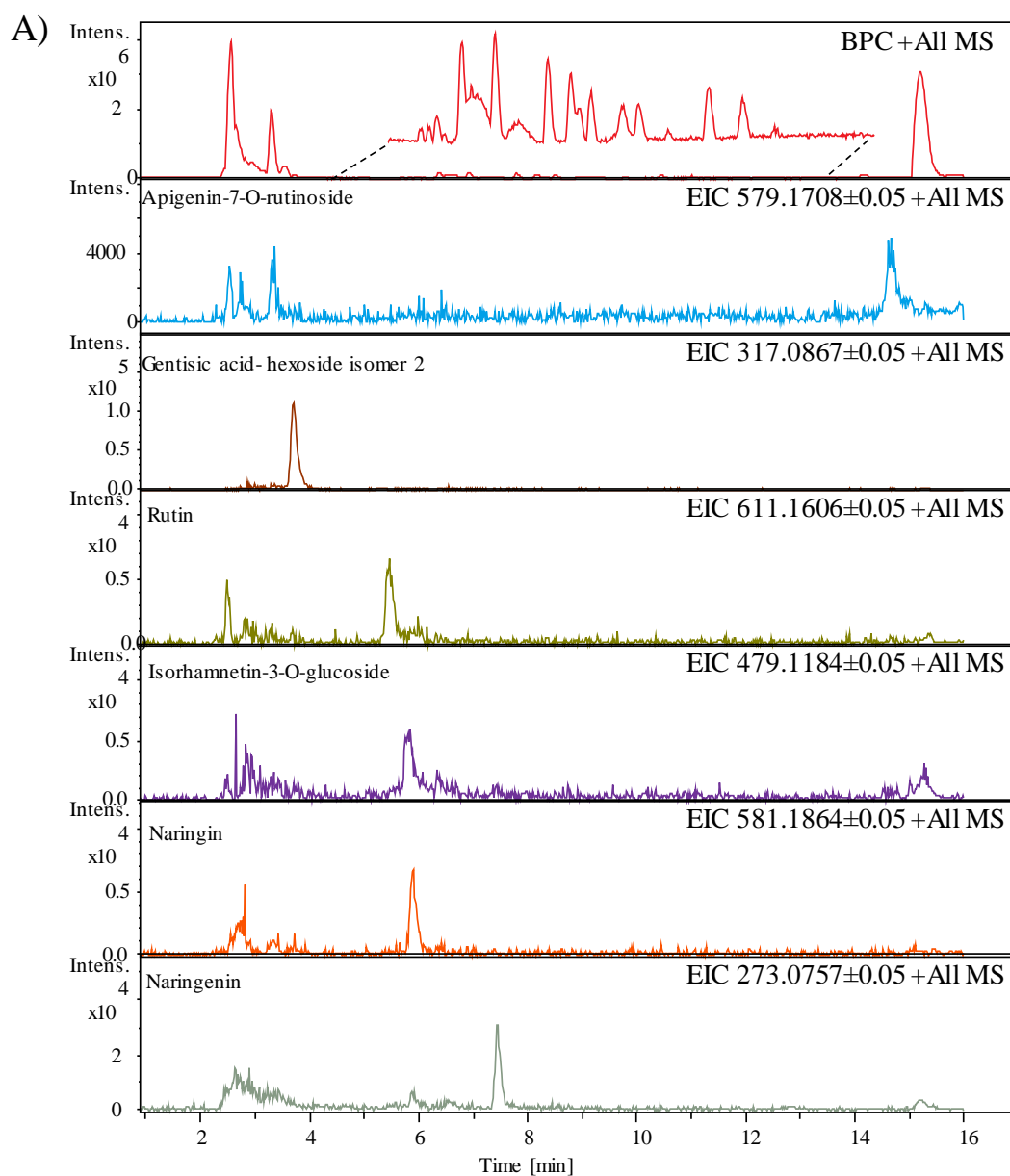
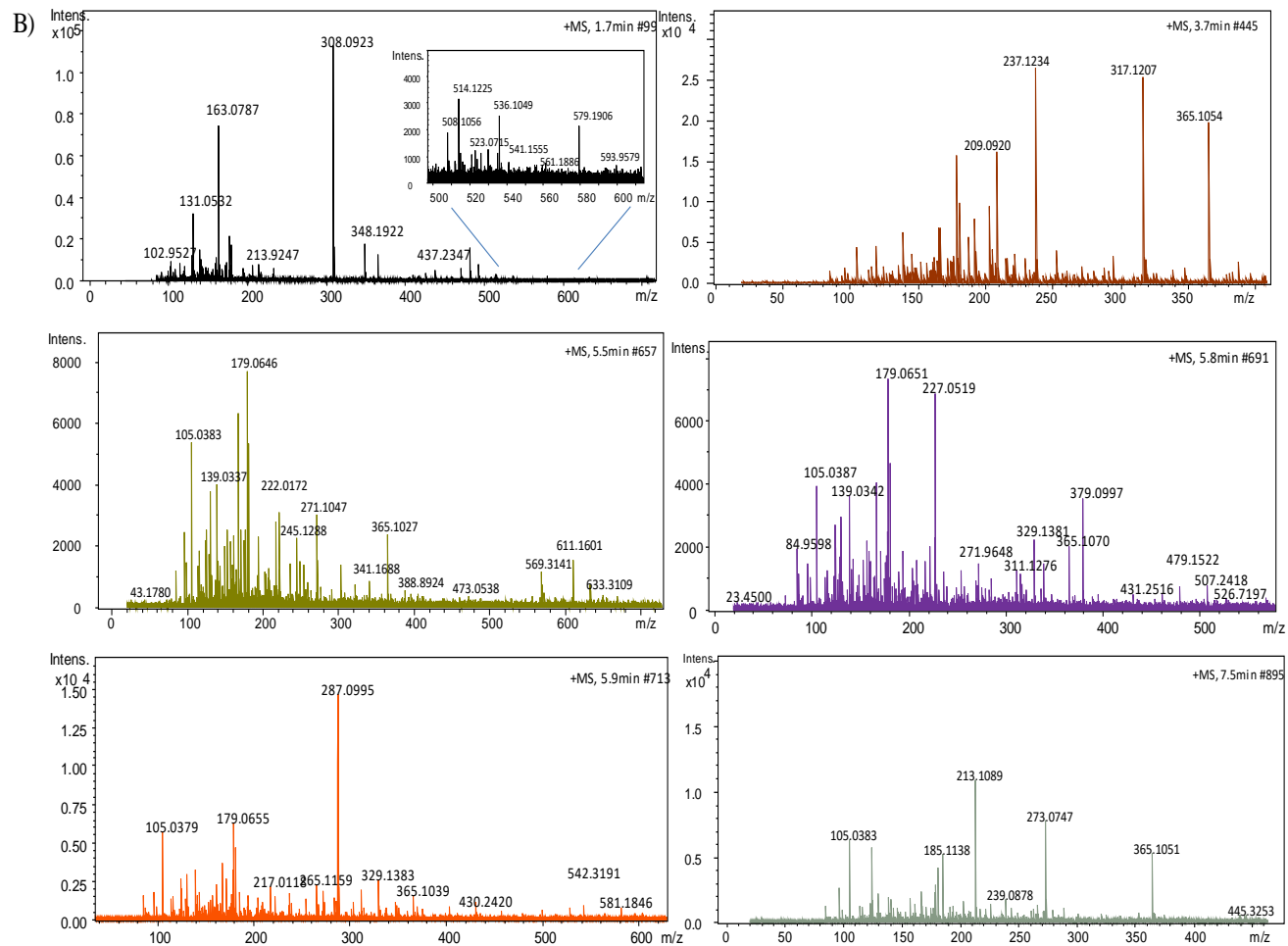


Figure 13. A) Total ion chromatogram and extracted ion chromatograms by LC-HR-ESI-QTOF-MS and B) tandem mass spectra of phenolic compounds identified from the methanol extracts of melon

Figure 13 Continued



CHAPTER V

CONCLUSION

A postharvest study of melon fruits stored for twenty days at low temperature showed interesting results and provided insights on the metabolic changes that occur during storage. The results show that bioactive compounds are actively involved in metabolic pathways: after the fruit is detached from the plant, metabolism continues and significantly affects carotenoids, ascorbic acid, and amino acids. Antioxidant activity decreased at the end of storage. The fluctuation in concentrations of bioactive compounds was observed to differ in each variety measured in this study, suggesting that the varietal differences play a significant role. Antioxidant activity measured on day 0 until day 20 showed a significant reduction in scavenging activity while changes in total phenolics content depicts minimal loss. Trend in carotenoids in melon shows that carotenoid compounds are converted to lower metabolites such as β -carotene used to form β -cryptoxanthin, ζ -carotene isomers were involved in the formation of lycopene isomers and α -carotene. Ascorbic acid (AA) and its oxidized form, dehydroascorbic acid (DHA), increased significantly when measured on day 5, but AA and DHA degradation occurred after 5 days, suggesting that AA and DHA have broken down to lower products and complete loss at the end of storage was observed. Amino acids measured in each variety showed different patterns during storage; their biosynthetic pathway likely initiated as a stress response. Ester, alcohol, aldehyde and ketone compounds were abundant and contributed to the pleasant aroma of melons. The total volatile compounds increased as

the storage period progressed indicating the ripeness of the melon. The C₉ aldehyde and C₉ alcohol compounds discriminated the cantaloupe and honeydew varieties and their increase in concentration at end of the storage indicated the development of off-flavors and senescence in fruit. Volatiles derived from carotenoids such as β -ionone, geranylacetone, and β -cyclocitral are important components of flavor and aroma. This study used whole melons without chemical treatment, which gives insight about metabolites in different varieties; however melons treated chemically may be suited for prolonged storage and changes in bioactive compounds can be understood.

Study of extraction efficiency showed that careful selection of solvents helps complete recovery of phenolic compounds. Ecofriendly solvents with lower toxicity such as water may be adopted and combinations with ethanol, methanol with water also showed good recovery and antioxidant properties in the extracts. Results obtained using FBBB assay showed negligible differences between successive measurements acquired over similar operating conditions from multiple samples. One drawback of the FBBB method is that some interactions with the phenolic standards persist, which results in differences in absorbance values. This area requires further study to get a clear distinction regarding the specificity of TPC determination. The structural and compositional diversity of natural products and the polarity of the solvents affect the samples and therefore, different possibly unpredictable behaviors are expected. With the optimized FBBB assay, we were able to analyze TPC accurately. Moreover, the assay was miniaturized to examine samples efficiently in a short time. Antioxidant compounds behave in varied manner *in vivo* through different mechanisms and there is no single method that can fully evaluate the

total antioxidant capacity of foods. In conclusion, the antioxidant activity was found to be influenced by the polarity of the melon extracts and the type of phenolic compounds present in the extracts. In the present work, LC-MS analytical technique was used for separating and detecting phenolic compounds in methanolic extracts of *Cucumis melo* L. With this method, six compounds were identified based on chromatographic separation, and MS/MS fragmentation. This work provided a better understanding of the efficiency of solvents to obtain phenolic compounds in melon samples and antioxidant properties of cantaloupe melon.

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APPENDIX

Table A-1. Amino acid contents of Western Shipper cantaloupe variety represented as mean \pm SE

Western Shipper					
Amino acid	0 Days	5 Days	10 Days	15 Days	20 Days
Arginine	117.64 \pm 4.58b	228.14 \pm 23.5b	146.8 \pm 16.06b	222.6 \pm 15.28b	174.89 \pm 6.27b
Asparagine	914.65 \pm 9.71a	810.96 \pm 66.25b	564.85 \pm 54.84c	641.31 \pm 44.65bc	475.66 \pm 72.27c
Glutamine	717.43 \pm 72.72b	1149.74 \pm 58.57a	725.28 \pm 32.31b	1226.13 \pm 37.22a	1359.16 \pm 182.34a
Citrulline	275.63 \pm 8.18ab	356.49 \pm 26.8a	263.09 \pm 38.54ab	278.61 \pm 41.25ab	234.48 \pm 4.96b
Serine	197.96 \pm 5.87ab	243.19 \pm 20.95a	155.47 \pm 5.13b	186.11 \pm 13.68b	187.85 \pm 4.48b
Aspartic acid	1074.22 \pm 170.13c	1781.14 \pm 99.02a	1026.65 \pm 27.91c	1588.67 \pm 242.15ab	1203.17 \pm 22.29bc
Hydroxy Proline	529.23 \pm 13.31b	383.01 \pm 35.4c	326.01 \pm 49.07c	336.31 \pm 15.05c	744.69 \pm 49.44a
Glycine	170.07 \pm 26.93b	281.99 \pm 15.67a	162.53 \pm 4.41b	251.51 \pm 38.33a	95.05 \pm 5.95b
Threonine	72.44 \pm 8.67a	44.43 \pm 4.55b	47.04 \pm 1.06b	54.69 \pm 2.34b	10.84 \pm 0.43c
Beta-alanine	124.32 \pm 5.64a	105.81 \pm 6.3a	52.42 \pm 8.05b	69.69 \pm 8.97b	78.74 \pm 2.91b
Alanine	23.25 \pm 0.86b	81.01 \pm 6.39b	96.46 \pm 2.8b	83.22 \pm 2.86b	549.95 \pm 57.98a
GABA	2330.66 \pm 81.61b	2693.77 \pm 77.78a	1298.85 \pm 102.42c	1375.92 \pm 75.51c	1507.3 \pm 100.76c
Proline	104.72 \pm 6.71a	112.56 \pm 6.14a	64.12 \pm 6.25b	15.71 \pm 1.91c	105.36 \pm 0.85a
Methionine	25.41 \pm 4c	84.99 \pm 5.42a	22.57 \pm 3.97c	52.08 \pm 3.71b	59.19 \pm 2.42b
Valine	613.6 \pm 18.15a	629.12 \pm 38.7a	411.4 \pm 13.36c	208.2 \pm 4.97d	517.77 \pm 13.59b
Tryptophan	49.28 \pm 0.83c	83.76 \pm 5.08ab	53.86 \pm 6.19c	107.13 \pm 12.68ab	58.34 \pm 1.56bc
Phenylalanine	161.61 \pm 3.47a	205.3 \pm 35.44a	168.77 \pm 21.03a	123.06 \pm 15.47a	170.4 \pm 16.26a
Isoleucine	21.77 \pm 0.68b	30.9 \pm 2.77b	16.69 \pm 0.26b	139.43 \pm 17.68a	30.26 \pm 1.42b
Leucine	25.05 \pm 0.93c	22.95 \pm 5.22bc	19.51 \pm 0.87d	33.02 \pm 1.42ab	36.24 \pm 1.58a

Table A-2. Amino acid contents of Infinite Gold cantaloupe variety represented as mean ± SE

Infinite Gold					
Amino acid	0 Days	5 Days	10 Days	15 Days	20 days
Arginine	108.3±8.54b	76.98±7.95b	94.29±12.12b	121.39±22.03b	171.61±4.08a
Asparagine	858.18±51.46a	677.76±63.41ab	554.55±90.05b	640.1±77.48ab	902.06±24.32a
Glutamine	1219.03±37.04a	1243.18±128.89a	647.98±112.01b	1125.74±90.56a	1103.03±48.1a
Citrulline	371.22±8.49a	281.45±12.28b	225.59±19.93bc	214.81±21.32c	273.27±11.69bc
Serine	144.07±4.86a	135.15±12.6a	183.34±27.52a	162.22±25.34a	187.64±4.76a
Aspartic acid	948.73±72.97a	1034.24±171.3a	1357.14±93.96a	1107.62±179.69a	1442.89±66.23a
Hydroxy Proline	380.6±12.16b	265.95±16.43b	287.91±13.14b	290.74±63.7b	687.27±47.79a
Glycine	150.2±11.55ab	167.09±28.23ab	214.86±14.87ab	171.21±26.61ab	105.07±3.8b
Threonine	81.33±5.19a	31.32±2.57b	35.94±4.77b	43.64±5.37b	11.2±0.64c
Beta-alanine	113.04±4.33a	37.02±6.19c	51.08±1.92bc	80.32±17.5ab	87.35±6.68ab
Alanine	51.94±2.09b	96.55±5.01b	114.26±4.24b	85.29±10.85b	695.43±34.65a
GABA	2660.83±151.16a	2464.07±136.6a	2279.68±46.97ab	2228.21±250.16ab	1793.63±67.79b
Proline	88.13±2.65bc	112.62±4.95ab	73.08±1.31c	32.35±10.44d	116.61±9.74a
Methionine	38.9±3.05b	46.24±4.29b	42.71±1.95b	67.72±9.4a	49.01±0.72ab
Valine	414.9±16.63ab	337.47±41.8b	381.84±11.36b	166.2±22.83c	505.05±13.46a
Tryptophan	49.54±1.69b	58.55±4.85ab	44.93±0.65b	80.87±12.04a	82.52±4.46a
Phenylalanine	109.14±4.03b	110.35±26.73b	121.72±6.15b	103.98±10.68b	210.62±8.3a
Isoleucine	10.3±0.74b	7.69±0.97b	12.32±0.54b	72.72±15.87a	21.22±0.31b
Leucine	11.37±1.09c	9.35±1.44c	14.07±0.92c	20.31±1.81b	27.63±0.63a

Table A-3. Amino acid contents of Da Vinci cantaloupe variety represented as mean ± SE

Da Vinci					
Amino acid	0 Days	5 Days	10 Days	15 Days	20 days
Arginine	94.94±6.36a	96.82±9.67a	101.43±4.32a	107.5±4.12a	109.89±3.17a
Asparagine	787.15±34.42a	793.94±63.79a	672.83±44.18a	732.83±37.33a	614.38±11.67a
Glutamine	933.45±22.47ab	1059.86±43.39a	885.83±35.28b	699.99±10.79c	900.15±152.63ab
Citrulline	173.12±7.3a	182.13±16.46a	161.15±5.6a	186.56±6.44a	169.76±23.74a
Serine	155.42±4.77ab	157.86±8.6b	163.01±4.25ab	192.82±10.7a	176.89±4.64ab
Aspartic acid	1092.84±105.79c	2054.39±172.14ab	1499.71±62.93bc	2315.52±217.21a	2402.02±176.63a
Hydroxy Proline	476.86±40.73a	294.59±21.34b	345.85±9.64b	368.61±14.49b	550.3±23.57a
Glycine	173.02±16.74c	321.81±26.23ab	237.43±9.96bc	391.38±30.59a	86.99±2.32d
Threonine	108.75±9.05a	26.61±2.3bc	34.87±0.63b	41.01±1.57b	11.68±1.34c
Beta-alanine	105.02±4.01b	40.5±3.36d	62.51±0.7c	100.26±3.58b	152.27±8.85a
Alanine	27.57±1.61c	82.22±10.07b	90.89±2.35b	82.72±0.89b	775.56±73.41a
GABA	2985.04±79.17a	2734.19±124.51b	2194.5±36.63c	2487.18±122.1bc	2426.89±102.57bc
Proline	85.84±7.41b	94.74±6.04b	82.66±1.2b	16.44±0.51c	135.98±9.42a
Methionine	40.53±3.51bc	33.36±2.84bc	33.06±1.51c	82.75±2.25a	48.36±5.16b
Valine	510.83±22.48a	375.67±20.45b	378.73±7.6b	126.6±10.31c	578.57±30.33a
Tryptophan	56.75±5.87b	58.62±4.81b	47.42±4.7b	99.26±6.27a	66.65±1.94b
Phenylalanine	208.08±14.69a	189.32±14.39ab	145.39±14.53b	106.13±9.49c	211.13±5.97a
Isoleucine	16.97±2.11bc	15.89±1.18c	18.32±0.59bc	131.31±6.77a	32.46±1.51b
Leucine	18.01±2.07b	21.21±1.85b	23.09±0.81b	26.87±2.27b	45.7±1.9a

Table A-4. Amino acid contents of Orange Casaba honeydew variety represented as mean ± SE

Orange Casaba					
Amino acid	0 Days	5 Days	10 Days	15 Days	20 days
Arginine	87.2±3.26ab	77.43±6.08b	47.15±19.04b	97.35±7.43ab	129.12±2.34a
Asparagine	568.85±20.54a	609.77±31.1a	429.47±86.85a	654.49±51.78a	656.26±9.5a
Glutamine	1517.1±59.83b	2092.82±194.51a	1573.42±24.95b	1859.96±127.96ab	1921.87±100.82ab
Citrulline	150.92±6.44c	183.33±12.14bc	137.98±3.47c	224.9±25.96b	335.8±30.38a
Serine	131.3±6.34a	151.47±4.25a	125.2±10.36a	157.59±12.2a	153.06±9.21a
Aspartic acid	717.91±154.36c	1898.62±136.43ab	1178.29±97.18c	2563.02±201.19a	1192.08±36.68bc
Hydroxy Proline	448.39±21.69b	254.72±17.75c	237.98±10.38c	373.28±56.66bc	626.16±29.11a
Glycine	113.66±24.43c	296.81±23.71ab	186.54±15.38bc	430.26±33.6a	75.81±1.96bc
Threonine	62.77±4.67a	29.89±2.14b	31.83±3.99b	27.57±2.06b	20.54±2.13b
Beta-alanine	75.32±6.05b	75.66±4.7b	40.08±0.88c	55.43±6.27c	108.43±4.53a
Alanine	50.51±3.87bc	31.92±6.88c	86.25±2.05b	63.26±5.6bc	282.07±29.42a
GABA	2245.14±218.68a	2311.1±114.21a	1751.52±55.67a	1744.59±177.92a	1812.6±83.37a
Proline	48.39±4.56b	41.83±7b	35.98±2.01b	73.7±6.92a	79.42±2.26a
Methionine	46.28±1.11b	67.21±6.86a	42.23±2.06b	53.51±3.46ab	75.23±6.9a
Valine	336.02±27.77a	326.45±38.16a	322.13±11.8a	385.95±29.95a	457.83±31.5a
Tryptophan	50.32±2.61bc	68.73±5.92ab	42.95±1.95c	66.38±7.01ab	78.11±4.15a
Phenylalanine	110.32±2.5b	99.64±10.16bc	74.63±5.47c	135.34±11.54ab	171.42±8.91a
Isoleucine	22.81±0.81a	34.48±4.28a	22.96±0.4a	39.64±9.2a	34.85±2.25a
Leucine	25.31±0.76b	28.21±1.48b	23.79±1.07b	35.62±2.63a	40.02±0.99a

Table A-5. Amino acid contents of honeydew variety HD150 represented as mean \pm SE

HD150					
Amino acid	0 Days	5 Days	10 Days	15 Days	20 days
Asparagine	557.42 \pm 34.31a	533.11 \pm 10.58a	509.28 \pm 135.51a	708.81 \pm 47.6a	638.89 \pm 33.32a
Glutamine	1091.21 \pm 110.42c	2080.6 \pm 46.08a	1588.15 \pm 12.71b	1419.42 \pm 97.1bc	1658.41 \pm 136.53b
Citrulline	206.91 \pm 11.49ab	212.98 \pm 3.74ab	198.04 \pm 22.99ab	177.67 \pm 13b	255.68 \pm 28.54a
Serine	138.98 \pm 5.18b	184.77 \pm 6.01a	118.77 \pm 6.27b	190.74 \pm 10.37a	177.16 \pm 12.68a
Aspartic acid	1157.26 \pm 118.87bc	1468.73 \pm 66.11ab	963.02 \pm 18.64c	1718.3 \pm 226.3a	1265.13 \pm 98.51abc
Hydroxy Proline	332.16 \pm 53.92bc	252 \pm 14.66cd	149.56 \pm 12.41d	425.35 \pm 25.22b	701.7 \pm 76.55a
Glycine	183.21 \pm 18.81b	224.12 \pm 12.91ab	152.46 \pm 2.95bc	260.21 \pm 35.15a	80.35 \pm 7.56c
Threonine	35.29 \pm 5.3b	37.04 \pm 2.93b	47.63 \pm 0.63b	72.39 \pm 8.45a	15.23 \pm 1.76c
Beta-alanine	49.19 \pm 7.52b	105.06 \pm 4.3a	25.66 \pm 1.44c	38.48 \pm 5.44bc	97.06 \pm 4.91a
Alanine	45.7 \pm 8.19b	77.27 \pm 9.33b	77.4 \pm 0.91b	72.3 \pm 11.16b	356.74 \pm 27.39a
GABA	1757.8 \pm 59.79a	1771.6 \pm 56.54a	881.06 \pm 90.76c	1191.47 \pm 57.37b	1609.51 \pm 81.9a
Proline	58.2 \pm 2.26ab	38.44 \pm 8.32bc	29.55 \pm 0.7c	35.35 \pm 9bc	73.81 \pm 4.28a
Methionine	27.65 \pm 1.27b	48.57 \pm 2.5a	28.15 \pm 1.46b	30.4 \pm 2.35b	57.05 \pm 3.38a
Valine	275.77 \pm 5.86b	224.45 \pm 34.56bc	167.08 \pm 7.9c	210.67 \pm 28.46bc	376.93 \pm 14.56a
Tryptophan	29.63 \pm 2.96c	48.24 \pm 5.72b	38.63 \pm 1.65bc	50.17 \pm 3.19b	67.4 \pm 2.33a
Phenylalanine	77 \pm 6.01b	76.52 \pm 4.53b	79.79 \pm 1.26b	99.87 \pm 7.12b	148.23 \pm 14.58a
Isoleucine	17.47 \pm 0.75bc	41.38 \pm 5.92a	13.25 \pm 0.15c	34.68 \pm 5.76a	29.05 \pm 17.79ab
Leucine	22.24 \pm 0.92c	31.87 \pm 2.61ab	17.56 \pm 0.76c	29.9 \pm 2.86b	37.7 \pm 4.92a

Table A-6. Amino acid contents of HD252 Honeydew variety represented as mean \pm SE

HD252					
Amino acid	0 Days	5 Days	10 Days	15 Days	20 Days
Asparagine	646.78 \pm 19.27a	471.68 \pm 13.1b	433.52 \pm 49.78b	434.15 \pm 78.13ab	589 \pm 27ab
Glutamine	1422.9 \pm 35.43a	1413.83 \pm 103.33a	1499.46 \pm 90.03a	1678.07 \pm 121.38a	1323.58 \pm 136.79a
Citrulline	192.75 \pm 6.58a	158.9 \pm 13.06a	159.82 \pm 18.58a	185.82 \pm 11.48a	203.92 \pm 20.57a
Serine	137.75 \pm 4.22a	127.43 \pm 6.23a	192.38 \pm 77.32a	144.49 \pm 9.2a	176.72 \pm 9.98a
Aspartic acid	1370.83 \pm 261.6a	1277.95 \pm 80.38a	1123.15 \pm 28.76a	1188.39 \pm 71.81a	1253.05 \pm 89.35a
Hydroxy Proline	337.04 \pm 124.3ab	255.15 \pm 6.62b	139.51 \pm 6.25b	239.12 \pm 58.06b	556.76 \pm 54.08a
Glycine	217.03 \pm 41.41a	198.48 \pm 13.29a	177.81 \pm 4.55a	207.46 \pm 11.82a	65.56 \pm 5.87b
Threonine	42.42 \pm 3.07b	41.97 \pm 2.73b	44.96 \pm 1.37ab	54.7 \pm 2.15a	11.66 \pm 2.05c
Beta-alanine	48.97 \pm 8.35bc	55.22 \pm 12.6b	24.59 \pm 2.74c	36.76 \pm 4.33bc	87.05 \pm 5.24a
Alanine	33.79 \pm 5.51b	76.52 \pm 9.57b	77.83 \pm 2.66b	54.4 \pm 7.4b	321.65 \pm 28.59a
GABA	2352.2 \pm 96.34a	1802.75 \pm 152.77b	1281.75 \pm 19.37c	1468.16 \pm 82.01bc	1624.14 \pm 143.16bc
Proline	58.93 \pm 1.26a	55.94 \pm 1.59a	33.67 \pm 3.3b	58.75 \pm 5.83a	65.16 \pm 3.5a
Methionine	46.07 \pm 1.87ab	47.3 \pm 1.03ab	40.6 \pm 2.06b	55.5 \pm 4.5a	45.69 \pm 4.42ab
Valine	377.1 \pm 14.21a	304.52 \pm 12.15b	215.7 \pm 5.12c	278.71 \pm 19.89b	303.85 \pm 19.29b
Tryptophan	34.09 \pm 0.81b	38.9 \pm 3.78b	28.36 \pm 3.1b	35.57 \pm 2.33b	52.84 \pm 3.44a
Phenylalanine	104.22 \pm 2.92a	93.45 \pm 8.93a	62.57 \pm 4.94b	93.18 \pm 5.74a	112.83 \pm 10.16a
Isoleucine	29.97 \pm 1.59a	30.3 \pm 1.51a	20.29 \pm 1.21a	25.05 \pm 1.71a	35.51 \pm 11.67a
Leucine	33.97 \pm 1.6a	38.01 \pm 1.48a	22.6 \pm 0.64b	28.94 \pm 4.66a	33.32 \pm 4.76a

Table A-7. Volatile compounds content of Western Shipper cantaloupe variety represented as mean \pm SE

SL NO	RT	Compound	Measured RI	ID	Library RI	0 Days	5 Days	10 Days	15 Days	20 Days
1	4.08	Ethyl 2-methylbutanoate	1029.35	MS,KI	1073	-	-	-	-	-
2	4.44	Hexanal	1044.44	MS,KI	1080	2.78 \pm 0.44	2.91 \pm 0.73	7.19 \pm 1.1	-	-
3	6.76	Limonene	1141.72	MS,KI,ST	1194	3.26 \pm 0.3	8.65 \pm 0.03	-	-	-
4	6.8	1,8-Cineole	1143.40	MS,KI	1198	-	-	-	-	-
5	7.28	2-Methylbutanol	1163.52	MS,KI	1212	-	-	-	20.39 \pm 3.48	-
6	7.7	2-Pentylfuran	1181.13	MS,KI	1240	4.48 \pm 0.43	10.19 \pm 3.55	-	-	-
7	7.9	Ethyl hexanoate	1189.52	MS,KI,ST	1244	1.3 \pm 0.02	-	-	-	-
8	8.27	Styrene	1204.59	MS,KI	1261	-	-	-	-	-
9	8.73	Octanal	1222.18	MS,KI	1278	-	-	-	-	3.99 \pm 2.23
10	9.42	3-Hydroxybutan-2-one	1248.57	MS,KI	1289	-	-	-	-	-
11	9.8	2,2,6-Trimethylcyclohexan-1-one	1263.10	MS,KI	n/a	0.6 \pm 0.03	3.2 \pm 0.86	3.59 \pm 0.22	-	-
12	10.04	6-Methylhept-5-en-2-one	1272.28	MS,KI,ST	1337	-	5.62 \pm 1.1	-	-	-
13	10.7	Dimethyl trisulfide	1297.51	MS,KI,ST	1383	-	-	-	6.82 \pm 1.94	-
14	11.58	Nonanal	1331.17	MS,KI	1396	4.96 \pm 0.52	6.37 \pm 1.34	17.6 \pm 1.21	14.39 \pm 4.34	74.18 \pm 33.08
15	12.3	(E)-4-Nonenal	1358.70	MS,KI	1435	-	-	-	-	-
16	12.38	(E)-2-Octenal	1361.76	MS,KI	1432	6.32 \pm 0.2	3.55 \pm 1.69	2.49 \pm 0	10.62 \pm 1.98	40.73 \pm 35.36
17	12.67	Ethyl caprylate	1372.85	MS,KI	1438	-	-	-	-	-
18	12.84	3,7-Dimethyloctan-3-ol	1379.35	MS,KI	n/a	-	-	8.3 \pm 0.66	-	-
19	12.85	(Z)-6-Nonenal	1379.73	MS,KI	1453	-	-	-	-	-
20	13.05	Ethyl 2-(methylthio)acetate	1387.38	MS,KI	1452	-	-	-	16.81 \pm 6.6	-
21	13.2	Acetic acid	1393.12	MS,KI	1455	-	-	-	-	-
22	13.22	1-Octen-3-ol	1393.88	MS,KI,ST	1456	1.48 \pm 0.21	4.05 \pm 1.68	-	-	-
23	13.4	(E,Z)-2,4-Heptadienal	1400.78	MS,KI	1464	-	-	-	-	-
24	13.6	Ethyl 2,4-hexadienoate	1408.61	MS,KI	1501	-	-	-	-	-

Table A-7 Continued

SL NO	RT	Compound	Measured RI	ID	Library RI	0 Days	5 Days	10 Days	15 Days	20 Days
25	13.8	(E,E)-2,4-Heptadienal	1416.44	MS,KI	1497	0.78±0.06	4.07±1.65	9.41±3.83	16.78±5.73	10.58±5.53
26	14.27	Decanal	1434.83	MS,KI,ST	1506	2.92±0.14	50.29±43.05	-	-	-
27	14.44	Benzaldehyde	1441.49	MS,KI,ST	1530	62.53±9.61	239.26±13.88	295.46±39.82	188.03±46.05	692.85±346.29
28	14.89	(E)-2-Nonenal	1459.10	MS,KI,ST	1543	42.93±1.9	90.5±20.73	87.2±11.93	134.17±27.52	451.98±81.26
29	15.7	(E,E)-3,5-Octadien-2-one	1490.80	MS,KI	1539	8.68±1.84	6.37±0.86	-	-	-
30	15.95	Ethyl 3-(methylthio)propionate	1500.59	MS,KI	1577	-	-	10.61±2.8	6.57±1.44	-
31	16.07	(E,Z)-2,6-nonadienal	1510.76	MS,KI,ST	1596	29.1±3.71	95.48±16.68	59.22±3.41	88.13±5.96	70.97±44.89
32	16.35	^{2,4} -Dimethylcyclohexanol	1524.07	MS,KI	n/a	3.61±0.77	19.22±10.13	-	-	-
33	16.66	Methyl benzoate	1545.60	MS,KI	1610	-	-	-	-	-
34	16.71	β-Cyclocitral	1535.03	MS,KI,ST	1623	14.6±3.03	41.2±12.7	86.08±0.23	85.67±3.84	119.73±53.55
35	17.2	Pheynlacetalddehyde	1559.30	MS,KI	1640	-	-	16.17±4.09	-	-
36	17.38	(E)-2-Decenal	1576.13	MS,KI	1643	2.04±0.71	15.6±0.69	-	-	-
37	17.38	Ethyl decanoate	1556.56	MS,KI,ST	1647	-	-	-	-	142.03±78.93
38	17.81	Ethyl benzoate	1573.39	MS,KI,ST	1658	1.81±0.77	49.66±38.81	41.42±2.24	387.22±94.75	264.12±98.3
39	18.08	(Z)-Citral	1583.95	MS,KI	1681	-	-	-	-	-
40	18.78	(Z)-3-Nonen-1-ol	1614.99	MS,KI	1682	-	-	-	-	-
41	18.93	α-Terpineol	1622.74	MS,KI	1688	22.26±0.3	55.32±4.27	23.71±5.31	139.82±9.27	107.16±49.65
42	19.3	(E)-Citral	1641.86	MS,KI	1733	-	-	-	-	-
43	19.34	Dodecanal	1643.93	MS,KI	1720	-	-	-	-	-
44	19.56	Ethyl 3-(methylthio)-(E)-2-propenoate	1655.30	MS,KI	1733	-	-	-	-	-
45	19.7	(E)-2-Undecenal	1662.53	MS,KI	n/a	2.01±0.36	8.28±6.67	12.05±3.39	40.3±10.86	93.44±68.21
46	20.18	(E,Z)-3,6-Nonadien-1-ol	1687.34	MS,KI	1749	-	-	-	-	-
47	20.3	1-Decanol	1693.54	MS,KI,ST	1760	-	-	-	-	-
48	20.35	α-Farnesene	1696.12	MS,KI	1750	-	-	-	-	-

Table A-7 Continued

SL NO	RT	Compound	Measured RI	ID	Library RI	0 Days	5 Days	10 Days	15 Days	20 Days
49	20.67	3-Phenylpropanal	1712.66	MS,KI	n/a	1.79±0.66	1.51±0.82	10.68±4.4	84.15±10.17	246.34±219.43
50	20.76	Methoxy-phenyl-oxime	1717.31	MS,KI	1773	-	-	-	-	-
51	20.98	(E,E)-2,4-Decadienal	1728.68	MS,KI	1819	-	-	-	-	-
52	21.77	(E)- α -Ionone	1769.51	MS,KI	n/a	1.48±0.11	7.95±1	-	-	-
53	21.8	(E)-Carveol	1771.06	MS,KI,ST	1858	-	-	-	-	-
54	22.06	Geranylacetone	1785.07	MS,KI,ST	1867	127.93±12.34	258.81±36.56	575.69±85.54	871.19±35.74	1821.43±193.94
55	22.44	3-Hydroxy-2,4,4-trimethylpentyl 2-methylpropanoate	1803.98	MS,KI	n/a	1.97±0.39	16.05±8.94	39.64±6.09	19.65±1.11	363.17±251.66
56	22.45	Benzyl alcohol	1804.48	MS,KI	1880	-	-	-	-	-
57	23	Phenylethyl alcohol	1831.84	MS,KI	1915	-	-	-	-	-
58	23.02	α -Calacorene	1832.84	MS,KI	n/a	1.54±0.19	5.17±1.12	32.14±7.55	65.1±8.7	29.11±2.62
59	23.3	2-Phenyl-2-butenal	1846.77	MS,KI	n/a	-	-	-	124.29±61.72	-
60	23.5	β -Ionone	1856.72	MS,KI,ST	1947	103.63±13.87	166.91±58.43	363.53±10.1	521.72±25.05	1130.83±183.26
61	24.5	β -Ionone epoxide	1906.47	MS,KI	1995	-	-	-	-	-
62	24.6	β -Ionol	1911.44	MS,KI,ST	1968	72.19±5.45	107.92±21.22	181.73±1.16	220.5±11.46	26.34±5.25
63	25.8	3-Phenylpropanol	1971.14	MS,KI	2058	2.22±0.56	2.39±0.51	6.21±2.49	10.08±3.47	-
64	26.03	5-Pentyl-2(5H)-furanone	1982.59	MS,KI	2068	2.74±0.44	2.6±1.61	-	-	-
65	26.5	Elemol	2006.47	MS,KI	2099	2.83±0.41	11.11±0.57	37.03±1.57	59.4±12.61	49.09±10.34
66	26.5	Globulol	2006.47	MS,KI	2073	-	-	-	-	-
67	26.71	1-Tridecanol	2017.79	MS,KI	n/a	-	-	-	-	-
68	27.36	Hexadecanal	2052.83	MS,KI	n/a	-	-	-	-	-
69	28	Eugenol	2087.33	MS,KI	2167	-	-	-	-	-
70	28.56	δ -Cadinol	2117.52	MS,KI	2179	1.42±0.2	5.96±0.2	11.57±1.94	23.87±2.83	-
71	28.8	α -Eudesmol	2130.46	MS,KI	2193	-	-	-	-	-
72	29.12	α -Cadinol	2147.71	MS,KI	2259	4.38±0.84	12.69±1.8	28.64±2.49	47.09±7.5	78.61±11.04

Table A-7 Continued

SL NO	RT	Compound	Measured RI	ID	Library RI	0 Days	5 Days	10 Days	15 Days	20 Days
73	29.13	Methyl hexadecanoate	2148.25	MS,KI	n/a	-	-	-	-	-
74	29.8	Ethyl hexadecenoate	2183.14	MS,KI	2288	-	-	-	-	-
75	30.2	Ethyl 9-hexadecenoate	2206.40	MS,KI	n/a	9.32±2.4	15.14±3.5	19.57±2.48	37.88±5.75	93.5±24.54
76	30.58	Dihydroactinidiolide	2228.49	MS,KI	2291	14.41±0.79	38.14±2.17	-	-	-
77	32	1-Hexadecanol	2311.05	MS,KI	2363	-	-	-	-	-
78	32.18	⁴ -Quinolinecarboxaldehyde	2321.51	MS,tn	2400	3.71±0.97	2.9±1	45.12±0.79	47.74±2.29	37.65±2.46
79	32.7	Farnesyl acetone	2351.74	MS,KI	n/a	13.85±1.06	15.97±2.92	8.22±1.56	12.78±2.05	148.9±42.95
73	29.13	Methyl hexadecanoate	2148.25	MS,KI	n/a	-	-	-	-	-

Table A-8. Volatile compound contents of Infinite Gold cantaloupe variety represented as mean \pm SE

SL NO	RT	Compound	Measured RI	ID	Library RI	0 Days	5 Days	10 Days	15 Days	20 Days
1	4.08	Ethyl 2-methylbutanoate	1029.35	MS,KI	1073	-	-	-	-	3.02 \pm 0.97
2	4.44	Hexanal	1044.44	MS,KI	1080	2.81 \pm 0.55	3.45 \pm 0.77	3.05 \pm 0.07	5.44 \pm 0.19	-
3	6.76	Limonene	1141.72	MS,KI,ST	1194	-	-	7.93 \pm 1.36	-	-
4	6.8	1,8-Cineole	1143.40	MS,KI	1198	-	-	-	-	-
5	7.28	2-Methylbutanol	1163.52	MS,KI	1212	-	4.07 \pm 0.51	10.21 \pm 5.55	-	-
6	7.7	2-Pentylfuran	1181.13	MS,KI	1240	-	-	-	9.32 \pm 0.63	-
7	7.9	Ethyl hexanoate	1189.52	MS,KI,ST	1244	-	-	-	-	-
8	8.27	Styrene	1204.59	MS,KI	1261	-	-	3.89 \pm 0.77	11.29 \pm 3.91	-
9	8.73	Octanal	1222.18	MS,KI	1278	-	-	1.62 \pm 1.02	3.41 \pm 0.52	-
10	9.42	3-Hydroxybutan-2-one	1248.57	MS,KI	1289	-	-	-	-	3.69 \pm 2.1
11	9.8	2,2,6-Trimethylcyclohexan-1-one	1263.10	MS,KI	n/a	-	-	-	3.48 \pm 0.91	4.06 \pm 0.88
12	10.04	6-Methylhept-5-en-2-one	1272.28	MS,KI,ST	1337	1.4 \pm 0.37	3.72 \pm 0.92	3 \pm 0.23	3.08 \pm 0.59	1.71 \pm 0.19
13	10.7	Dimethyl trisulfide	1297.51	MS,KI,ST	1383	-	-	2.8 \pm 0.09	4.79 \pm 1.16	-
14	11.58	Nonanal	1331.17	MS,KI	1396	5.45 \pm 1.81	18.83 \pm 2.15	10.8 \pm 3.63	11.51 \pm 0.48	8.19 \pm 0.38
15	12.3	(E)-4-Nonenal	1358.70	MS,KI	1435	-	4.02 \pm 0.86	-	-	-
16	12.38	(E)-2-Octenal	1361.76	MS,KI	1432	-	-	-	-	-
17	12.67	Ethyl caprylate	1372.85	MS,KI	1438	-	-	-	-	-
18	12.84	3,7-Dimethyloctan-3-ol	1379.35	MS,KI	n/a	-	-	-	-	-
19	12.85	(Z)-6-Nonenal	1379.73	MS,KI	1453	-	9.85 \pm 2.58	6.67 \pm 0.9	6.74 \pm 0.96	13.43 \pm 2.37
20	13.05	Ethyl 2-(methylthio)acetate	1387.38	MS,KI	1452	-	-	-	-	-
21	13.2	Acetic acid	1393.12	MS,KI	1455	-	-	-	8.65 \pm 2.26	22.78 \pm 5.85
22	13.22	1-Octen-3-ol	1393.88	MS,KI,ST	1456	1.75 \pm 0.39	4.89 \pm 1.06	6.63 \pm 2.86	-	-
23	13.4	(E,Z)-2,4-Heptadienal	1400.78	MS,KI	1464	-	-	-	-	-
24	13.6	Ethyl 2,4-hexadienoate	1408.61	MS,KI	1501	-	-	4.18 \pm 1.72	-	-

Table A-8 Continued

SL NO	RT	Compound	Measured RI	ID	Library RI	0 Days	5 Days	10 Days	15 Days	20 Days
25	13.8	(E,E)-2,4-Heptadienal	1416.44	MS,KI	1497	1.37±0.21	2.08±0.42	-	-	-
26	14.27	Decanal	1434.83	MS,KI,ST	1506	1.89±0.59	5.41±0.2	3.59±0.91	-	-
27	14.44	Benzaldehyde	1441.49	MS,KI,ST	1530	59.03±8.25	220.54±111.87	237.53±133.07	138.16±46.23	232.36±69.27
28	14.89	(E)-2-Nonenal	1459.10	MS,KI,ST	1543	75.04±17.37	65.58±8.54	73.27±1.96	61.81±7.78	66.1±2.03
29	15.7	(E,E)-3,5-Octadien-2-one	1490.80	MS,KI	1539	2.01±0.69	-	-	-	-
30	15.95	Ethyl 3-(methylthio)propionate	1500.59	MS,KI	1577	-	4.67±0.83	5.38±1.01	10.75±1.01	-
31	16.07	(E,Z)-2,6-nonadienal	1510.76	MS,KI,ST	1596	69.18±20.57	58.48±6.15	75.8±14.51	56.62±15.32	105.31±39.77
32	16.35	2,4-Dimethylcyclohexanol	1524.07	MS,KI	n/a	-	4.76±1.03	11.22±3.72	4.77±0.75	57.86±41.49
33	16.66	Methyl benzoate	1545.60	MS,KI	1610	-	-	-	-	69.12±8.83
34	16.71	β-Cyclocitral	1535.03	MS,KI,ST	1623	23.11±8.79	40.69±3.94	79±12.09	54.92±0.85	-
35	17.2	Pheynlacetalddehyde	1559.30	MS,KI	1640	-	10.01±1.95	15.22±0.43	17.86±5.31	-
36	17.38	(E)-2-Decenal	1576.13	MS,KI	1643	-	-	-	-	-
37	17.38	Ethyl decanoate	1556.56	MS,KI,ST	1647	-	-	-	-	-
38	17.81	Ethyl benzoate	1573.39	MS,KI,ST	1658	1.41±0.17	30.62±14.47	21.22±2.48	18.72±4.94	9.99±2.3
39	18.08	(Z)-Citral	1583.95	MS,KI	1681	-	-	-	-	-
40	18.78	(Z)-3-Nonen-1-ol	1614.99	MS,KI	1682	-	44.19±27.37	41.63±10.57	65.82±27.63	38.58±9.8
41	18.93	α-Terpineol	1622.74	MS,KI	1688	13.96±7.27	45.66±2.35	35.06±14.84	87.34±8.4	75.48±16.28
42	19.3	(E)-Citral	1641.86	MS,KI	1733	-	-	4.16±1.41	-	-
43	19.34	Dodecanal	1643.93	MS,KI	1720	-	-	-	-	-
44	19.56	Ethyl 3-(methylthio)-(E)-2-propenoate	1655.30	MS,KI	1733	-	-	3.82±1.64	-	-
45	19.7	(E)-2-Undecenal	1662.53	MS,KI	n/a	-	-	-	-	-
46	20.18	(E,Z)-3,6-Nonadien-1-ol	1687.34	MS,KI	1749	-	47.09±32.61	17.6±0.27	11.04±3.08	4.11±1.33
47	20.3	1-Decanol	1693.54	MS,KI,ST	1760	-	-	11.81±6.86	27.23±22.13	37.6±17.7
48	20.35	α-Farnesene	1696.12	MS,KI	1750	-	-	-	-	-

Table A-8 Continued

SL NO	RT	Compound	Measured RI	ID	Library RI	0 Days	5 Days	10 Days	15 Days	20 Days
49	20.67	3-Phenylpropanal	1712.66	MS,KI	n/a	2.03±0.55	3.97±0.19	-	-	-
50	20.76	Methoxy-phenyl-oxime	1717.31	MS,KI	1773	-	-	27.11±15.94	6.86±2.45	-
51	20.98	(E,E)-2,4-Decadienal	1728.68	MS,KI	1819	-	-	-	-	-
52	21.77	(E)- α -Ionone	1769.51	MS,KI	n/a	-	-	-	-	-
53	21.8	(E)-Carveol	1771.06	MS,KI,ST	1858	-	-	8.59±1.57	9.6±1.23	-
54	22.06	Geranylacetone	1785.07	MS,KI,ST	1867	60.58±6.81	118.06±9.62	187.1±37.83	238.46±38.9	223.75±75.92
55	22.44	3-Hydroxy-2,4,4-trimethylpentyl 2-methylpropanoate	1803.98	MS,KI	n/a	-	-	-	-	-
56	22.45	Benzyl alcohol	1804.48	MS,KI	1880	12.86±0.37	78.4±1.9	88.95±13.41	69.07±12.73	56.36±23.01
57	23	Phenylethyl alcohol	1831.84	MS,KI	1915	-	-	-	-	-
58	23.02	α -Calacorene	1832.84	MS,KI	n/a	-	-	16.07±0.01	42.85±36.38	24.17±11.55
59	23.3	2-Phenyl-2-butenal	1846.77	MS,KI	n/a	1.59±0.48	17.97±6.14	44.25±27.87	-	-
60	23.5	β -Ionone	1856.72	MS,KI,ST	1947	84.8±6.49	159.18±17.41	309.83±69.42	281.83±40.54	254.99±84.68
61	24.5	β -Ionone epoxide	1906.47	MS,KI	1995	-	-	-	-	-
62	24.6	β -Ionol	1911.44	MS,KI,ST	1968	34.8±7.45	63.72±8.77	32.54±29.16	83.22±43.53	96.65±3.05
63	25.8	3-Phenylpropanol	1971.14	MS,KI	2058	1.34±0.35	6.88±0.52	21.37±0	7.4±1.37	4.52±0.27
64	26.03	5-Pentyl-2(5H)-furanone	1982.59	MS,KI	2068	-	-	-	-	-
65	26.5	Elemol	2006.47	MS,KI	2099	1.79±0.61	16.27±3.48	24.25±9.52	28.31±3.13	29.18±9.02
66	26.5	Globulol	2006.47	MS,KI	2073	-	-	-	-	-
67	26.71	1-Tridecanol	2017.79	MS,KI	n/a	-	-	-	-	-
68	27.36	Hexadecanal	2052.83	MS,KI	n/a	-	-	-	-	-
69	28	Eugenol	2087.33	MS,KI	2167	-	-	5.66±0.78	6.03±0.84	15.59±5.16
70	28.56	δ -Cadinol	2117.52	MS,KI	2179	-	4.38±1.32	1.87±0.6	6.58±1.36	22.01±8.37
71	28.8	α -Eudesmol	2130.46	MS,KI	2193	-	-	5.2±0.77	3.68±0.35	-
72	29.12	α -Cadinol	2147.71	MS,KI	2259	-	-	20.52±2.52	19.4±2.16	24.36±1.78

Table A-8 Continued

SL NO	RT	Compound	Measured RI	ID	Library RI	0 Days	5 Days	10 Days	15 Days	20 Days
73	29.13	Methyl hexadecanoate	2148.25	MS,KI	n/a	-	9.68±2.06	-	-	-
74	29.8	Ethyl hexadecenoate	2183.14	MS,KI	2288	-	-	-	-	-
75	30.2	Ethyl 9-hexadecenoate	2206.40	MS,KI	n/a	-	-	-	27.51±3.12	42.98±15.81
76	30.58	Dihydroactinidiolide	2228.49	MS,KI	2291	-	-	-	-	-
77	32	1-Hexadecanol	2311.05	MS,KI	2363	-	-	5.61±0.7	40.1±17.88	21.74±3.71
78	32.18	4-Quinolinecarboxaldehyde	2321.51	MS,tn	2400	2.35±0.46	-	-	-	-
79	32.7	Farnesyl acetone	2351.74	MS,KI	n/a	3.62±0.79	11.71±3.85	-	-	-

Table A-9. Volatile compound contents of Da Vinci cantaloupe variety represented as mean \pm SE

SL NO	RT	Compound	Measured RI	ID	Library RI	0 Days	5 Days	10 Days	15 Days	20 Days
1	4.08	Ethyl 2-methylbutanoate	1029.35	MS,KI	1073	95.6 \pm 61.07	4.02 \pm 2.3	24.36 \pm 4.37	180.82 \pm 5	-
2	4.44	Hexanal	1044.44	MS,KI	1080	56.02 \pm 17.17	11.27 \pm 7.56	-	-	-
3	6.76	Limonene	1141.72	MS,KI,ST	1194	30.77 \pm 2.63	6.12 \pm 0.9	-	-	-
4	6.8	1,8-Cineole	1143.40	MS,KI	1198	-	-	11.32 \pm 4.94	60.94 \pm 19.23	-
5	7.28	2-Methylbutanol	1163.52	MS,KI	1212	42.06 \pm 20.45	6.3 \pm 0.82	11.11 \pm 1.37	-	-
6	7.7	2-Pentylfuran	1181.13	MS,KI	1240	-	-	-	-	27 \pm 6.52
7	7.9	Ethyl hexanoate	1189.52	MS,KI,ST	1244	-	4.58 \pm 1.04	14.29 \pm 1.45	40.23 \pm 2.54	30.95 \pm 5.97
8	8.27	Styrene	1204.59	MS,KI	1261	7.97 \pm 2.33	3.9 \pm 1.28	-	-	-
9	8.73	Octanal	1222.18	MS,KI	1278	-	-	4.49 \pm 0.47	4.68 \pm 0.66	-
10	9.42	3-Hydroxybutan-2-one	1248.57	MS,KI	1289	-	-	-	-	-
11	9.8	2,2,6-Trimethylcyclohexan-1-one	1263.10	MS,KI	n/a	-	-	-	14.34 \pm 5.25	-
12	10.04	6-Methylhept-5-en-2-one	1272.28	MS,KI,ST	1337	-	-	-	-	-
13	10.7	Dimethyl trisulfide	1297.51	MS,KI,ST	1383	-	-	-	-	3.34 \pm 0.93
14	11.58	Nonanal	1331.17	MS,KI	1396	66.6 \pm 19.59	11.37 \pm 1.05	12.55 \pm 0.85	-	-
15	12.3	(E)-4-Nonenal	1358.70	MS,KI	1435	-	-	-	-	-
16	12.38	(E)-2-Octenal	1361.76	MS,KI	1432	58.51 \pm 2.56	6.27 \pm 1.36	24.76 \pm 8.56	43.66 \pm 4.29	62.71 \pm 16.26
17	12.67	Ethyl caprylate	1372.85	MS,KI	1438	-	-	-	-	-
18	12.84	3,7-Dimethyloctan-3-ol	1379.35	MS,KI	n/a	-	-	-	-	-
19	12.85	(Z)-6-Nonenal	1379.73	MS,KI	1453	28.08 \pm 5.06	14.53 \pm 4.23	-	-	-
20	13.05	Ethyl 2-(methylthio)acetate	1387.38	MS,KI	1452	-	-	6.78 \pm 3.59	23.01 \pm 4.51	-
21	13.2	Acetic acid	1393.12	MS,KI	1455	-	-	-	-	-
22	13.22	1-Octen-3-ol	1393.88	MS,KI,ST	1456	-	-	-	-	-
23	13.4	(E,Z)-2,4-Heptadienal	1400.78	MS,KI	1464	-	-	-	-	8.28 \pm 4.32
24	13.6	Ethyl 2,4-hexadienoate	1408.61	MS,KI	1501	-	-	-	-	-

Table A-9 Continued

SL NO	RT	Compound	Measured RI	ID	Library RI	0 Days	5 Days	10 Days	15 Days	20 Days
25	13.8	(E,E)-2,4-Heptadienal	1416.44	MS,KI	1497	-	2.89±1.05	-	-	-
26	14.27	Decanal	1434.83	MS,KI,ST	1506	-	-	-	254.18±38.05	-
27	14.44	Benzaldehyde	1441.49	MS,KI,ST	1530	969.13±121.83	121.25±11.85	108.6±49.79	177.35±30.7	167.62±128.09
28	14.89	(E)-2-Nonenal	1459.10	MS,KI,ST	1543	496.34±75.99	42.39±11.98	6.09±1.73	32.19±11.54	195.05±87.13
29	15.7	(E,E)-3,5-Octadien-2-one	1490.80	MS,KI	1539	-	6.55±1.37	-	-	-
30	15.95	Ethyl 3-(methylthio)propionate	1500.59	MS,KI	1577	-	-	43.41±9.19	-	-
31	16.07	(E,Z)-2,6-nonadienal	1510.76	MS,KI,ST	1596	-	37.46±10.45	-	103.51±14.92	154.78±27.36
32	16.35	2,4-Dimethylcyclohexanol	1524.07	MS,KI	n/a	-	7.03±0.53	7.52±1.74	108.58±12.33	-
33	16.66	Methyl benzoate	1545.60	MS,KI	1610	-	-	-	-	-
34	16.71	β-Cyclocitral	1535.03	MS,KI,ST	1623	223.95±53.84	49.9±4.11	75.79±20.44	91.51±48.42	188.74±58.44
35	17.2	Pheynlacetaldehyde	1559.30	MS,KI	1640	42.9±6.76	12.24±0.76	13.39±0.57	-	-
36	17.38	(E)-2-Decenal	1576.13	MS,KI	1643	48.68±5.98	-	-	-	-
37	17.38	Ethyl decanoate	1556.56	MS,KI,ST	1647	-	-	-	-	-
38	17.81	Ethyl benzoate	1573.39	MS,KI,ST	1658	-	-	48.1±10.16	178.24±13.19	-
39	18.08	(Z)-Citral	1583.95	MS,KI	1681	34.59±3.95	-	-	-	-
40	18.78	(Z)-3-Nonen-1-ol	1614.99	MS,KI	1682	-	-	-	156.64±23.1	119.87±23.03
41	18.93	α-Terpineol	1622.74	MS,KI	1688	60.76±10.25	51.24±8.64	33.85±11.88	115.66±34.62	144.96±10.8
42	19.3	(E)-Citral	1641.86	MS,KI	1733	-	3.38±1.05	5.15±0.99	25.7±15.32	-
43	19.34	Dodecanal	1643.93	MS,KI	1720	16.59±4.23	-	-	-	-
44	19.56	Ethyl 3-(methylthio)-(E)-2-propenoate	1655.30	MS,KI	1733	-	-	-	-	-
45	19.7	(E)-2-Undecenal	1662.53	MS,KI	n/a	-	-	9.36±3.7	-	-
46	20.18	(E,Z)-3,6-Nonadien-1-ol	1687.34	MS,KI	1749	-	-	-	20.01±1.78	106.17±46.85
47	20.3	1-Decanol	1693.54	MS,KI,ST	1760	-	-	11.44±1.6	-	-
48	20.35	α-Farnesene	1696.12	MS,KI	1750	-	-	-	20.92±7.42	-

Table A-9 Continued

SL NO	RT	Compound	Measured RI	ID	Library RI	0 Days	5 Days	10 Days	15 Days	20 Days
49	20.67	3-Phenylpropanal	1712.66	MS,KI	n/a	10.66±0.37	3.06±0.77	-	-	-
50	20.76	Methoxy-phenyl-oxime	1717.31	MS,KI	1773	-	-	50.25±12.53	104.83±46	206.08±53.06
51	20.98	(E,E)-2,4-Decadienal	1728.68	MS,KI	1819	45.79±2.03	-	-	-	-
52	21.77	(E)- α -Ionone	1769.51	MS,KI	n/a	-	-	-	-	-
53	21.8	(E)-Carveol	1771.06	MS,KI,ST	1858	-	-	26.48±15.06	40.41±4.8	-
54	22.06	Geranylacetone	1785.07	MS,KI,ST	1867	1550.22±160.92	208.38±28.91	355.46±154.42	-	-
55	22.44	3-Hydroxy-2,4,4-trimethylpentyl 2-methylpropanoate	1803.98	MS,KI	n/a	-	-	-	-	-
56	22.45	Benzyl alcohol	1804.48	MS,KI	1880	83.52±10.61	35.71±5.09	68.97±2.27	132.72±59.29	851.39±238.12
57	23	Phenylethyl alcohol	1831.84	MS,KI	1915	-	17.18±6.33	32.15±4.23	-	-
58	23.02	α -Calacorene	1832.84	MS,KI	n/a	51.18±21.07	-	-	-	-
59	23.3	2-Phenyl-2-butenal	1846.77	MS,KI	n/a	-	-	-	118.5±32.53	-
60	23.5	β -Ionone	1856.72	MS,KI,ST	1947	2010.02±21.9	247.33±25.33	382.11±82.45	812.61±150.94	34.57±6.33
61	24.5	β -Ionone epoxide	1906.47	MS,KI	1995	-	-	185.95±40.88	-	-
62	24.6	β -Ionol	1911.44	MS,KI,ST	1968	1102.83±62.02	112.64±8.16	-	322.68±67.49	-
63	25.8	3-Phenylpropanol	1971.14	MS,KI	2058	-	-	-	8.8±1.28	457.44±206.07
64	26.03	5-Pentyl-2(5H)-furanone	1982.59	MS,KI	2068	-	-	-	-	8.6±5.21
65	26.5	Elemol	2006.47	MS,KI	2099	74.88±26.81	-	-	-	-
66	26.5	Globulol	2006.47	MS,KI	2073	-	31.58±2.11	-	-	-
67	26.71	1-Tridecanol	2017.79	MS,KI	n/a	-	-	-	-	14.24±6.63
68	27.36	Hexadecanal	2052.83	MS,KI	n/a	51.51±2.33	-	-	-	-
69	28	Eugenol	2087.33	MS,KI	2167	-	-	-	-	-
70	28.56	δ -Cadinol	2117.52	MS,KI	2179	1.41±0.48	6.77±2.82	16.65±1.29	27.07±11.81	66.74±1.85
71	28.8	α -Eudesmol	2130.46	MS,KI	2193	-	-	-	-	-
72	29.12	α -Cadinol	2147.71	MS,KI	2259	71.77±4.87	12.21±1.25	9.67±0.66	23.94±2.88	45.34±4.12

Table A-9 Continued

SL NO	RT	Compound	Measured RI	ID	Library RI	0 Days	5 Days	10 Days	15 Days	20 Days
73	29.13	Methyl hexadecanoate	2148.25	MS,KI	n/a	-	-	-	-	-
74	29.8	Ethyl hexadecenoate	2183.14	MS,KI	2288	-	8.89±0.68	-	-	-
75	30.2	Ethyl 9-hexadecenoate	2206.40	MS,KI	n/a	-	-	14.9±0.93	168.03±38.23	43.74±11.68
76	30.58	Dihydroactinidiolide	2228.49	MS,KI	2291	258.05±7.98	36.15±5.96	48.42±9.79	58.98±20.67	-
77	32	1-Hexadecanol	2311.05	MS,KI	2363	-	-	-	-	-
78	32.18	4-Quinolinecarboxaldehyde	2321.51	MS,tn	2400	76.99±2.68	6.61±1.97	-	-	-
79	32.7	Farnesyl acetone	2351.74	MS,KI	n/a	-	10.03±2.21	24.05±7.79	101.81±22.69	87.01±6.32

Table A-10. Volatile compound contents of Orange Casaba honeydew variety represented as mean \pm SE

SL NO	RT	Compound	Measured RI	ID	Library RI	0 Days	5 Days	10 Days	15 Days	20 Days
1	4.5	Hexanal	1046.96	MS,KI	1080	2.45 \pm 0.92	13.71 \pm 1.49	23.7 \pm 2.16	30.2 \pm 19.78	-
2	6.5	Heptanal	1130.82	MS,KI	1080	-	-	-	-	-
3	6.7	Limonene	1139.20	MS,KI,ST	1194	3.69 \pm 0.5	24.54 \pm 5.91	-	-	-
4	7.41	(E)-2-Hexanal	1168.97	MS,KI	1198	-	-	-	-	-
5	7.82	Styrene	1186.16	MS,KI	1212	-	-	-	-	-
6	7.97	2-Pentylfuran	1192.45	MS,KI	1240	6.97 \pm 1.12	21.56 \pm 3.88	42.29 \pm 3.48	21.31 \pm 0.52	46.5 \pm 7.33
7	8.8	Octanal	1227.25	MS,KI	1244	-	-	-	-	-
8	9.34	(E)-2-(2-Pentenyl)furan	1245.51	MS,KI	1261	1.74 \pm 0.37	6.16 \pm 0.94	-	-	-
9	9.81	(E)-2-Heptenal	1263.48	MS,KI	1278	-	5.04 \pm 1.34	-	-	-
10	10.24	3-Hexenyl acetate	1279.92	MS,KI	1289	-	7.34 \pm 3.05	-	-	-
11	10.28	6-Methylhept-5-en-2-one	1281.45	MS,KI,ST	n/a	0.73 \pm 0.05	11.29 \pm 2.4	13.12 \pm 2.61	6.96 \pm 2.88	4.44 \pm 1.41
12	10.6	Dimethyl trisulfide	1293.69	MS,KI,ST	1337	-	-	3.81 \pm 1.2	5.52 \pm 3.02	4.28 \pm 1.78
13	11.6	Nonanal	1331.93	MS,KI	1383	5.95 \pm 1.63	24.3 \pm 2.74	13.64 \pm 2.54	24.21 \pm 2.17	55.13 \pm 19.57
14	11.92	1-Octen-3-ol	1344.17	MS,KI,ST	1396	1.71 \pm 0.18	-	-	-	-
15	12.4	(E)-2-Octenal	1362.52	MS,KI	1435	1.6 \pm 0.22	14.27 \pm 1.21	35.63 \pm 2.1	7.51 \pm 2.85	31.89 \pm 20.57
16	12.74	3,7-Dimethyloctan-3-ol	1375.53	MS,KI	1432	-	-	-	-	-
17	12.93	(Z)-6-Nonenal	1382.79	MS,KI	1438	1.9 \pm 0.69	16.89 \pm 2.25	10.9 \pm 1.66	19.59 \pm 9.63	12.75 \pm 3.87
18	13.14	(E,Z)-2,4-Heptadienal	1390.82	MS,KI	n/a	-	-	24.27 \pm 3.35	11.77 \pm 7.54	8.22 \pm 2.58
19	13.28	(E,E)-2,4-Heptadienal	1396.18	MS,KI	1453	1.03 \pm 0.03	9.14 \pm 0.43	42.33 \pm 0.77	18.91 \pm 4.48	32.08 \pm 17.22
20	13.3	Acetic acid	1396.94	MS,KI	1452	-	-	-	-	-
21	14.12	Decanal	1428.30	MS,KI,ST	1455	-	-	-	-	-
22	14.5	(E,Z)-3,5-Octadien-2-one	1442.83	MS,KI	1456	-	-	45.69 \pm 1.13	39.03 \pm 11.82	37.27 \pm 10.73

Table A-10 Continued

SL NO	RT	Compound	Measured RI	ID	Library RI	0 Days	5 Days	10 Days	15 Days	20 Days
23	14.55	Benzaldehyde	1445.79	MS,KI,ST	1464	9.73±1.56	19.38±0.97	52.92±5.86	17.2±8.78	-
24	14.89	(E)-2-Nonenal	1459.10	MS,KI,ST	1501	50.32±3.55	56.9±7.69	18.3±8.57	82.8±22.13	50.39±24.08
25	15.61	Ethyl 3-(methylthio)propionate	1487.28	MS,KI	1497	-	-	15.36±3.99	29.3±20.32	33.82±15.54
26	15.7	1-Octanol	1490.80	MS,KI	1506	-	-	-	-	-
27	16.07	(E,Z)-2,6-nonadienal	1505.28	MS,KI,ST	1530	80.82±9.1	51.84±15.65	18.73±6.24	41.88±26.93	61.93±20.76
28	16.3	2,4-Dimethylcyclohexanol	1514.29	MS,KI	1543	-	-	30.3±4.23	51.71±23	26.83±11.44
29	16.7	β-Cyclocitral	1529.94	MS,KI,ST	1539	10.95±0.3	25.53±8.96	33.38±4.61	84.23±33.51	56.52±9.17
30	17.04	(E)-2-Octen-1-ol	1543.25	MS,KI	1577	-	-	-	-	-
31	17.18	Pheynlactaldehyde	1510.76	MS,KI	1596	-	-	-	-	38.47±27.36
32	17.36	(E)-2-Decenal	1524.07	MS,KI	n/a	-	-	-	-	-
33	17.43	Ethyl decanoate	1545.60	MS,KI,ST	1610	-	84.62±51.38	-	-	-
34	17.74	Ethyl benzoate	1535.03	MS,KI,ST	1623	-	-	59.49±6.38	127.19±57.03	22.2±12.6
35	18.1	1-Nonanol	1559.30	MS,KI	1640	-	-	-	-	-
36	18.58	(Z)-3-Nonen-1-ol	1576.13	MS,KI	1643	47.15±10.49	94.27±30.48	190.34±35.19	181.09±35.54	174.59±88.87
37	18.58	Diethyl butanedioate	1603.52	MS,KI	1647	-	-	-	-	-
38	18.72	α-Terpineol	1609.00	MS,KI	1658	33.01±5.88	370.05±45.55	135.29±51.58	125.15±47.55	361.38±33.82
39	19.3	(E)-Citral	1631.70	MS,KI	1681	-	-	-	-	43.15±33.68
40	19.31	(Z)-6-nonen-1-ol	1642.38	MS,KI	1682	-	107.7±39.41	6.76±0.33	52.15±33.94	-
41	19.78	(E)-2-Undecenal	1666.67	MS,KI	1688	-	-	-	-	-
42	20	(E,Z)-3,6-Nonadien-1-ol	1678.04	MS,KI	1733	31.91±15.07	260.9±20.69	51.82±2.79	109.08±61.38	91.01±68.59
43	20.31	1-Decanol	1694.06	MS,KI,ST	1720	-	-	15.8±0.31	38.07±21.39	50.78±4.02
44	20.8	Methoxy-phenyl-oxime	1719.38	MS,KI	1733	-	-	22.74±4.02	88.41±29.9	31.62±5.22

Table A-10 Continued

SL NO	RT	Compound	Measured RI	ID	Library RI	0 Days	5 Days	10 Days	15 Days	20 Days
45	20.94	(E,E)-2,4-Decadienal	1726.61	MS,KI	n/a	-	-	-	-	54.51±24.17
46	21.47	4-Oxononanal	1754.01	MS,KI	1749	-	-	36.09±19.57	10.76±3.28	84.04±60.1
47	21.68	(E)-Carveol	1764.86	MS,KI,ST	1760	-	-	-	-	8.48±0.14
48	22.06	Geranylacetone	1784.50	MS,KI,ST	1750	50.12±4.42	208.49±36.57	204.28±68.19	200.72±19.78	348.72±53.4
49	22.32	Phenylethyl alcohol	1797.93	MS,KI	n/a	-	-	140.6±9.06	59.36±17.49	-
50	22.5	trans-Isolimonene	1807.24	MS,KI	1773	-	108.85±41.91	-	-	-
52	23.05	α -Calacorene	1835.66	MS,KI	1819	2.25±0.63	-	-	-	-
53	23.5	β -Ionone	1858.91	MS,KI,ST	n/a	57.73±2.59	154.31±30.74	179.34±24.88	210.31±25.12	240.17±102.77
54	23.66	Benzothiazole	1867.18	MS,KI	1858	-	-	-	-	-
55	23.7	Heptanoic acid	1866.67	MS,KI	1867	-	-	-	-	-
56	24.5	β -Ionol	1906.47	MS,KI,ST	n/a	16.39±0.67	99.37±27.33	97.37±51.71	113.17±26.95	118.87±20.13
57	24.55	1-Dodecanol	1908.96	MS,KI	1880	-	31.01±8.31	-	-	-
58	24.6	β -Ionone epoxide	1911.44	MS,KI	1915	-	-	-	-	-
59	24.94	Cis-4,5-Epoxy-(E)-2-decenal	1928.36	MS,KI	n/a	-	-	-	-	-
60	24.95	Trans-4,5-Epoxy-(E)-2-decenal	1928.86	MS,KI	n/a	-	-	-	-	125.85±34.11
61	25.37	3-Phenylpropanol	1949.75	MS,KI	1947	0.85±0.13	6.5±2.59	35.06±11.83	26.96±11.69	-
62	26.17	5-Pentyl-2(5H)-furanone	1989.55	MS,KI	1995	1.21±0.31	-	-	-	7.57±0.72
63	26.29	Globulol	1995.52	MS,KI	1968	-	-	6.58±2.27	6.09±2.38	-
64	26.5	Elemol	2005.97	MS,KI	2058	-	-	-	-	-
65	26.51	1-Tridecanol	2006.47	MS,KI	2068	-	-	-	-	-
66	27.2	Z3,Z6,Z8-Dodecatrien-1-ol	2044.20	MS,KI	2099	-	-	-	-	13.14±6.03
67	28.35	Nonanoic acid	2106.20	MS,KI	2073	-	-	-	-	-

Table A-10 Continued

SL NO	RT	Compound	Measured RI	ID	Library RI	0 Days	5 Days	10 Days	15 Days	20 Days
68	28.6	δ -Cadinol	2119.68	MS,KI	n/a	1.73 \pm 0.19	5.91 \pm 0.59	12.41 \pm 1.58	14.84 \pm 6.72	34.5 \pm 6.93
69	29.11	α -Cadinol	2147.17	MS,KI	n/a	-	-	23.97 \pm 1.26	27.95 \pm 8.4	28.28 \pm 6.57
70	29.5	Methyl hexadecanoate	2168.19	MS,KI	2167	-	-	6.78 \pm 0.25	22.92 \pm 7.24	16.95 \pm 6.79
71	29.81	Ethyl hexadecanoate	2184.91	MS,KI	2179	7.58 \pm 1.34	19.76 \pm 1.22	18.25 \pm 0.75	30.59 \pm 3.36	56.46 \pm 2.66
72	30.23	Ethyl 9-hexadecenoate	2207.55	MS,KI	2193	20.34 \pm 8.89	22.29 \pm 0.31	18.14 \pm 3.1	31.22 \pm 11.69	42.35 \pm 16.15
73	31.24	Farnesyl acetone	2261.99	MS,KI,ST	2259	4.04 \pm 1.06	8.29 \pm 2.88	-	-	-
74	31.7	2,3-Dihydrobenzofuran	2286.79	MS,KI	n/a	7.23 \pm 1.29	-	-	-	-
75	32.18	4-Quinolinecarboxaldehyde	2321.51	MS,KI	2288	-	-	3.76 \pm 0.8	12.13 \pm 3.15	-
76	32.5	1-Hexadecanol	2340.12	MS,tn	n/a	-	-	-	-	56.92 \pm 13.12

Table A-11. Volatile compound contents of HD150 Honeydew variety represented as mean \pm SE

SL NO	RT	Compound	Measured RI	ID	Library RI	0 Days	5 Days	10 Days	15 Days	20 Days
1	4.5	Hexanal	1046.96	MS,KI	1080	9.7 \pm 2.93	29.35 \pm 11.48	40.41 \pm 9.03	29.46 \pm 4.34	50.09 \pm 4.01
2	6.5	Heptanal	1130.82	MS,KI	1080	-	-	6.2 \pm 0.69	16.55 \pm 5.32	-
3	6.7	Limonene	1139.20	MS,KI,ST	1194	-	16.96 \pm 1.54	-	-	-
4	7.41	(E)-2-Hexanal	1168.97	MS,KI	1198	-	-	-	-	-
5	7.82	Styrene	1186.16	MS,KI	1212	-	-	12.8 \pm 1.22	9.6 \pm 7.84	16.66 \pm 8.1
6	7.97	2-Pentylfuran	1192.45	MS,KI	1240	21.02 \pm 2.12	25.77 \pm 5.77	16.83 \pm 2.09	-	-
7	8.8	Octanal	1227.25	MS,KI	1244	-	-	-	-	4.83 \pm 0.77
8	9.34	(E)-2-(2-Pentenyl)furan	1245.51	MS,KI	1261	-	14 \pm 1.9	-	-	-
9	9.81	(E)-2-Heptenal	1263.48	MS,KI	1278	7.94 \pm 0.5	28.45 \pm 4.18	14.13 \pm 1.46	14.47 \pm 1.9	21.06 \pm 0.18
10	10.24	3-Hexenyl acetate	1279.92	MS,KI	1289	-	-	-	-	-
11	10.28	6-Methylhept-5-en-2-one	1281.45	MS,KI,ST	n/a	-	9.31 \pm 0.44	-	-	-
12	10.6	Dimethyl trisulfide	1293.69	MS,KI,ST	1337	-	-	-	-	-
13	11.6	Nonanal	1331.93	MS,KI	1383	29.64 \pm 2.9	37.66 \pm 15.63	11.02 \pm 1.93	12.23 \pm 2.53	13.96 \pm 1.45
14	11.92	1-Octen-3-ol	1344.17	MS,KI,ST	1396	-	-	-	-	32.32 \pm 1.72
15	12.4	(E)-2-Octenal	1362.52	MS,KI	1435	21.36 \pm 3.43	69.56 \pm 7.3	67.26 \pm 9.88	57.51 \pm 15.85	97.51 \pm 3.03
16	12.74	3,7-Dimethyloctan-3-ol	1375.53	MS,KI	1432	-	-	-	-	-
17	12.93	(Z)-6-Nonenal	1382.79	MS,KI	1438	15.46 \pm 0.48	29.89 \pm 10.75	-	19.96 \pm 9.22	13.51 \pm 3.88
18	13.14	(E,Z)-2,4-Heptadienal	1390.82	MS,KI	n/a	-	-	-	-	-
19	13.28	(E,E)-2,4-Heptadienal	1396.18	MS,KI	1453	8.25 \pm 1.65	44.46 \pm 8.22	42.54 \pm 13.46	51.18 \pm 18.47	48.87 \pm 9.55
20	13.3	Acetic acid	1396.94	MS,KI	1452	-	-	-	-	-
21	14.12	Decanal	1428.30	MS,KI,ST	1455	-	-	-	-	-
22	14.5	(E,Z)-3,5-Octadien-2-one	1442.83	MS,KI	1456	-	-	29.06 \pm 2.15	39.04 \pm 10.24	28.55 \pm 5.68

Table A-11 Continued

SL NO	RT	Compound	Measured RI	ID	Library RI	0 Days	5 Days	10 Days	15 Days	20 Days
23	14.55	Benzaldehyde	1445.79	MS,KI,ST	1464	29.85±2.94	52.07±6.22	-	-	-
24	14.89	(E)-2-Nonenal	1459.10	MS,KI,ST	1501	-	75.95±18.64	47.63±1.47	51.18±11.54	59.49±24.54
25	15.61	Ethyl 3-(methylthio)propionate	1487.28	MS,KI	1497	-	-	-	-	-
26	15.7	1-Octanol	1490.80	MS,KI	1506	-	-	9.85±0.95	9±1.7	11.79±1.66
27	16.07	(E,Z)-2,6-nonadienal	1505.28	MS,KI,ST	1530	52.94±25.9	80.85±42.05	9.75±0.56	25.32±7.95	9.73±4.19
28	16.3	2,4-Dimethylcyclohexanol	1514.29	MS,KI	1543	-	-	-	-	-
29	16.7	β-Cyclocitral	1529.94	MS,KI,ST	1539	7.03±0.46	-	-	-	-
30	17.04	(E)-2-Octen-1-ol	1543.25	MS,KI	1577	-	-	3.71±0.47	3.78±1.02	4.91±0.91
31	17.18	Pheynlacetalddehyde	1510.76	MS,KI	1596	-	-	-	-	-
32	17.36	(E)-2-Decenal	1524.07	MS,KI	n/a	-	-	-	5.72±0.84	11.11±2.26
33	17.43	Ethyl decanoate	1545.60	MS,KI,ST	1610	-	-	-	-	-
34	17.74	Ethyl benzoate	1535.03	MS,KI,ST	1623	-	-	-	-	-
35	18.1	1-Nonanol	1559.30	MS,KI	1640	-	-	-	-	165.14±6.77
36	18.58	(Z)-3-Nonen-1-ol	1576.13	MS,KI	1643	-	-	109.15±12.11	132.25±61.62	144.08±66.03
37	18.58	Diethyl butanedioate	1603.52	MS,KI	1647	-	198.92±33.71	-	-	-
38	18.72	α-Terpineol	1609.00	MS,KI	1658	55.9±3.79	155.78±48.53	64.78±10.43	129.91±27.77	101.5±75.95
39	19.3	(E)-Citral	1631.70	MS,KI	1681	-	-	-	-	-
40	19.31	(Z)-6-nonen-1-ol	1642.38	MS,KI	1682	-	-	33.66±3.06	73.7±3.67	34.75±4.86
41	19.78	(E)-2-Undecenal	1666.67	MS,KI	1688	-	-	22.24±2.95	43.56±16.45	35.01±5.76
42	20	(E,Z)-3,6-Nonadien-1-ol	1678.04	MS,KI	1733	81.46±11.54	140.97±51.49	16.43±6.24	63.42±28.19	32.16±4.45
43	20.31	1-Decanol	1694.06	MS,KI,ST	1720	-	-	13.67±1.28	18.99±3.79	28.34±3.16
44	20.8	Methoxy-phenyl-oxime	1719.38	MS,KI	1733	-	-	9.1±3.01	18.08±10.93	19.99±5.81

Table A-11 Continued

SL NO	RT	Compound	Measured RI	ID	Library RI	0 Days	5 Days	10 Days	15 Days	20 Days
45	20.94	(E,E)-2,4-Decadienal	1726.61	MS,KI	n/a	-	-	12.16±2.94	10.83±2.03	32.01±13.63
46	21.47	4-Oxononanal	1754.01	MS,KI	1749	-	-	-	-	9.97±3.02
47	21.68	(E)-Carveol	1764.86	MS,KI,ST	1760	-	-	11.47±6.44	-	-
48	22.06	Geranylacetone	1784.50	MS,KI,ST	1750	12.54±4.72	20.72±3.75	16.85±2.62	21.72±2.35	-
49	22.32	Phenylethyl alcohol	1797.93	MS,KI	n/a	31.27±9.1	91.13±6.84	-	-	-
50	22.5	trans-Isolimonene	1807.24	MS,KI	1773	-	-	-	-	-
52	23.05	α -Calacorene	1835.66	MS,KI	1819	-	-	-	-	-
53	23.5	β -Ionone	1858.91	MS,KI,ST	n/a	17.77±8.07	40.55±13.64	-	-	-
54	23.66	Benzothiazole	1867.18	MS,KI	1858	-	-	-	12±2.63	-
55	23.7	Heptanoic acid	1866.67	MS,KI	1867	-	-	-	-	3.71±1.01
56	24.5	β -Ionol	1906.47	MS,KI,ST	n/a	10.99±2.11	80.28±15.93	-	-	-
57	24.55	1-Dodecanol	1908.96	MS,KI	1880	-	-	21.91±8.72	45.61±7.64	20.42±5.02
58	24.6	β -Ionone epoxide	1911.44	MS,KI	1915	-	-	-	-	43.77±10.17
59	24.94	Cis-4,5-Epoxy-(E)-2-decenal	1928.36	MS,KI	n/a	13.88±1.29	47.93±2.55	-	-	-
60	24.95	Trans-4,5-Epoxy-(E)-2-decenal	1928.86	MS,KI	n/a	-	-	-	-	-
61	25.37	3-Phenylpropanol	1949.75	MS,KI	1947	-	-	-	-	-
62	26.17	5-Pentyl-2(5H)-furanone	1989.55	MS,KI	1995	4.17±0.74	10.33±1.72	17.28±6.41	-	-
63	26.29	Globulol	1995.52	MS,KI	1968	-	-	-	-	-
64	26.5	Elemol	2005.97	MS,KI	2058	-	-	-	-	-
65	26.51	1-Tridecanol	2006.47	MS,KI	2068	-	-	12.93±2.78	16.73±4.8	-
66	27.2	Z3,Z6,Z8-Dodecatrien-1-ol	2044.20	MS,KI	2099	-	-	-	-	-
67	28.35	Nonanoic acid	2106.20	MS,KI	2073	-	-	-	-	-

Table A-11 Continued

SL NO	RT	Compound	Measured RI	ID	Library RI	0 Days	5 Days	10 Days	15 Days	20 Days
68	28.6	δ -Cadinol	2119.68	MS,KI	n/a	3.47 \pm 0.38	8.79 \pm 1.43	6.38 \pm 1.96	7.72 \pm 2.58	77.38 \pm 25.95
69	29.11	α -Cadinol	2147.17	MS,KI	n/a	15.31 \pm 4.79	12.15 \pm 2.48	-	-	-
70	29.5	Methyl hexadecanoate	2168.19	MS,KI	2167	-	-	-	-	-
71	29.81	Ethyl hexadecanoate	2184.91	MS,KI	2179	13.9 \pm 2.48	13.1 \pm 3.28	5.79 \pm 0.56	10.27 \pm 2.08	11.38 \pm 1.81
72	30.23	Ethyl 9-hexadecenoate	2207.55	MS,KI	2193	12.96 \pm 5.66	13.31 \pm 4.01	2.12 \pm 0.3	9.98 \pm 4.09	5.87 \pm 0.27
73	31.24	Farnesyl acetone	2261.99	MS,KI,ST	2259	4.65 \pm 0.71	3.36 \pm 0.72	-	-	-
74	31.7	2,3-Dihydrobenzofuran	2286.79	MS,KI	n/a	-	-	-	-	-
75	32.18	4-Quinolincarboxaldehyde	2321.51	MS,KI	2288	-	-	-	-	-
76	32.5	1-Hexadecanol	2340.12	MS,tn	n/a	5.25 \pm 1.31	6.03 \pm 1.05	4.65 \pm 0.52	10.38 \pm 3.56	5.12 \pm 1.05

Table A-12. Volatile compound contents of HD252 Honeydew variety represented as mean \pm SE

SL NO	RT	Compound	Measured RI	ID	Library RI	0 Days	5 Days	10 Days	15 Days	20 Days
1	4.5	Hexanal	1046.96	MS,KI	1080	-	24.83 \pm 20.74	6.18 \pm 1.55	16.82 \pm 11.24	42.41 \pm 6.6
2	6.5	Heptanal	1130.82	MS,KI	1080	-	-	-	16.5 \pm 6.15	-
3	6.7	Limonene	1139.20	MS,KI,ST	1194	-	-	-	-	-
4	7.41	(E)-2-Hexanal	1168.97	MS,KI	1198	-	14.82 \pm 7.66	-	-	-
5	7.82	Styrene	1186.16	MS,KI	1212	-	-	12.78 \pm 6.7	16.88 \pm 0.73	-
6	7.97	2-Pentylfuran	1192.45	MS,KI	1240	18.29 \pm 4.11	12.54 \pm 3.17	20.88 \pm 3.85	16.24 \pm 3.52	21.78 \pm 2.99
7	8.8	Octanal	1227.25	MS,KI	1244	-	-	-	-	3.31 \pm 1.17
8	9.34	(E)-2-(2-Pentenyl)furan	1245.51	MS,KI	1261	-	-	-	6.75 \pm 0.44	2.78 \pm 0.43
9	9.81	(E)-2-Heptenal	1263.48	MS,KI	1278	6.16 \pm 4.73	16.95 \pm 9.99	8.89 \pm 3.65	10.15 \pm 3.04	9.28 \pm 2.91
10	10.24	3-Hexenyl acetate	1279.92	MS,KI	1289	-	-	-	-	-
11	10.28	6-Methylhept-5-en-2-one	1281.45	MS,KI,ST	n/a	-	-	2.94 \pm 0.36	6.28 \pm 1.74	11.25 \pm 3.82
12	10.6	Dimethyl trisulfide	1293.69	MS,KI,ST	1337	-	-	111.08 \pm 106.09	9 \pm 3.12	5.43 \pm 1.93
13	11.6	Nonanal	1331.93	MS,KI	1383	10.1 \pm 3.57	39.21 \pm 18	8.98 \pm 1.79	19.3 \pm 0.38	9.38 \pm 0.81
14	11.92	1-Octen-3-ol	1344.17	MS,KI,ST	1396	8.16 \pm 5.79	-	36.48 \pm 26.44	14.01 \pm 4.01	-
15	12.4	(E)-2-Octenal	1362.52	MS,KI	1435	7.03 \pm 4.29	-	239.85 \pm 220	29.83 \pm 20.55	33.47 \pm 15.87
16	12.74	3,7-Dimethyloctan-3-ol	1375.53	MS,KI	1432	-	-	8.65 \pm 1.04	17 \pm 3.78	17.11 \pm 9.74
17	12.93	(Z)-6-Nonenal	1382.79	MS,KI	1438	31.74 \pm 23.21	-	-	-	-
18	13.14	(E,Z)-2,4-Heptadienal	1390.82	MS,KI	n/a	-	-	-	43.1 \pm 16.35	77.51 \pm 5.65
19	13.28	(E,E)-2,4-Heptadienal	1396.18	MS,KI	1453	6.62 \pm 3.91	23 \pm 10.89	26.45 \pm 4.8	-	-
20	13.3	Acetic acid	1396.94	MS,KI	1452	-	-	15.78 \pm 11.18	30.29 \pm 12.73	33.16 \pm 13.47
21	14.12	Decanal	1428.30	MS,KI,ST	1455	-	-	-	51.8 \pm 17.65	54.82 \pm 18.82
22	14.5	(E,Z)-3,5-Octadien-2-one	1442.83	MS,KI	1456	-	-	-	-	67.46 \pm 30.49

Table A-12 Continued

SL NO	RT	Compound	Measured RI	ID	Library RI	0 Days	5 Days	10 Days	15 Days	20 Days
24	14.89	(E)-2-Nonenal	1459.10	MS,KI,ST	1501	57.2±12.59	75.37±28.94	26.37±12.69	81.87±2.22	103.53±18.9
25	15.61	Ethyl 3-(methylthio)propionate	1487.28	MS,KI	1497	-	-	-	-	-
26	15.7	1-Octanol	1490.80	MS,KI	1506	-	-	-	-	-
27	16.07	(E,Z)-2,6-nonadienal	1505.28	MS,KI,ST	1530	124.47±4.6	53.68±19.92	12.7±5.92	35.54±27.75	7.98±1.03
28	16.3	2,4-Dimethylcyclohexanol	1514.29	MS,KI	1543	-	-	-	-	-
29	16.7	β-Cyclocitral	1529.94	MS,KI,ST	1539	9.46±3.63	18.58±10.06	19.8±12.21	59.94±37.25	12.05±2.69
30	17.04	(E)-2-Octen-1-ol	1543.25	MS,KI	1577	-	-	-	-	-
31	17.18	Pheynlacetalddehyde	1510.76	MS,KI	1596	-	-	8.85±3.06	7±2.33	155.85±55.6
32	17.36	(E)-2-Decenal	1524.07	MS,KI	n/a	-	-	-	-	-
33	17.43	Ethyl decanoate	1545.60	MS,KI,ST	1610	-	60.77±53.72	-	-	-
34	17.74	Ethyl benzoate	1535.03	MS,KI,ST	1623	-	-	-	-	-
35	18.1	1-Nonanol	1559.30	MS,KI	1640	-	-	-	-	-
36	18.58	(Z)-3-Nonen-1-ol	1576.13	MS,KI	1643	42.38±13.15	111.82±51.99	101.16±37.88	284.27±62.24	136.91±70.88
37	18.58	Diethyl butanedioate	1603.52	MS,KI	1647	-	-	-	-	-
38	18.72	α-Terpineol	1609.00	MS,KI	1658	58.07±14.75	171.95±16.21	138.4±46.2	173.48±101.14	209.28±27.19
39	19.3	(E)-Citral	1631.70	MS,KI	1681	-	-	-	-	-
40	19.31	(Z)-6-nonen-1-ol	1642.38	MS,KI	1682	-	170.6±38.93	38.31±15.4	78.05±38.58	119.74±48.95
41	19.78	(E)-2-Undecenal	1666.67	MS,KI	1688	-	-	-	-	-
42	20	(E,Z)-3,6-Nonadien-1-ol	1678.04	MS,KI	1733	64.02±28.93	66.95±33.66	52.75±17.64	86.04±33.05	60.47±37.58
43	20.31	1-Decanol	1694.06	MS,KI,ST	1720	-	-	-	-	-
44	20.8	Methoxy-phenyl-oxime	1719.38	MS,KI	1733	-	-	20.95±6.08	60.65±12.79	51.83±9.28

Table A-12 Continued

SL NO	RT	Compound	Measured RI	ID	Library RI	0 Days	5 Days	10 Days	15 Days	20 Days
45	20.94	(E,E)-2,4-Decadienal	1726.61	MS,KI	n/a	-	-	-	-	-
46	21.47	4-Oxononanal	1754.01	MS,KI	1749	-	-	-	-	-
47	21.68	(E)-Carveol	1764.86	MS,KI,ST	1760	-	-	8.27±0.67	18.8±6.16	54.12±41.54
48	22.06	Geranylacetone	1784.50	MS,KI,ST	1750	33.51±22.6	87.25±62.83	94.63±61.65	157.51±35.25	134.35±89.33
49	22.32	Phenylethyl alcohol	1797.93	MS,KI	n/a	6.24±1.31	-	15.36±8.93	29.21±22.76	10.45±0.76
50	22.5	trans-Isolimonene	1807.24	MS,KI	1773	-	-	-	-	-
52	23.05	α-Calacorene	1835.66	MS,KI	1819	4.89±1.38	-	-	-	-
53	23.5	β-Ionone	1858.91	MS,KI,ST	n/a	45.13±29.36	89.58±56.71	100.78±75.34	128.67±64.23	94.39±74.44
54	23.66	Benzothiazole	1867.18	MS,KI	1858	-	-	-	-	-
55	23.7	Heptanoic acid	1866.67	MS,KI	1867	-	-	-	-	-
56	24.5	β-Ionol	1906.47	MS,KI,ST	n/a	13.15±6.14	-	-	-	-
57	24.55	1-Dodecanol	1908.96	MS,KI	1880	-	-	17.88±5.9	146.95±70.47	41.38±4.27
58	24.6	β-Ionone epoxide	1911.44	MS,KI	1915	-	-	-	-	-
59	24.94	Cis-4,5-Epoxy-(E)-2-decenal	1928.36	MS,KI	n/a	-	-	-	-	-
60	24.95	Trans-4,5-Epoxy-(E)-2-decenal	1928.86	MS,KI	n/a	-	-	-	-	-
61	25.37	3-Phenylpropanol	1949.75	MS,KI	1947	-	-	-	-	-
62	26.17	5-Pentyl-2(5H)-furanone	1989.55	MS,KI	1995	2.76±0.34	6.88±2.76	4.36±0.64	10.19±2.84	14.05±1.64
63	26.29	Globulol	1995.52	MS,KI	1968	-	-	-	-	-
64	26.5	Elemol	2005.97	MS,KI	2058	-	-	-	-	-
65	26.51	1-Tridecanol	2006.47	MS,KI	2068	-	-	-	-	-
66	27.2	Z3,Z6,Z8-Dodecatrien-1-ol	2044.20	MS,KI	2099	-	-	-	-	-
67	28.35	Nonanoic acid	2106.20	MS,KI	2073	-	-	-	-	42.56±7.45

Table A-12 Continued

SL NO	RT	Compound	Measured RI	ID	Library RI	0 Days	5 Days	10 Days	15 Days	20 Days
68	28.6	δ -Cadinol	2119.68	MS,KI	n/a	4.4 \pm 2	12.51 \pm 6.34	7.91 \pm 2.37	8.76 \pm 5.01	-
69	29.11	α -Cadinol	2147.17	MS,KI	n/a	13.17 \pm 1.89	-	-	-	-
70	29.5	Methyl hexadecanoate	2168.19	MS,KI	2167	-	-	9.3 \pm 1.91	12.3 \pm 3.07	-
71	29.81	Ethyl hexadecanoate	2184.91	MS,KI	2179	12.73 \pm 1.55	14.93 \pm 2.54	12.49 \pm 4.17	24.66 \pm 6.84	23.69 \pm 11.8
72	30.23	Ethyl 9-hexadecenoate	2207.55	MS,KI	2193	30.45 \pm 7.25	22.5 \pm 3.06	13.44 \pm 4.53	22.25 \pm 6.16	17.85 \pm 5.71
73	31.24	Farnesyl acetone	2261.99	MS,KI,ST	2259	6.24 \pm 1.16	8.6 \pm 2.45	11.19 \pm 4.6	16.63 \pm 4.81	24.08 \pm 5.35
74	31.7	2,3-Dihydrobenzofuran	2286.79	MS,KI	n/a	-	-	-	-	-
75	32.18	4-Quinolinecarboxaldehyde	2321.51	MS,KI	2288	-	-	-	-	-
76	32.5	1-Hexadecanol	2340.12	MS,tn	n/a	4.06 \pm 0.95	3.13 \pm 0.4	19.05 \pm 4.45	12.09 \pm 4.23	-

Table A-13. Identifiers of the commonly shared metabolites, which includes CAS, PubChem CID, ChEBI, KEGG, METLIN IDs, Measured RIs, and Library RIs. NA indicates metabolites without assigned DB identifiers.

SL NO	RT	Compound	Formula	CAS	Pubchem CID	ChEBI	KEGG	Metlin	Measured RI	Library RI
1	4	Ethyl 2-methylbutanoate	C7H14O2	7452-79-1	24020	N/A	N/A	N/A	1144.19	893
2	4.5	Hexanal	C6H12O	66-25-1	6184	17998	C02373	N/A	1046.96	0
3	6.5	Heptanal	C7H14O	111-71-7	8130	34787	C14390	N/A	1130.82	1018
4	6.77	Limonene	C10H16	5989-27-5	22311	15383	C00521	6911	1142.14	1040
5	6.8	1,8-Cineole	C10H18O	470-82-6	12031	27961	C09844	N/A	1143.40	1073
6	7.77	2-Pentylfuran	C9H14O	3777-69-3	19602	89197	N/A	N/A	1184.07	1075
7	7.83	2-Methylbutanol	C5H12O	137-32-6	8723	48945	N/A	N/A	1186.58	1077
8	8	Styrene	C8H8	100-42-5	7501	27452	N/A	N/A	1193.71	1080
9	8.2	Ethyl hexanoate	C8H16O2	123-66-0	31265	86055	N/A	N/A	1402.10	0
10	8.8	Octanal	C8H16O	124-13-0	454	17935	C01545	6033	1427.25	1123
11	9.22	2,2,6-Trimethylcyclohexan-1-one	C9H16O	2408-37-9	17000	N/A	N/A	N/A	1444.86	1132
12	9.34	(E)-2-(2-Pentenyl)furan	C9H12O	70424-14-5	5370006	N/A	N/A	N/A	1449.90	1138
13	9.82	cis-4,5-Epoxy-(E)-2-decenal	C7H12O	18829-55-5	N/A	N/A	N/A	N/A	1470.02	0
14	10.24	3-Hexenyl acetate	C8H14O2	1708-82-3	5352557	61316	C19757	N/A	1487.63	1152
15	10.27	6-Methylhept-5-en-2-one	C8 H14 O	110-93-0	9862	16310	C07287	N/A	1488.89	0
16	10.56	Dimethyl trisulfide	C2H6S3	3658-80-8	19310	4614	C08372	N/A	1501.05	1180
17	11.4	Nonanal	C9H18O	124-19-6	31289	84268	N/A	N/A	1536.27	1194
18	12.3	(E)-2-Octenal	C8H14O	2548-87-0	16900	61725	N/A	N/A	1574.00	0
19	12.67	Ethyl caprylate	C10H20O2	106-32-1	7799	87426	C12292	N/A	1589.52	1194
20	12.7	(Z)-6-Nonenal	C9H16O	2277-19-2	5283338	N/A	N/A	N/A	1590.78	0
21	12.71	Ethyl 2-(methylthio)acetate	C5H10O2S	4455-13-4	78199	47870	C03173	N/A	1591.19	1198
22	12.84	3,7-Dimethyloctan-3-ol	C10H22O	78-69-3	6548	84242	N/A	N/A	1596.65	1212

Table A-13 Continued

SL NO	RT	Compound	Formula	CAS	Pubchem CID	ChEBI	KEGG	Metlin	Measured RI	Library RI
23	13.2	1-Octen-3-ol	C8H16O	3391-86-4	18827	34118	C14272	N/A	1611.74	1201
24	13.4	(E,Z)-2,4-Heptadienal	C7H10O	4313-02-4	11788274	N/A	N/A	N/A	1820.13	1225
25	13.6	Ethyl 2,4-hexadienoate	C8H12O2	2396-84-1	1550470	72819	N/A	N/A	1828.51	1240
26	14.1	Decanal	C10H20O	112-31-2	582698	31457	C12307	N/A	1849.48	1244
27	14.35	Benzaldehyde	C7H6O	100-52-7	3559	17169	C00261	N/A	1637.09	0
28	14.5	(E,Z)-3,5-Octadien-2-one	C8H12O	4173-41-5	5352876	N/A	N/A	N/A	1642.83	1254
29	14.74	(E)-2-Nonenal	C9H16O	18829-56-6	5283335	142592	N/A	N/A	1652.01	1261
30	15.61	Ethyl 3-(methylthio)propionate	C6H12O2S	13327-56-5	61592	87503	N/A	N/A	1685.28	0
31	15.69	(E,E)-3,5-Octadien-2-one	C8H12O	38284-27-4	181575	N/A	N/A	N/A	1688.34	1269
32	15.7	1-Octanol	C8H18O	111-87-5	4018	16188	C00756	6063	1688.72	0
33	15.98	(E,Z)-2,6-nonadienal	C9 H14 O	557-48-2	11196	7610	N/A	N/A	1699.43	1289
34	16.37	2,4-Dimethylcyclohexanol	C8H16O	69542-91-2	98251	88852	N/A	N/A	1714.34	0
35	16.66	β -Cyclocitral	C10H16O	432-25-7	9895	53177	N/A	N/A	1725.43	1282
36	17.04	(E)-2-Octen-1-ol	C8H16O	18409-17-1	29060	142616	N/A	N/A	1739.96	1278
37	17.22	Phenylacetaldehyde	C8H8O	122-78-1	998	16424	N/A	N/A	1746.85	1290
38	17.36	(E)-2-Decenal	C10H18O	3913-81-3	5283345	133455	N/A	N/A	1752.20	0
39	17.38	Ethyl decanoate	C12H24O2	110-38-3	8048	87430	N/A	N/A	1752.96	1321
40	17.75	Ethyl benzoate	C9H10O2	93-89-0	7165	32807	N/A	N/A	1767.11	1318
41	18.08	(Z)-Citral	C10 H16 O	106-26-3	643779	29020	C09847	N/A	1779.73	1331
42	18.1	1-Nonanol	C9H20O	143-08-8	17395695	35986	C14696	N/A	1780.50	1337
43	18.5	(Z)-3-Nonen-1-ol	C9H18O	10340-23-5	5364631	N/A	N/A	N/A	1995.79	0
44	18.58	Diethyl butanedioate	C8H14O4	123-25-1	31249	N/A	N/A	N/A	1998.85	1354

Table A-13 Continued

SL NO	RT	Compound	Formula	CAS	Pubchem CID	ChEBI	KEGG	Metlin	Measured RI	Library RI
45	18.73	alpha-Terpineol	C10H18O	98-55-5	442501	22469	C16772	N/A	2004.59	1383
46	19.31	(Z)-6-nonen-1-ol	C9 H18 O	35854-86-5	142603	142603	N/A	N/A	2026.77	1378
47	19.32	(E)-Citral	C10H16O	141-27-5	4668	16980	C01499	N/A	2027.15	1390
48	19.34	Dodecanal	C12H24O	112-54-9	8194	27836	C02278	N/A	2027.92	1389
49	19.56	Ethyl 3-(methylthio)-(E)-2-propenoate	C6H10O2S	136115-65-6	5369325	87503	N/A	N/A	2036.33	1396
50	19.7	(E)-2-Undecenal	C11H20O	53448-07-0	5283356	132843	N/A	N/A	2041.68	1412
51	19.97	(E,Z)-3,6-Nonadien-1-ol	C9H16O	53046-97-2	6434541	N/A	N/A	N/A	2052.01	#N/A
52	20.3	1-Decanol	C10H22O	112-30-1	8174	28903	C01633	N/A	2064.63	1400
53	20.35	alpha-Farnesene	C15H24	502-61-4	5281516	10280	C09665	N/A	2066.54	1432
54	20.6	3-Phenylpropanal	C9H10O	104-53-0	7707	N/A	N/A	N/A	2076.10	1435
55	20.73	Methoxy-phenyl-oxime	C8H9NO2	N/A	9602988	N/A	N/A	N/A	2081.07	1438
56	20.98	(E,E)-2,4-Decadienal	C10 H16 O	25152-84-5	5283349	N/A	N/A	N/A	2090.63	0
57	21.5	4-Oxononanal	C9H16O2	74327-29-0	156288	N/A	N/A	N/A	2110.52	1452
58	21.6	(E)-Carveol	C10H16O	1197-07-5	94221	15389	C00964	N/A	2114.34	1453
59	21.77	(E)-alpha-Ionone	C13H20O	127-41-3	5282108	32319	C12286	N/A	2120.84	1455
60	22.05	Geranylacetone	C13H22O	689-67-8	1549778	67206	N/A	N/A	2331.55	1456
61	22.44	3-Hydroxy-2,4,4-trimethylpentyl 2-methylpropanoate	C12H24O3	N/A	551387	N/A	N/A	N/A	2154.60	1464
62	22.5	trans-Isolimonene	C10H16	6876-12-6	22831540	85183	N/A	N/A	2156.95	0
63	22.9	alpha-Calacorene	C15H20	21391-99-1	12302243	N/A	N/A	N/A	2172.60	1470
64	23	Phenylethyl alcohol	C8H10O	60-12-8	6054	49000	C05853	N/A	2176.52	0
65	23.3	2-Phenyl-2-butenal	C10H10O	4411-89-6	6429333	89904	N/A	N/A	2188.26	1497
66	23.5	beta-Ionone	C13H20O	14901-07-6	638014	32325	C12287	N/A	2196.09	1501

Table A-13 Continued

SL NO	RT	Compound	Formula	CAS	Pubchem CID	ChEBI	KEGG	Metlin	Measured RI	Library RI
67	23.66	Benzothiazole	C7H5NS	95-16-9	7222	45993	N/A	N/A	2202.35	0
68	23.7	Heptanoic acid	C7H14O2	111-14-8	8094	45571	C17714	5636	2203.91	1501
69	24.5	beta-Ionol	C13H22O	22029-76-1	5373729	32325	N/A	N/A	2235.23	0
70	24.53	1-Dodecanol	C12H26O	112-53-8	8193	28878	C02277	N/A	2236.40	1492
71	24.6	β -Ionone epoxide	C13H20O2	23267-57-4	5352481	87546	N/A	N/A	2239.14	1506
72	24.63	β -Ionol	C13H22O	22029-76-1	5373729	N/A	N/A	N/A	2240.31	1530
73	24.95	Trans-4,5-Epoxy-(E)-2-decenal	C10H16O2	134454-31-2	5352429	N/A	N/A	N/A	2252.84	1539
74	25.41	3-Phenylpropanol	C9H10O	122-97-4	7707	39940	N/A	N/A	2270.84	1540
75	26.1	5-Pentyl-2(5H)-furanone	C9H14O2	21963-26-8	N/A	N/A	N/A	N/A	2297.85	#N/A
76	26.48	Elemol	C15H26O	8024-27-9	92138	141221	C21698	N/A	2312.72	1543
77	26.5	Globulol	C15H26O	51371-47-2	101716	N/A	N/A	N/A	2513.50	1545
78	26.71	1-Tridecanol	C13H28O	112-70-9	31423	8207	C14509	N/A	2521.72	#N/A
79	27.2	Z3,Z6,Z8-Dodecatrien-1-ol	C12H20O	19926-64-8	6442192	N/A	N/A	N/A	2540.90	1553
80	27.36	Hexadecanal	C16H32O	629-80-1	984	17600	C06123	N/A	2547.16	1561
81	28	Eugenol	C10H12O2	97-53-0	3314	4917	C10453	4022	2572.21	#N/A
82	28.35	Nonanoic acid	C9H18O2	112-05-0	8158	29019	C01601	5810	2585.91	1573
83	28.6	δ -Cadinol	C15H26O	36564-42-8	3084311	132905	N/A	N/A	2595.69	1577
84	28.8	alpha-Eudesmol	C15H26O	473-16-5	92762	10278	C09663	N/A	2603.52	1578
85	29.12	α -Cadinol	C15H26O	481-34-5	6431302	132905	N/A	N/A	2616.05	1576
86	29.13	Methyl hexadecanoate	C17H34O2	112-39-0	8181	69187	C16995	N/A	2616.44	1596
87	29.8	Ethyl hexadecenoate	C18H36O2	628-97-7	12366	84932	N/A	N/A	2642.66	1563
88	30.2	Ethyl 9-hexadecenoate	C18H34O2	56219-10-4	5364759	84934	N/A	N/A	2658.32	0

Table A-13 Continued

SL NO	RT	Compound	Formula	CAS	Pubchem CID	ChEBI	KEGG	Metlin	Measured RI	Library RI
89	30.5	Dihydroactinidiolide	C11H16O2	15356-74-8	27209	na	N/A	N/A	2670.06	1588
90	31.2	Farnesyl acetone	C18H30O	1117-52-8	1711945	67252	N/A	N/A	2697.46	1616
91	31.7	4-Quinolinecarboxaldehyde	C10H7NO	4363-93-3	78072	51934	N/A	N/A	2717.03	1623
92	32	1-Hexadecanol	C16H34O	36653-82-4	2682	16125	C00823	N/A	2728.77	1626