Evaluation of Physicochemical and Storability Attributes of Muscadine Grapes (*Vitis rotundifolia* Michx.)

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Abstract. Muscadine grapes (*Vitis rotundifolia* Michx.) are native to the southeastern United States and have potential for greater fresh-market sales if postharvest storage can be improved, but limited information is available on postharvest storability. In 2012 and 2013, physiochemical and storability attributes were measured in 17 muscadine genotypes (selections and cultivars) from the muscadine breeding program at the University of Arkansas or commercial cultivars. The postharvest and physiochemical attributes of the muscadines were measured at harvest and during storage for 3 weeks at 2 °C. Nutraceutical compounds were measured initially after harvest. As a result of extreme differences in weather in 2012 and 2013, the data were analyzed by year. Genotypes significantly affected storage attributes [weight loss (%), and unmarketable berries (%)] and physiochemical attributes such as penetration force (force to penetrate berry skin), titratable acidity (TA), pH, soluble solids (%), berry color (L*, chroma, and hue) as well as the nutraceutical compounds. The postharvest attributes of weight loss and unmarketable berries and the physiochemical attribute of penetration force were significantly affected by postharvest storage, but berry composition attributes remained fairly constant during storage. Overall, University of Arkansas selections AM 04, AM 26, AM 28, and the cultivar Southern Jewel had the highest potential for postharvest storage, whereas the genotypes AM 01, AM 15, AM 18, and ‘Nesbitt’ had the least potential. Genotypes AM 03, AM 04, AM 27, and ‘Ison’ had the highest nutraceutical contents [total anthocyanins, total phenolics, total flavonols, resveratrol, and oxygen radical absorbance capacity (ORAC)], whereas AM 18, AM 28, ‘Supreme’, and ‘Tara’ had the lowest contents. Postharvest storage potential, berry composition, berry color, and nutraceutical content were genotype-specific, but commercially viable genotypes were identified that can provide genetic material for breeding programs and postharvest evaluation protocol for commercial use.

Muscadine grapes (*Vitis rotundifolia* Michx.) are relatively insect- and disease-resistant native plants and are commonly grown in the southeastern United States to diversify farm operations (Conner, 2009; Silva et al., 1994; Striegler et al., 2005; Walker et al., 2001). Muscadine berries have a unique flavor but are often thick-skinned and vary in color, shape, and size. The reported high nutraceutical content of muscadine grapes and products from muscadine grapes has increased consumer demand (Perkins-Veazie et al., 2012; Striegler et al., 2005). Major limiting factors of fresh-market production of muscadines include uneven ripening, short harvest season, fruit softening, seediness, and high perishability of the fruit during postharvest storage (James et al., 1999; Morris, 1980;

Perkins-Veazie et al., 2012). Both public and private muscadine breeding programs are addressing these limiting factors through the development and evaluation of muscadine genotypes (selections and cultivars).

Muscadine maturity and type and percentage of dry/wet stem scars have been shown to impact texture (firmness/crispness), weight loss, decay, shriveling, browning, and leakage during storage (Ballinger and Nesbitt, 1982a, 1982b; Conner and Maclean, 2012; James et al., 1997, 1999; Lane, 1978; Lane and Flora, 1980; Smit et al., 1971). Muscadines can be successfully stored for 2 to 3 weeks (Perkins-Veazie et al., 2012; Takeda et al., 1982) under recommended conditions of 1 to 5 °C with 85% to 95% relative humidity (RH) (Silva et al., 1994; Takeda et al., 1983; Walker et al., 2001).

Muscadine grapes have one of the highest nutraceutical levels among fruit crops, but levels vary among genotypes (Greenspan et al., 2005; Marshall et al., 2012). Polyphenol concentrations usually increase in muscadines

as fruit ripens (Lee et al., 2005) and are higher in wine than in unfermented juices (Musungu et al., 2001; Talcott and Lee, 2002). Muscadine grapes contain nutraceutical compounds such as phenolic acids, flavonols, anthocyanins, ellagic acid, resveratrol, and numerous ellagic-acid derivatives (Boyle and Hsu, 1990; Haung et al., 2009; Lee et al., 2005; Pastrana-Bonilla et al., 2003; Stringer et al., 2009; Talcott and Lee, 2002). The nutraceutical compounds in muscadines have demonstrated anticarcinogenic (Ector, 2001; Yi et al., 2005) and anti-inflammatory (Greenspan et al., 2005) activities and have also been shown to reduce levels of glucose, insulin, and glycated hemoglobin in people with diabetes (Banini et al., 2006).

The University of Arkansas muscadine breeding program was implemented in 2005 with the goal to improve fresh-market muscadine potential by advancing selections through traditional breeding efforts based on flower type, fruit size, time of ripening, winter-hardiness, and field evaluations. There is limited information on the physiochemical attributes of the University of Arkansas genotypes. The objective of this study was to expand on the work of Barchenger et al. (2014) and evaluate postharvest storage performance, physicochemical attributes, and initial nutraceutical concentrations of a wide range of muscadine genotypes including commercial cultivars and breeding selections to provide input for breeding programs in the development of new cultivars for commercial use.

Materials and Methods

Experimental design

In 2012 and 2013, postharvest attributes, physiochemical attributes, and nutraceutical compounds were measured in 17 muscadine genotypes. The postharvest and physiochemical attributes were measured initially (immediately after harvest) and during storage weekly for 3 weeks at 2 °C and the study was designed as a split plot with three replications of each genotype with the split as storage (Weeks 0, 1, 2, and 3). The nutraceutical compounds were only measured initially (immediately after harvest) as a complete randomized design with three replications of each genotype. As a result of differences in year, likely the result of extreme differences in weather, the data were analyzed separately for each year of the study. A single vine was used as an experimental unit.

Vineyard

Muscadines were harvested from vines grown at the University of Arkansas Fruit Research Station, Clarksville, AR (lat. 35°31'58" N, long. 93°24'12" W), in Linker fine sandy loam, in U.S. Department of Agriculture hardiness zone 7a, where average annual minimum temperature reached –15 °C. Vines were of varying ages between each genotype; most of the cultivars were ≈6 to 7 years old, whereas many of the advanced selections were 3 to 4 years old. Vines were spaced 6.1 m apart with rows spaced 3.0 m

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apart. Vines were trained to a bilateral cordon on a single-wire trellis. The vines were dormant-pruned annually in February using spur pruning with spurs retained of two to four buds in length. Weeds were controlled with pre- and post-emergence herbicides as needed, and vines did not have any stress from weed competition. Vines were drip-irrigated as needed. Vines were fertilized with nitrogen annually in March at $\approx 70 \text{ kg} \cdot \text{ha}^{-1}$. No insecticides, fungicides, or other pest control compounds were applied to the vines. The vines produced full crops during the study, and there was no crop reduction as a result of winter injury or other limitations. Daily maximum and minimum temperatures and rainfall were recorded (data not shown) (Barchenger et al., 2014).

Genotypes, harvest, and handling

In this study, 17 muscadine genotypes (cultivars and selections) were evaluated. The black muscadine cultivars were Delicious, Ison, Nesbitt, Southern Jewel, and Supreme, and the black selections were AM 02, AM 04, AM 18, AM 27, and AM 28. The bronze muscadine cultivars were Fry, Summit, and Tara, and the bronze selections were AM 01, AM 03, AM 15, and AM 26. The muscadines were once-over hand-harvested. Harvest date/maturity was based on soluble solids of 18% to 22% in 2012 and 15% to 18% in 2013 (as a result of differences in summer temperature and precipitation), ease of release from the pedicel, and berry color. The muscadines were transported to the University of Arkansas Institute of Food Science and Engineering, Fayetteville, AR, on the same day of harvest and placed in cold storage (2 °C) on arrival.

The muscadine berries were hand-sorted to remove any split, shriveled, or decayed fruit. Like with a commercial product, only sound, marketable fruit were used. The berries were placed into hinged standard vented clamshells (18.4 cm $\times$ 12.1 cm $\times$ 8.9 cm) (H116; FormTex Plastics Corporation, Houston, TX) and stored at 2 °C with 90% RH. From the harvested berries, six clamshell containers were filled to $\approx 500 \text{ g}$ (three replications each for postharvest and physicochemical attributes). For nutraceutical measurements three berries in triplicate per genotype were selected, placed in plastic bags, and stored at -20 °C until analysis.

Postharvest storage

Postharvest attributes including percent weight loss and percent unmarketable berries were measured at harvest and during storage. Total clamshell weight was determined at the date of harvest, and percent weight loss was calculated as percent weight decrease from this initial value. Postharvest storage performance was evaluated by removing all the fruit from each clamshell and counting the number of fruit that showed signs of unmarketability, which included individual or a combination of characteristics of browning, softness, mold, rot, leakage, splitting, and shriveling (Conner, 2013; Conner and Maclean, 2012; Perkins-Veczise

et al., 2012). Both the unmarketable and marketable berries were returned to the appropriate clamshell each week, and storage measurements were discontinued once the percent unmarketable in all three clamshells reached 50% or greater or after 3 weeks of storage.

Physicochemical analysis

For physicochemical measurements, three berries removed from each of the three clamshells were used to measure exterior berry color (chroma, hue, L^*), soluble solids (%), TA, pH, and penetration force of the skin and flesh. The physicochemical procedures used were modeled from previously reported protocols (Conner, 2013; Conner and Maclean, 2012; Striegler et al., 2005; Threlfall et al., 2007; Walker et al., 2001). Physicochemical measurements were discontinued once the percent unmarketable berries in all three clamshells reached 50% or greater or after 3 weeks of storage.

Exterior skin color measurements were determined on each of the three berries every 7 d using a Chroma Meter CR 300 series (Konica Minolta Holdings Inc., Ramsey, NJ). The Commission Internationale de l'Eclairage Laboratory transmission " L^* " value indicates how dark or light the skin is with 0 being black and 100 being white. Hue angle describes color in angles from 0° to 360°: 0° = red; 90° = yellow; 180° = green; 270° = blue; and 360° = back to red. Chroma is the aspect of color by which the skin colors appear different from gray of the same lightness and corresponds to intensity of the perceived color.

Penetration force, or the maximum force to penetrate skin and flesh tissues, was determined using three whole berries per replication. A TA-XT2 Texture Analyzer (Stable Micro Systems, Haslemere, U.K.) with a 2-mm-diameter cylinder probe was used to penetrate the skin and mesocarp tissues (flesh) to a depth of 10 mm in each berry at a rate of $10 \text{ mm} \cdot \text{s}^{-1}$. Measurements are expressed as force in Newtons (N), and the data were analyzed using Texture Expert Version 1.17 (Texture Technologies Corp., Scarsdale, NY).

For soluble solids (%), pH, and TA, the berries were mashed and the berry juice was strained through cheesecloth to remove any solids. Titratable acidity and pH were measured by an 877 Titrino Plus (Metrohm AG, Herisau, Switzerland) with an automated titrimer and electrode standardized to pH 2.0, 4.0, 7.0, and 10.0 buffers. Titratable acidity was determined by titrating 0.1 N sodium hydroxide to 6 g of juice diluted with 50 mL of deionized, degassed water to an endpoint of pH 8.2, with results expressed as percent tartaric acid. Soluble solids were measured using a Bausch and Lomb Inc. Abbe Mark II refractometer (Rochester, NY).

Nutraceutical analysis

Three whole berries per replication were used for nutraceutical analyses. Samples were homogenized using a Euro Turrax T18

Tissuemizer (Tekmar-Dohrman Corp., Mason, OH) for 1 min with alternating washes of extraction solution containing methanol/water/formic acid (60:37:3 v/v/v) and acetone/water/acetic (70:29.5:0.5 v/v/v). Homogenates were centrifuged for 5 min at 10,000 rpm and filtered through Miracloth (CalBiochem, La Jolla, CA). The samples were adjusted to a final volume with extraction solvent and stored at -70 °C until further analysis. Before high-performance liquid chromatography (HPLC) analysis, samples were filtered through 0.45- μm filters (Whatman PLC, Maidstone, U.K.).

Total phenolics analysis. Total phenolics were measured using the Folin-Ciocalteu assay (Slinkard and Singleton, 1977) on a diode array spectrophotometer (8452A; Hewlett Packard, Palo Alto, CA) with a gallic acid standard and a consistent standard curve based on sequential dilutions. Samples were prepared with 1 mL 0.2 N Folin's reagent, 0.8 mL Na_2CO_3 (75 $\text{g} \cdot \text{L}^{-1}$), and 0.2 mL of extracted sample with a reaction time of 2 h. Absorbance was measured at 760 nm with results expressed as milligrams of gallic acid equivalents per 100 g fresh weight.

Anthocyanin, ellagitannin, flavonol, and resveratrol analysis. For anthocyanin analysis, subsamples (5 mL) of extract supernatant were evaporated to dryness using a SpeedVac® concentrator (ThermoSavant, Holbrook, NY) with no radiant heat applied and suspended in 1 mL of aqueous 3% formic acid solution. Samples (1 mL) were analyzed using a Waters HPLC system equipped with a Model 600 pump, a Model 717 Plus autosampler, and a model 996 photodiode array detector. Separation of anthocyanins was carried out on a 4.6 mm $\times$ 250-mm Symmetry® C18 column (Waters Corp., Milford, MA) preceded by a 3.9 mm $\times$ 20-mm Symmetry® C18 guard column using the conditions of Cho et al. (2004). The mobile phase was a linear gradient of 5% formic acid (A) and methanol (B) from 2% to 60% B for 60 min at $1 \text{ mL} \cdot \text{min}^{-1}$. Before each injection, the system was equilibrated for 20 min at the initial gradient. Detection wavelength was 510 nm for anthocyanins. Individual anthocyanin diglycosides were quantified as cyanidin, delphinidin, petunidin, peonidin, and malvidin glycoside equivalents using external calibration curves containing a mixture of authentic anthocyanin glucoside standards (Polyphenols, Sandnes, Norway). Total anthocyanins were calculated as the sum of individual glycosides with results expressed as milligrams per 100 g^{-1} fresh weight.

For total flavonol, total ellagitannin, and resveratrol analysis, samples (5 mL) of extract supernatant were evaporated to dryness using a SpeedVac® concentrator with no radiant heat applied and suspended in 1 mL of aqueous 50% methanol solution. The samples were analyzed using the same HPLC system described previously. Separation was carried out using a 4.6 mm $\times$ 250-mm Aqua® C18 column (Phenomenex, Torrance, CA) preceded by a 3.0 mm $\times$ 4.0-mm ODS® C18 guard column (Phenomenex) using the

conditions described by Cho et al. (2005) and Hager et al. (2008). The mobile phase was a gradient of 2% acetic acid (A) and 50% acetonitrile containing 0.5% acetic acid (B) from 10% B to 55% B in 50 min and from 55% B to 100% B from 50 to 60 min. Before each injection, the system was equilibrated for 20 min at the initial gradient. A detection wavelength of 360 nm was used for flavonols, 280 nm for ellagitannins, and 220 nm for resveratrol at a flow rate of 1 mL·min⁻¹. Flavonols and ellagitannins were expressed as milligrams of rutin equivalents 100 g fresh weight and milligrams of ellagic acid equivalents 100 g fresh weight, respectively. Resveratrol {3,4',5-Trihydroxy-*trans*-stilbene, 5-[(1*E*)-2-(4-Hydroxyphenyl)ethenyl]-1,3-benzenediol} was quantified using external calibration curves of an analytical standard (Sigma-Aldrich Co. LLC, St. Louis, MO) with results expressed as milligrams 100 g fresh weight.

High-performance liquid chromatography/mass spectrometry. An extract from representative black and bronze genotypes was analyzed using HPLC/mass spectrometry (MS) for flavonol and ellagitannin confirmation following the procedures of Cho et al. (2005) and Hager et al. (2008). For HPLC/MS analysis, the HPLC apparatus was interfaced to a Bruker Esquire (Bruker Corporation, Billerica, MA) liquid chromatography/MS ion trap mass spectrometer. Mass spectral data were collected with the Bruker software, which also controlled the instrument and collected the signal at 280 nm for ellagitannins or 360 nm for flavonols. Typical conditions for mass spectral analysis in negative ion electrospray mode included a capillary voltage of 4000 V, a nebulizing pressure of 30.0 psi, a drying gas flow of 9.0 mL·min⁻¹ and a temperature of 300 °C. Data were collected in full-scan mode over a mass range of *m/z* 50 to 1000 at 1.0 s/cycle. Characteristic ions were used for peak assignment with results compared with previous mass to charge (*m/z*) values reported for flavonols (Sandhu and Gu, 2010) and ellagic acid derivatives (Lee et al., 2005; Sandhu and Gu, 2010) in muscadines.

Oxygen radical absorbance capacity analysis. The ORAC of muscadine extracts was measured using the method of Prior et al. (2003) modified for use with a FLUOstar Optima microplate reader (BMG Labtechnologies, Durham, NC) using fluorescein as fluorescent probe. Muscadine extracts were diluted 1600-fold with phosphate buffer (75 mM, pH 7) before ORAC analysis. The assay was carried out in clear 48-well Falcon plates (VWR, St. Louis, MO), each well having a final volume of 590 µL. Initially, 40 µL of diluted sample, Trolox equivalents (TE) standards (6.25, 12.5, 25, 50 µM), and blank solution of phosphate buffer were added to each well. The FLUOstar Optima instrument equipped with two automated injectors was programmed to add 400 µL of fluorescein (0.108 µM) followed by 150 µL of 2,2'-azobis(2-amidino-propane) dihydrochloride (AAPH) (31.6 mM) to each well. Fluorescence readings (excitation 485 nm, emission 520 nm) were recorded after the addition of fluorescein and AAPH and every

192 s for 112 min to reach 95% loss of fluorescence. Results were based on differences in areas under the fluorescein decay curve among the blank, samples, and standards and expressed relative to the initial reading. The standard curve was obtained by plotting the four concentrations of Trolox against the net area under the curve of each standard. Final ORAC values were calculated using the regression equation between TE concentration and the net area under the curve with results expressed as µmol TE equivalents per gram fresh weight.

Statistical analysis

The data were analyzed by analysis of variance (ANOVA) using JMP® (Version 11.0; SAS Institute Inc., Cary, NC). Tukey's honestly significant difference was used for mean separations (*P* = 0.05). Associations among all dependent variables were determined using multivariate pairwise correlation coefficients of the mean values using JMP® (Version 11.0; SAS Institute Inc.).

Results and Discussion

Mean temperatures and precipitation varied greatly between 2012 and 2013 growing and harvest seasons with mean temperatures up to 5 °C warmer and half as much precipitation in 2012 compared with 2013 (data not shown). Muscadines in this study had average soluble solids of 18% to 22% in 2012 vs. 15% to 18% in 2013; thus, the data were analyzed by year as a result of the extreme environmental differences.

Postharvest storage analysis. In 2012 and 2013, the ANOVA *F*-test indicated significant two-way interactions of week of storage by genotype for percent weight loss (*P* < 0.0001) and percent unmarketable berries (*P* < 0.0001).

The performance of the genotypes varied by year. After 3 weeks of storage, the genotypes with the least percent weight loss in 2013 were AM 28, 'Southern Jewel', and 'Nesbitt' (2.2%, 1.9%, 2.0%, respectively), whereas in 2012, AM 27, AM 03, 'Delicious', and 'Tara' had the least (4.3%, 4.5%, 4.7%, and 4.7%, respectively) (Fig. 1). The genotypes with the greatest percent weight loss after 3 weeks of storage in 2013 were AM 03, AM 01, and 'Fry' (4.2%, 4.1%, and 4.1%, respectively), whereas in 2012, 'Nesbitt', AM 04, and AM 18 had the greatest weight loss (6.5%, 6.2%, and 5.9%, respectively) (Fig. 1). In 2013, the genotypes with the least percent unmarketable berries after 3 weeks of storage were AM 26, AM 28, AM 04, and AM 03 (8.9%, 11.8%, 12.6%, and 18.5%, respectively), whereas in 2012, AM 03, 'Summit', 'Southern Jewel', and 'Supreme' had the least percent unmarketable berries (15.3%, 20.7%, 23.2%, and 24.1%, respectively) (Fig. 2). The genotypes with the highest percent unmarketable berries were AM 01, 'Fry', and 'Tara' (94.9%, 73.9%, and 70.5%, respectively), whereas in 2012, the genotypes with the highest percent unmarketable berries were 'Fry',

AM 04, and AM 26 (65.8%, 64.8%, and 60.7%, respectively) (Fig. 2). This shows the impact of environmental factors (rainfall and temperature) on storage performance of muscadines and the importance of multiple year evaluations. Ballinger and Nesbitt (1982b) found 'Nesbitt' had acceptable post-harvest storage, James et al. (1997, 1999) found 'Summit' had the greatest percent unmarketable and weight loss, whereas 'Summit' performed moderately during storage in our study.

Unmarketability of muscadines was primarily the result of browning (especially in bronze genotypes), leakage from torn or wet stem scars, and shriveling, which was consistent with similar work reported by Perkins-Veazie et al. (2012). The browning of the bronze berries (especially AM 01 in 2013) was likely caused by chilling injury (CI). This abiotic disorder is common in many horticultural crops and can increase susceptibility to decay by providing media for the growth of pathogens (Wang, 1990). The primary symptom of CI identified in this study was brown discoloration of the skin, pulp, and vascular strands of fruit. Although CI has been reported in muscadines stored at 1.7 °C or below (Himelrick, 2003), CI is not usually observed in muscadine grapes stored at 2 to 3 °C. Leakage and shriveling were also common causes of unmarketability during storage but can be managed by removing berries with wet stem scars before storage and maintaining high RH during storage (Perkins-Veazie et al., 2012; Smit et al., 1971). In general, during storage for 3 weeks at 2 °C, the black genotypes had a 39% increase in unmarketable fruit, and the bronze genotypes had a 48% increase.

Physiochemical analysis. In 2012, the ANOVA *F*-test indicated significant two-way interactions of week of storage by genotype for force to penetrate berry skin (*P* < 0.0001), soluble solids (*P* = 0.0038), TA (*P* < 0.0001), and pH (*P* < 0.0001). Conversely, in 2013, the ANOVA *F*-test indicated significant two-way interactions of week of storage by genotype for force (*P* = 0.0008) and soluble solids (*P* < 0.0001) and the main effects of week and storage for TA (*P* < 0.0001) and the main effect of genotype for pH (*P* < 0.0001).

Force to penetrate muscadine skin has been shown to be a useful characteristic to assess berry firmness and texture as well as berry quality (Conner, 2013); however, use of force to determine storability of muscadine grapes has previously shown results with no clear trend (Silva et al., 1994; Walker et al., 2001). Muscadines require a force up to 13.9 N to penetrate the skin at date of harvest, which is nearly twice that of *V. vinifera* cultivars (Conner, 2013). Similarly, we found that 'Nesbitt' had among the highest penetration force, requiring up to 13.2 N to penetrate the skin at date of harvest in 2013. Berries stored in 2013 were generally firmer than in 2012 (Fig. 3). In 2013, penetration force ranged from 13.2 N ('Nesbitt' at Week 0) to 3.3 N ('Tara' at Week 3), whereas in 2012, penetration force ranged from 10.4 N

Fig. 1. Percent berry weight loss of muscadine genotypes stored at 2 °C for 3 weeks. Values at Week 0 (date of harvest) were excluded. Each SE bar is constructed using 1 SE from the mean (2012 and 2013). Bronze genotypes were AM 01, AM 03, AM 15, AM 26, 'Fry', 'Summit', and 'Tara' and the black genotypes were AM 02, AM 04, AM 18, AM 27, AM 28, 'Delicious', 'Ison', 'Nesbitt', 'Southern Jewel', and 'Supreme'.

Fig. 2. Percent unmarketable berries of muscadine genotypes stored at 2 °C for 3 weeks. Values at Week 0 (date of harvest) were excluded. Each SE bar is constructed using 1 SE from the mean (2012 and 2013). Bronze genotypes were AM 01, AM 03, AM 15, AM 26, 'Fry', 'Summit', and 'Tara' and the black genotypes were AM 02, AM 04, AM 18, AM 27, AM 28, 'Delicious', 'Ison', 'Nesbitt', 'Southern Jewel', and 'Supreme'.

(AM 28 at Week 0) to 1.8 N (Tara at Week 2) (Fig. 3). Percent unmarketable berries was negatively correlated with force ($r = -0.74$), potentially illustrating that berries requiring greater force to penetrate the berry skin store better because they were firmer and likely this is one of the more important relationships among variables measured to assist in evaluating a genotype's postharvest storage potential. Overall, berry penetration force decreased during storage but was occasionally lowest after 2 weeks of storage (Fig. 3),

similar to the findings of James et al. (1999), and this could possibly be the result of water loss during storage. It was found that the genotypes requiring the most force to penetrate the skin at date of harvest also required the most force to penetrate the skin after 3 weeks of storage (especially in 2013) (Fig. 3), indicating force is a good indicator of storage performance. In general, during storage for 3 weeks at 2 °C, the black genotypes had a 30% reduction in penetration force, and the bronze genotypes had a 36% reduction in penetration force.

Titrateable acidity, pH, and soluble solids remained relatively constant during storage (data not shown), which was consistent with the results of other studies (James et al., 1997, 1999; Takeda et al., 1983; Walker et al., 2001). Titrateable acidity and soluble solids were greater in 2012 compared with 2013, whereas pH was generally greater in 2013 (Table 1). This result contradicted Jackson (1986), who found that high pH was often associated with warmer temperatures during the growing season. The percent soluble solids were uncharacteristically higher in 2012, whereas TA was uncharacteristically low in 2013 (Table 1). In 2012, AM 04 had the highest percent TA (0.60%), whereas AM 03 and AM 18 had the lowest values (0.29% and 0.26%, respectively) (Table 1). In 2013, AM 01 and 'Delicious' had the highest TA (0.45%) and AM 28 had the lowest value (0.23%) (Table 1). Berry pH ranged from 3.25 ('Ison') to 3.83 (AM 04) in 2012 and from 3.40 (AM 15) to 3.96 (AM 02) in 2013 (Table 1).

In 2012, the ANOVA f-test indicated significant two-way interactions of week of storage by genotype for L* ($P < 0.0001$), hue ($P = 0.0092$), and chroma ($P = 0.0200$). Similarly, in 2013 the ANOVA f-test indicated significant two-way interactions of week of storage by genotype for L* ($P < 0.0001$) and chroma ($P < 0.0001$), whereas the main effects of week ($P = 0.0143$) and genotype ($P < 0.0001$) were significant for hue.

The effect of storage on the exterior berry color attributes (L* value, chroma, and hue angle) of muscadine grapes is widely unstudied. The U.S. Department of Agriculture (USDA) has no standards available to grade muscadine berries for L* value, chroma, and hue angle. The standards for exterior berry color of muscadines state the berries should be well-colored to be considered marketable; for black and red cultivars, 75% of the surface of the berry must have characteristic color for the variety, whereas no color requirement exists for bronze genotypes except that for 'Carlos', 'Fry', or similar cultivars, which can show any amount of blush or bronze color on the berry (USDA, 2006). Additionally the USDA states that black cultivar colors can include reddish purple, purple, and black; red cultivar colors include light pink, pink, red, dark red, and purple; and bronze cultivar colors include light green, straw, amber, and bronze with allowance for an amount of blush or pink color that may also be characteristic for certain cultivars (USDA, 2006).

L* values were generally greater for the bronze genotypes compared with the black genotypes and were often greater in 2013 compared with 2012 (Table 1). L* values ranged from 45.2 (AM 03) to 26.3 (AM 02) in 2012 and from 91.2 (AM 01) to 25.1 (AM 04) in 2013 (Table 1). There was a negative correlation between hue angle and L* value ($r = -0.80$), showing that as L* increased (berries became lighter), hue angle decreased. Hue angles were generally higher for the black genotypes compared with the

bronze genotypes and, similar to L*, were generally greater in 2013 compared with 2012 (Table 1). This difference in exterior color among years might be the result of less berry sunburn in 2012 as compared with 2013. A

positive correlation was observed among L* and soluble solids ($r = 0.71$), indicating L* could be used as a ripeness indicator.

In 2012, hue angles ranged from 359.4° ('Supreme') to 54.0° ('Summit'), whereas in

2013, hue angles ranged from 349.5° (AM 28) to 90.6° (AM 26) (Table 1). Conversely, Conner and MacLean (2013) found hue values that ranged from 1.5 to 91.8°, Threlfall et al. (2007) found values ranging from 53.4 to 98.6° and Walker et al. (2001) found values that ranged from 76.5 to 237.7°.

Walker et al. (2001) found that chroma of the bronze cultivar Fry ranged from 12.1 to 14.2 based on maturity level; this is comparable to range of chroma 13.1 to 16.3 observed in this study. Conner and MacLean (2013) found chroma values ranging from 2.4 to 22.8 and Threlfall et al. (2007) found chroma values ranging from 8.0 to 52.8 on four black cultivars (Black Beauty, Ison, Nesbitt, and Supreme) and two bronze cultivars (Granny Val and Summit) with the bronze genotypes generally having lower chroma values, both of which are consistent with our findings (Table 1). Chroma values were generally higher in 2012 than in 2013. In 2012, AM 03 had the highest chroma (17.9) and AM 27 and 'Delicious' had the lowest (2.6 and 2.3, respectively), whereas in 2013, 'Fry' had the highest chroma (14.1) and AM 18 and AM 27 had the lowest (2.1 and 2.0, respectively) (Table 1). There was a strong negative correlation between chroma and hue angle ($r = -0.93$) and a positive correlation between chroma and L* ($r = 0.72$). In general during storage for 3 weeks at 2 °C,

Fig. 3. Force to penetrate skin of muscadine genotypes stored at 2 °C for 3 weeks. Each SE bar is constructed using 1 SE from the mean (2012 and 2013). Bronze genotypes were AM 01, AM 03, AM 15, AM 26, 'Fry', 'Summit', and 'Tara' and the black genotypes were AM 02, AM 04, AM 18, AM 27, AM 28, 'Delicious', 'Ison', 'Nesbitt', 'Southern Jewel', and 'Supreme'.

Table 1. Physicochemical attributes of muscadine genotypes in 2012 and 2013 averaged across Weeks 0, 1, 2, and 3 of storage.

Yr	Berry color	Genotype	Titratable acidity (%) ^a	pH	Soluble solids (%)	L*	Chroma	Hue (°)
2012	Bronze	AM 01	0.57 abc ^b	3.63 bc	25.5 ab	41.1 bc	13.8 bc	71.0 d
		AM 03	0.29 h	3.58 c	24.0 bc	45.2 a	17.9 a	57.7 d
		AM 15	0.53 def	3.38 ef	19.7 fg	41.6 bc	12.7 cd	57.2 d
		AM 26	0.51 f	3.81 a	22.0 de	40.0 c	11.8 d	76.7 d
		Fry	0.57 abcd	3.63 bc	22.5 cd	42.1 b	14.9 b	80.0 d
		Summit	0.58 ab	3.72 ab	25.6 a	40.1 c	15.1 b	54.0 d
		Tara	0.56 abcd	3.58 c	21.2 def	44.1 a	12.9 cd	80.9 d
	Black	AM 02	0.59 ab	3.79 a	20.1 f	26.3 d	2.8 fg	287.4 abc
		AM 04	0.60 a	3.83 a	20.5 ef	44.0 a	3.6 fg	199.5 c
		AM 18	0.26 h	3.79 a	17.9 h	27.2 d	3.0 fg	218.0 bc
		AM 27	0.53 cdef	3.42 e	19.8 fg	26.7 d	2.6 g	220.2 bc
		AM 28	0.56 bcde	3.56 cd	20.7 ef	27.3 d	3.1 fg	205.1 c
		Delicious	0.54 cdef	3.43 de	20.7 ef	27.2 d	2.3 g	236.7 bc
		Ison	0.45 g	3.25 f	17.9 h	27.0 d	4.5 ef	222.5 bc
		Nesbitt	0.56 abcd	3.59 bc	20.0 f	27.2 d	6.4 e	309.7 ab
		Southern Jewel	0.52 ef	3.41 e	18.3 gh	28.0 d	4.5 ef	235.6 bc
		Supreme	0.59 ab	3.76 a	19.9 f	27.5 d	5.7 e	359.4 a
			<0.0001	<0.0001	<0.0001	<0.0001	<0.0001	<0.0001
2013	Bronze	AM 01	0.45 a	3.50 fg	17.6 cde	91.2 a	4.9 fg	193.1 de
		AM 03	0.28 ghi	3.73 cd	18.2 bc	57.3 b	11.2 d	102.1 ef
		AM 15	0.40 bc	3.40 g	17.7 cd	51.7 c	11.7 cd	113.5 ef
		AM 26	0.26 hi	3.81 bc	15.7 i	43.5 d	13.1 b	90.6 f
		Fry	0.34 de	3.75 cd	19.1 a	42.3 de	14.1 a	94.5 f
		Summit	0.30 efgh	3.68 d	17.8 cd	39.8 e	12.1 c	85.9 f
		Tara	0.33 defg	3.71 d	16.9 efg	43.6 d	13.7 ab	95.1 f
	Black	AM 02	0.31 efgh	3.96 a	17.8 cd	25.7 g	2.3 kl	309.6 ab
		AM 04	0.34 de	3.73 cd	16.6 gh	25.1 g	3.3 ij	210.1 cd
		AM 18	0.34 def	3.69 d	15.9 hi	34.7 f	2.1 l	266.4 abcd
		AM 27	0.38 bcd	3.65 de	17.8 cd	26.1 g	2.0 l	335.4 ab
		AM 28	0.23 i	3.74 cd	14.3 j	25.2 g	4.3 gh	349.5 a
		Delicious	0.45 a	3.52 f	16.8 fg	35.3 f	3.0 jk	296.2 abc
		Ison	0.42 ab	3.47 fg	18.6 ab	26.5 g	2.4 kl	282.1 abcd
		Nesbitt	0.33 defg	3.81 bc	16.8 fg	27.0 g	3.8 hi	251.6 bcd
		Southern Jewel	0.36 cd	3.56 ef	18.9 ab	33.8 f	7.7 e	288.8 abc
		Supreme	0.28 fghi	3.88 ab	17.5 def	26.4 g	5.4 f	242.8 bcd
			<0.0001	<0.0001	<0.0001	<0.0001	<0.0001	<0.0001

^aTitratable acidity (%) expressed as tartaric acid.

^bMeans followed by the same letter are not significantly different within each year at $\alpha = 0.05$, separated by Tukey's honestly significant difference.

the black genotypes had a 25% reduction in L* and 36% reduction in chroma, whereas the bronze genotypes had a 20% reduction in L* and 36% reduction in chroma.

Nutraceutical analysis. In 2012 and 2013, the main effect of genotype significantly affected total anthocyanins ($P < 0.0001$), total ellagitannins ($P = 0.0262$ and < 0.0001 , respectively), ORAC ($P < 0.0001$), total flavonols ($P < 0.0001$), total phenolics ($P < 0.0001$), and resveratrol concentrations ($P = 0.0024$ and 0.0007 , respectively). Berry nutraceutical concentrations were not evaluated during storage. We found total anthocyanins, total ellagitannins, total phenolics, and resveratrol concentrations were comparable to those previously reported, whereas total flavonol concentrations were generally lower (Conner and MacLean, 2013; Lee et al., 2005; Marshall et al., 2012; Pastrana-Bonilla et al., 2003; Sandhu and Gu, 2010; Striegler et al., 2005; Stringer et al., 2009; Threlfall et al., 2007).

In both 2012 and 2013, the black genotype AM 27 had the highest anthocyanins (122.0 and 41.8 mg/100 g, respectively), but as expected, no anthocyanins were detected in the bronze genotypes (Table 2). Total anthocyanin concentrations in the black genotypes

were generally higher in 2012 than in 2013 (average values of 68.1 and 32.2 mg/100 g for 2012 and 2013, respectively). A negative correlation with total anthocyanins and chroma ($r = -0.87$) and a positive correlation with hue angle ($r = 0.75$) were found, showing that lower chroma values and greater hue angles were related to higher total anthocyanins, which was not surprising because bronze genotypes generally had higher chroma values and lower hue angles and no anthocyanins. Black genotypes had an average total anthocyanin concentration of 501.2 mg/100 g.

Total ellagitannin concentration was slightly higher in 2013 compared with 2012. In 2012, total ellagitannins ranged from 1.6 ('Supreme') to 12.4 mg/100 g ('Ison') and from 4.0 (AM 01) to 12.8 mg/100 g (AM 03) in 2013 (Table 2). Black and bronze genotypes had average total ellagitannins concentrations of 6.8 and 7.2 mg/100 g, respectively.

Oxygen radical absorbance capacity is widely accepted as being a good estimation of antioxidant capacity of fruits, although its significance is often questioned, because it does not accurately represent the bioactivity of the antioxidants in the human body. We found ORAC values that ranged from 47.7

('Tara') to 110.6 ('Ison') $\mu\text{mol TE/g}$ in 2012 and from 53.5 (AM 03) to 115.5 $\mu\text{mol TE/g}$ ('Ison') in 2013 (Table 2). ORAC values were similar to those previously reported by Sandhu and Gu (2010) and Talcott and Lee (2002) but were considerably higher than those reported by Lee et al. (2005), Striegler et al. (2005), and Threlfall et al. (2007). ORAC values were found to be higher in 2013 compared with 2012. The cultivar Ison had the highest ORAC values both years of the study, whereas 'Supreme' and 'Tara' had among the lowest both years. Conversely, Threlfall et al. (2007) reported 'Nesbitt' having among the lowest ORAC values, whereas Striegler et al. (2005) identified that 'Supreme' had among the highest of those in their reports. Black and bronze genotypes had average ORAC values of 82.3 and 68.2 $\mu\text{mol TE/g}$, respectively.

Generally, genotypes differed in total flavonol concentration among years with the exceptions of AM 15 and 'Summit', which had among the highest concentration both years of this study. Total flavonols ranged from 7.3 ('Supreme') to 70.6 mg/100 g (AM 03) in 2012, whereas in 2013, total flavonols ranged from 9.9 (AM 28) to 47.9 mg/100 g (Table 2). The bronze genotypes were generally

Table 2. Nutraceutical content of muscadine genotypes in 2012 and 2013 at harvest (Week 0).

Yr	Berry color	Genotype	Total anthocyanins (mg/100 g)	Total ellagitannins (mg/100 g)	ORAC ($\mu\text{mol TE/g}$) ^a	Total flavonols (mg/100 g)	Total phenolics (mg/100 g)	Resveratrol (mg/100 g)
2012	Bronze	AM 01	0.0 e ^{y,x}	2.1 d	71.2 e	41.0 bc	604.7 abc	13.2 ab
		AM 03	0.0 e	12.2 a	92.1 b	70.6 a	701.3 ab	5.5 b
		AM 15	0.0 e	4.4 bcd	86.7 bcd	29.3 cde	639.8 abc	3.9 b
		AM 26	0.0 e	1.5 d	34.2 g	22.7 cde	368.3 c	4.7 b
		Fry	0.0 e	4.4 bcd	84.6 cd	28.4 cde	583.8 abc	4.0 b
		Summit	0.0 e	10.5 ab	86.8 bcd	63.1 ab	492.1 bc	4.8 b
		Tara	0.0 e	2.9 cd	47.7 f	19.5 cde	439.0 bc	5.1 b
	Black	AM 02	61.2 bcd	5.7 abcd	87.1 bcd	19.4cde	581.2 abc	3.8 b
		AM 04	109.3 ab	7.1 abcd	71.3 e	26.1 cde	539.2 abc	7.0 ab
		AM 18	78.0 abc	2.6 d	48.4 f	11.5 de	358.8 c	4.0 b
		AM 27	122.0 a	8.0 abcd	81.7 d	21.5 cde	669.3 ab	16.7 a
		AM 28	55.1 bcde	2.9 cd	52.0 f	8.9 de	354.5 c	4.4 b
		Delicious	98.1 abc	10.0 abc	90.9 bc	32.6 cd	618.6 abc	5.2 b
		Ison	44.4 cde	12.4 a	110.6 a	22.1 cde	797.3 a	10.1 ab
		Nesbitt	17.9 de	2.0 d	72.2 e	17.3 cde	518.6 abc	4.3 b
		Southern Jewel	78.1 abc	4.3 bcd	67.6 e	9.1 de	694.7 ab	5.8 b
		Supreme	16.7 de	1.6 d	52.7 f	7.3 e	366.1 c	4.1 b
			<0.0001	0.0262	<0.0001	<0.0001	<0.0001	0.0024
2013	Bronze	AM 01	0.0 h	4.0 d	71.2 e	15.8 cdef	528.3 abcd	3.9 bc
		AM 03	0.0 h	12.8 a	53.5 h	33.8 b	575.9 ab	6.7 abc
		AM 15	0.0 h	11.9 a	87.2 d	47.9 a	604.3 a	3.8 bc
		AM 26	0.0 h	6.7 cd	57.3 gh	23.5 bcd	392.1 abcd	3.7 bc
		Fry	0.0 h	12.5 a	59.3 fgh	24.9 bc	527.4 abcd	6.2 abc
		Summit	0.0 h	11.2 a	57.6 gh	23.4 bcde	535.0 abcd	11.1 ab
		Tara	0.0 h	4.2 d	65.9 efg	12.7 def	476.6 abcd	3.3 c
	Black	AM 02	36.4 abcd	10.4 ab	106.3 ab	16.8 cdef	464.5 abcd	4.1 bc
		AM 04	41.1 ab	12.2 a	97.2 bc	18.4 cdef	558.3 ab	5.5 abc
		AM 18	32.7 bcde	5.7 cd	85.7 d	11.7 ef	326.8 cd	3.5 c
		AM 27	41.8 a	6.6 cd	94.8 cd	14.7 cdef	448.3 abcd	4.6 abc
		AM 28	26.5 efg	5.5 cd	68.6 ef	9.9 f	316.9 d	2.9 c
		Delicious	39.3 abc	10.5 ab	107.0 a	19.6 cdef	606.7 a	6.2 abc
		Ison	30.2 def	6.7 cd	115.5 a	12.7 def	544.3 abc	4.2 bc
		Nesbitt	22.5 fg	7.7 bc	67.8 ef	11.0 f	450.1 abcd	8.1 abc
		Southern Jewel	31.2 cdef	7.5 bc	111.3 a	15.7 cdef	579.0 ab	3.2 c
		Supreme	19.9 g	5.7 cd	57.8 gh	11.5 f	354.2 bcd	12.1 a
			<0.0001	<0.0001	<0.0001	<0.0001	<0.0001	0.0007

^aORAC = oxygen radical absorbance capacity [$\mu\text{mol Trolox equivalents (TE)/g}$].

^yMeans followed by the same letter are not significantly different within each year at $\alpha = 0.05$, separated by Tukey's honestly significant difference.

^x0.0 = concentrations lower than detectable level using high-performance liquid chromatography.

higher in total flavonols than the darker genotypes, which may be attributed to the presence of the flavonol myricetin in the bronze genotypes (Marshall et al., 2012). A positive correlation with total flavonols and soluble solids ($r = 0.73$) and a negative correlation with hue angle and total flavonols ($r = -0.73$) occurred. These correlations possibly illustrate that riper berries have higher flavonol concentrations, because soluble solids have been shown to be an indicator of muscadine berry ripeness and berries with lower hue angles had higher total flavonols, which is supported by the data because the bronze genotypes generally had higher total flavonol levels and lower hue angles. Black and bronze genotypes had average total flavonol concentrations of 15.9 and 32.6 mg/100 g, respectively.

Total phenolic concentrations were generally higher in 2012 compared with 2013, likely as a result of the added stress on the vines from hot and dry growing conditions and the plants responding with increased phenolic production. In 2012, total phenolics ranged from 354.5 (AM 28) to 797.3 mg/100 g ('Ison') and in 2013, total phenolics ranged from 316.9 (AM 28) to 606.7 mg/100 g ('Delicious') (Table 2). We found 'Summit' to have among the highest levels of total phenolics, which was similar to the findings of Threlfall et al. (2007). However, in our study, 'Supreme' had among the lowest total phenolics of the genotypes measured, whereas Striegler et al. (2005) found 'Supreme' to have among the highest total phenolic concentration of 6072 mg/kg⁻¹ (607.2 mg/100 g). Total phenolics were positively correlated to ORAC ($r = 0.78$). Black and bronze genotypes had average total phenolic concentrations of 507.4 and 533.5 mg/100 g, respectively.

Resveratrol concentrations were similar both years of the study. Resveratrol ranged from 3.8 (AM 02) to 16.7 mg/100 g (AM 27) in 2012, whereas in 2013, resveratrol ranged from 2.9 (AM 28) to 12.1 mg/100 g ('Supreme') (Table 2). No clear relationship between berry color and resveratrol concentrations were found; conversely Ector et al. (1996) found resveratrol to be greater in black genotypes. Magee et al. (2002) found the bronze 'Summit' to have among the highest concentration of resveratrol in a group of both black and bronze genotypes, which is similar to our study. Ector et al. (1996) found that resveratrol concentrations were higher for muscadines compared with *V. vinifera* table grapes. The different resveratrol concentrations found in our study could be the result of environment, maturity, or cultural management, because resveratrol is produced in response to environmental factors during the growing season (Marshall et al., 2012).

Overall, AM 03, AM 04, AM 27, and 'Ison' had the highest nutraceutical content (total anthocyanins, total ellagitannins, total phenolics, total flavonols, resveratrol, and ORAC), whereas AM 18, AM 28, 'Supreme', and 'Tara' had the lowest content.

Conclusions

After 3 weeks of storage at 2 °C, the black genotypes had a 25% reduction in L*, 36% reduction in chroma, 30% reduction in penetration force, and 39% increase in unmarketability, whereas the bronze genotypes had a 20% reduction in L*, 36% reduction in chroma, 36% reduction in penetration force, and 48% increase in unmarketability. Additionally, the bronze muscadines were visually darker, decayed more, and softened during storage.

Overall, both percent unmarketable berries and percent weight loss increased during storage, showing importance as storage parameters. Force to penetrate the berry skin generally decreased during storage, also showing potential as an important postharvest storage parameter, particularly because some genotypes had significantly less reduction in force during storage. Physicochemical parameters TA, pH, and soluble solids remained relatively constant during storage and therefore are not important postharvest storage measurements to routinely use in evaluating storage potential. The berry color measurements, L*, chroma, and hue angle, generally showed no clear pattern during storage.

Among the sources of variation in this study besides year, genotype was the most common source with differences among most dependent variable means. Differentiating the potential value of breeding selections, particularly for postharvest storage potential, adequate variation for a characteristic or trait is needed. Furthermore, the differences among years for many dependent variables indicated the importance of multiyear evaluations of breeding selections for storage potential. The information gained in this study expands on previous work (Barchenger et al., 2014) and provides input for breeding programs and postharvest evaluation protocol for commercial use that could lead to identifying and releasing improved cultivars for fresh-market production with enhanced postharvest potential.

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