

5-2022

## Arkansas Fresh-market Blackberries: Identifying Unique Attributes and Harvest Practices that Impact Marketability

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Arkansas Fresh-market Blackberries: Identifying Unique Attributes and Harvest Practices That  
Impact Marketability

A thesis submitted in partial fulfillment  
of the requirements for the degree of  
Master of Science in Food Science

by

Andrea Lea Myers  
University of Arkansas  
Bachelor of Science in Food Science, 2019

May 2022  
University of Arkansas

This thesis is approved for recommendation to the Graduate Council.

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## ABSTRACT

Fresh-market blackberries (*Rubus* subgenus *Rubus*) are sold worldwide and have attributes that appeal to consumers. The delicate-nature of the fruit requires hand harvesting, and minimal handling of the fruit postharvest. Objectives of this research on Arkansas fresh-market blackberries was to 1) identify the unique attributes 2) determine the best handling practices to increase postharvest quality, and 3) evaluate the potential of soft robotic gripper for harvesting. For the first objective, blackberry genotypes were harvested from the University of Arkansas System (UA System) Division of Agriculture Fruit Research Station in Clarksville, AR. Nineteen genotypes were harvested in 2020, eight genotypes were harvested in 2021. Physical and compositional attributes, and volatiles were evaluated and descriptive sensory attributes of six genotypes each year were evaluated. For both years, berries were 6-15 g, 24-44 mm long, 21-26 mm wide, 2-13 N firm, 9-15% soluble solids, 3.0-4.2 pH, 0.4-1.4% titratable acidity, and 8.2-32.2 soluble solids/titratable acidity ratio. ‘Sweet-Ark<sup>®</sup> Ponca’ had highest soluble solids in both years (14-15%). There were 159 volatile aroma compounds identified in Arkansas blackberry genotypes in 2020 and 103 in 2021, mainly monoterpenes, esters, aldehydes, and alcohols. In both years, five impactful volatiles, ethyl butanoate (fruity), linalool (floral), ethyl 2-methylbutanoate (fruity), 2-hexenal (green), and geraniol (sweet) were identified. For descriptive sensory attributes in 2020, the genotypes differed in fruity aroma, green/unripe aromatics, and sour basic tastes. In 2021, the genotypes differed in overall intensity of aromatics and basic tastes. For objective 2 in 2020 and 2021, cultivar, harvest method, and acclimation temperature were examined to increase postharvest quality. Physical and composition attributes were evaluated at harvest and marketability attributes were evaluated after postharvest storage for 21 days at 2 °C on four cultivars harvested using two harvest methods and two acclimation

treatments. For both years berries were within commercially acceptable ranges. Cultivar impacted marketability attributes. Overall, there were no clear trends on marketability degradation of the blackberries. For objective 2 in 2020 and 2021, evaluations were done to develop a prototype of a soft-robotic gripper to harvest fresh-market blackberries. In 2020, a custom-made force sensing apparatus (sensors) was developed to determine the force (N) to harvest. Then in 2021, this data was used to create a soft robotic gripper prototype (gripper) for harvesting blackberries. In both years, physical and compositional attributes were evaluated at harvest, and marketability attributes were evaluated after postharvest storage for 21 days at 2 °C of four Arkansas-grown cultivars. The force used by the thumb and middle finger (0.77 N and 0.37 N, respectively) were greatest for harvesting blackberries. A prototype of a 3-prong soft robotic gripper was designed using results from the force sensing apparatus. The forces applied to grab, stabilize, and harvest blackberries with the sensors or gripper did not cause excessive marketability damage to the blackberries at harvest or after 21 days at 2 °C postharvest storage. This project identified unique flavor attributes, determined the best handling practices to increase postharvest quality, and evaluated the potential for a soft robotic gripper for harvesting.

## ACKNOWLEDGEMENTS

First, I would like to thank Dr. Renee Threlfall, my advisor, for giving me the privilege and opportunity to work under her advisement. She saw something in me that I did not. She taught me the value of hard work, passion for fruit, and the benefits of organization.

Appreciation is owed to my committee members Dr. Luke Howard and his technician Cindi Brownmiller for their guidance and allowing me to take up many hours in their lab running tests. I would like to thank Dr. Amanda McWhirt for the guidance in my project. She has always been a friendly face and her knowledge in the industry is immense and much appreciated.

Thank you to Dr. Yue Chen and Anthony Gunderman for allowing me to collaborate on the robotic gripper. It has been a wonderful learning experience to see the engineering side of fruit harvesting. I can't wait to see where this project is headed.

Appreciation is also owed to Dr. John Clark and Dr. Margaret Worthington; without your groundwork I would not have the fruit to study. I would also like to thank both of you for allowing Carmen Johns to help me find fruit to harvest and for her overall guidance. I take pride in being an "honorary horticulture student".

Additional appreciation is owed to Dr. Jacquelyn Lee and the crew at the fruit research station in Clarksville. Thank you for the hard work you do year-round to supply the fruit for my research.

Thank you to The Dozier's at Sta-N-Step Farms and Neal Family Farms for allowing me to harvest blackberries at your farm. Thank you for taking care of the plants and providing fruit for my research.

Appreciation is owed to John and Elizabeth Aselage for the knowledge and support. A&A Orchard will be a part of my history no matter where my path takes me. Thank you for sharing your orchard with me.

Funding appreciation goes to the Arkansas Department of Agriculture, United States Department of Agriculture (AM190100XXXXG157) and the University of Arkansas for the Chancellors Fund for Innovation and Collaboration grant (IFA2019-026).

Appreciation is owed to my lab mates: Amanda, Cody, James, & Jordon. Thank you for helping me harvest and all the hours measuring berries and squeezing juice. Thank you for all your positive words, encouragement, and data proofing.

Finally, I would like to thank my friends and family that gave me encouragement and support. Thank you for understanding people, places, and things had to take a backseat to get us where we are today.

## **DEDICATION**

To Pepé, Jackie, Taco, Bob, Harley, Zeke, and all the fur babies I have loved

As I reflect, I can't help but to feel blessed. I remember a conversation years ago where someone chastised me for taking an internship at an orchard. Little did I know that internship would lead me to where I am today. Without that job I would not have been led to this master's program to learn the skills of agriculture research and to transition into my next endeavor. I have met many wonderful people here at the University of Arkansas and the list of people that pushed and supported me is endless. You know who you are.

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## OVERALL INTRODUCTION

Blackberry (*Rubus* subgenus *Rubus*) are grown domestically and internationally for both fresh and processing industries. Fresh-market blackberries are harvested for direct sale to consumers, while processing blackberries are harvested for other uses, such as freezing, canning, and beverages. Blackberries can be harvested by hand or machine, and the method of harvesting depends on the use of the fruit.

Over the last 20 years, blackberry cultivation has increased worldwide. North America was the largest producer by weight of fresh-market blackberries, with 7,200 hectares (17,700 acres) of commercially-cultivated blackberries primarily grown in Oregon, Washington, and California (USDA NASS, 2017). United States blackberry production was valued at \$31.1 million, with \$5.4 million from fresh-market sales and \$25.7 million from processed sales (USDA NASS, 2017).

Fresh-market blackberries are a valuable fruit and have become more commercially available in the last decade. Consumers that once only had fresh blackberries seasonally now expect to have year-round availability of fresh-market blackberries at retail commercial markets. Blackberries have established a more prominent place in the market due to enhanced shipping capability, prolonged postharvest storage, shelf-life, off-season availability, and double blossom/rosette disease resistance (Clark, 2005; Strik et al., 2007). To meet demand, both public and private blackberry breeding programs have focused on enhancing blackberry plant and fruit attributes. Fresh-market blackberries have unique attributes that impact consumer perception, harvest-handling, postharvest storage, and marketability.

One of the major factors impacting commercial availability of fresh-market blackberries is the postharvest potential or shelf-life. Proper harvest and handling of the berries can minimize

damage, which can decrease yield loss and increase consumer satisfaction. Fresh-market blackberries can have problems associated with leakage, decay, and red drupelet reversion, a postharvest disorder that causes individual drupelets on the berry that are black at harvest to turn, or revert, to a reddish color. The way blackberries are grown, harvested, and stored impacts the quality of the fruit which is an important driver for consumer purchasing. Identifying unique cultivar attributes, harvesting strategies, and postharvest storage practices of the fresh-market blackberries will help advance future breeding efforts for the expansion of the blackberry industry.

### **OBJECTIVES**

- 1) Identifying Unique Attributes of Arkansas Fresh-market Blackberries
- 2) Determining Best Handling Practices for Fresh-market Blackberries to Increase Postharvest Quality
- 3) Evaluate Potential for a Soft Robotic Gripper for Harvesting Fresh-market Blackberries

## LITERATURE REVIEW

### Arkansas Fresh-market Blackberries: Identifying Unique Attributes and Harvest

#### Practices that Impact Marketability

#### INTRODUCTION

Blackberry (*Rubus* subgenus *Rubus*) plants are native to several continents, including Asia, Europe, and North and South America, and are generally referred to as caneberries. Blackberries are grown domestically and internationally for both fresh and processing industries. Fresh-market blackberries are harvested for direct sale to consumers, while processing blackberries are harvested for other uses, such as freezing, canning, and beverages. Blackberries can be harvested by hand or machine, and the method of harvesting depends on the use of the fruit.

Over the last 20 years, blackberry cultivation has increased worldwide. According to Finn and Clark (2011), raspberry production gave rise to blackberry production, but blackberries have lower production costs, higher plant vigor, and greater disease tolerance than raspberries. Cultivated blackberries are grown in excess of 25,000 hectares (61,000 acres) worldwide, with Mexico, Spain, and Italy as the top producers of total hectares (Clark and Finn, 2014).

Data from the United States Department of Agriculture, National Agricultural Statistics Service (USDA NASS) (2017) showed that North America was the largest producer by weight of fresh-market blackberries, with 7,200 hectares (17,700 acres) of commercially-cultivated blackberries primarily grown in Oregon, Washington, and California. In 2017, Oregon produced 18,000 metric tons (20,100 tons) on 2,500 hectares (6,300 acres). Blackberry yields per 0.40 hectare (one acre) were 2.9 metric tons (3.19 tons) with 1,360 metric tons (3 million pounds) sold as fresh berries and 16,783 metric tons (37 million pounds) sold as processed product

(USDA NASS, 2017). United States blackberry production was valued at \$31.1 million, with \$5.4 million from fresh-market sales and \$25.7 million from processed sales (USDA NASS, 2017).

Blackberries have become popular among consumers because of their increased availability, reported health benefits, and unique flavors (Carvalho and Betancur, 2015; Koca and Karadeniz, 2009; Robinson et al., 2020 and Souza et al., 2014). Blackberries are a nutraceutical-rich fruit, meaning that the berries provide additional human health benefits, like antioxidants, other than the basic nutritional value when consumed.

Fresh-market blackberries in the United States have become more commercially available in the last decade. Consumers that once only had fresh blackberries seasonally, either harvested from a wild plant or pick-your-own operations, now expect to have year-round availability of fresh-market blackberries at retail commercial markets. Blackberries have established a more prominent place in the market due to enhanced shipping capability, prolonged postharvest storage, shelf-life, off-season availability, and double blossom/rosette disease resistance (Clark, 2005; Strik et al., 2007). To meet demand, both public and private blackberry breeding programs have focused on enhancing blackberry plant and fruit attributes. Fresh-market blackberries have unique attributes that impact consumer perception, harvest-handling, postharvest storage, and marketability.

One of the major factors impacting commercial availability of fresh-market blackberries is the postharvest potential or shelf-life. Proper harvest and handling of the berries can minimize damage, which can decrease yield loss and increase consumer satisfaction. Fresh-market blackberries can have problems associated with leakage, decay, and red drupelet reversion, a

postharvest disorder that causes individual drupelets on the berry that are black at harvest to turn, or revert, to a reddish color.

Fresh-market blackberries are a valuable fruit with unique attributes. The way blackberries are grown, harvested, and stored impacts the quality of the fruit which is an important driver for consumer purchasing. Identifying unique cultivar attributes, harvesting strategies, and postharvest storage practices of the fresh-market blackberries will help advance future breeding efforts for the expansion of the blackberry industry.

### **Blackberry plants**

Blackberries are in the family *Rosaceae*, genus *Rubus* and subgenus *Rubus*. Blackberries are perennial plants (roots survive each year), but the plant above the soil is biennial (canes grow for a year, bear fruit the next year, and then die). The first year, a new stem, also known as the primocane, grows but does not produce flowers. In its second year, the cane becomes a floricanes, and the stem does not grow longer, but the lateral buds break to produce flowering laterals with smaller leaves with three or five leaflets. The flowers are produced in late spring and early summer resulting in blackberry fruit that is ripe about a month after flowering.

Historically, blackberries were grown and harvested in the wild. Those plants were dense and thorny making the ripe fruit difficult to harvest. In the 1800s, plants were selected for cultivation with fruit production for sale beginning the 1900s (Clark and Finn, 2014). Over the years, fruit breeders selected blackberries that had more erect canes that could be trained to grow on trellis systems in addition to selecting cultivars for breeding that were thornless for easier harvest.

Cultivated blackberries have three different types of cane growth, semi-erect, erect, and trailing. The semi-erect canes grow erect primocanes, but later the canes branch to the side to



bend down and touch the ground. When the cane tip reaches the ground, it has the ability to form a new plant. The erect plants can be self-supporting but do benefit from using a trellis because the long canes can be trained to stimulate production of side branches. Trailing blackberry canes are long and thin and grow along the ground unless supported by a trellis. Erect blackberry plants can grow to up to three feet.

Although most blackberry plants are floricanes fruiting, blackberry breeders developed primocane-fruiting plants that produce canes that flower and fruit the first year. The first primocane-fruiting cultivars, 'Prime-Jim<sup>®</sup>' and 'Prime-Jan<sup>®</sup>', released for commercial sale were developed at the UA System Division of Agriculture (UA System) Fruit Research Station in Clarksville, AR in the 1990's. The benefit of primocane-fruiting plants was to shift fruiting time from mid-summer to late summer or fall, extending the growing season depending on the climate in the region. Primocane-fruiting cultivars do not grow well in regions where temperatures are too hot during bloom. According to Clark (2008), summer heat in Arkansas can reach over 32 °C causing damage to the flower and the fruit. However, these primocane-fruiting cultivars grow well in the moderate summer temperatures of Oregon and northern California. Primocane-fruiting canes can be cut back to the ground after harvest for one crop per year. The plants can also be double cropped, canes produce a crop in the fall on the primocane, and the floricanes will produce a crop in the summer the following season. The method of cropping can be adapted to the climate where the plants are grown.

Tipping, or topping, is a practice commonly used for canopy management of blackberry canes. Primocanes are tipped in the summer to promote branch formation from axillary buds below the cut. Takeda et al. (2020) determined that in 'Prime-Ark<sup>®</sup> Traveler', a primocane-fruiting plant from the UA System, grown at the Appalachian Fruit Research Station in

Kearneysville, WV, had primocanes that emerged in April, and those primocanes had 64% more flower shoots than those that emerged after May after tipping the previous year. Cutting plants back prevents winter freeze injury, and the previous years primocane grows back as a fruit-producing florican (Black et al., 2019).

### **Chilling requirements in blackberry plants**

Chilling is a measurement in time that a plant needs to be dormant before the plant can have adequate growth or bud break the following growing season. Blackberries need 100 to 900 hours of winter chill (below 7 °C) depending on climate and cultivar (Lin and Agehara, 2020; Clark et al., 2019; Clark and Finn, 2014). Insufficient winter chill can cause poor and erratic budbreak, prolonged flowering, and low fruit yield limiting commercial production in subtropical climates. Flower buds develop on primocanes in late fall. Buds will stay dormant during winter, and budbreak will be brought on by warm spring temperatures. Bud dormancy has two stages, endodormancy, which is regulated by endogenous physiological factors during which buds cannot sprout even under optimal conditions until they are exposed to a certain amount of winter chill (Harkess, 2020). The second stage is ecodormancy where budbreak is induced by a certain period of warm temperatures. A major benefit of primocanes is that they do not need to overwinter. Eliminating the need for overwintering gives the blackberries the opportunity to grow in areas where they would typically not go dormant. In regions where there is inadequate chilling for florican-fruiting blackberries, primocane production is possible.

In the 1980s, cultural manipulations were developed to force florican-fruiting blackberries into fruiting without a dormancy period (Clark and Finn, 2014). ‘Brazos’ was developed by Texas A&M University and had a chilling period of 300 hours. The globally popular ‘Tupy’ was developed to have a similar low chilling requirement making both cultivars

popular in Central Mexico where temperatures are moderate compared to the northern United States and Europe. Lin and Agehara (2020) added gibberellic acid to three cultivars, ‘Natchez’, ‘Navaho’, and ‘Ouachita’ grown in subtropical climates for two growing seasons at the University of Florida’s Gulf Coast Research and Education Center in Balm, Florida. They found that exogenous gibberellic acid application was an effective bud dormancy-breaking compound. Similarly, Güçlü et al. (2018) investigated blackberries grown in Isparta, Turkey and found the addition of gibberellic acid worked as a growth stimulator when used in warmer climates without sufficient chilling hours.

### **Blackberry trellis systems**

Another advancement in blackberry cultivation is the use of trellis systems to train the canes of the plants. The selection of the trellis system is dependent on the type of cane growth of the plant. The I-trellis is a single or double wire spaced apart and secured to posts at approximately 0.61 and 1.22 m (2 and 4 ft) off the ground. A V-trellis has two posts in a V configuration about 20 to 30 degrees from vertical. The T-trellis has a post where wires are secured to both sides of the trellis to make room for primocanes to grow upright between the wires.

The Rotating Cross Arm (RCA) is a relatively new development combining trellis design with cane training (Fernandez et al., 2015; Takeda et al., 2020). In an RCA trellis, the positions of the canes are moved during the growing season. In spring, the trellis is moved to a horizontal position so that the plants flower on one side. After flowering the trellis is rotated to a vertical position for harvest. In late November, the trellis is rotated back to the horizontal position, near the soil level to protect the plants from winter injury. McWhirt et al., 2020 reported that using an RCA system can improve plant yield and lower postharvest decay due to increased fruit firmness

for ‘Ouachita’. Henderson et al., 2020 evaluated the RCA in 2019 and 2020 at the Fruit Research Station in Clarksville, AR on three blackberry cultivars (Prime-Ark<sup>®</sup> Traveler, Ouachita, and Osage) and research indicated the RCA had potential to minimize postharvest floricane weight loss, decay, leakage, and reduced Spotted Wing Drosophila (SWD) (*Drosophila suzukii*) pest pressure while improving size, and weight, of floricane berries.

Maughan et al. (2018) found that harsh winters and late spring in Utah can result in significant blackberry cane damage and crop loss in traditional blackberry production.

Nonnecke et al. (2017) used an RCA trellis in Iowa where winter temperatures can reach -13 °C. The study showed potential for commercial yield using the RCA system, where other trellising systems would not protect the blackberry plants in harsh winters.

Trellis systems are used to control cane growth on blackberry plants making floricane removal easier, keeping the fruit off the ground, increasing light penetration into the canopy, increasing air movement, facilitating spray applications, and making berries more accessible for harvest. Erect blackberries are grown for fresh-market use and are easier to harvest by hand (Strick and Finn, 2012). Erect blackberries can be grown without support, but research done by Maughan et al. (2018) found a trellis will reduce cane breakage from wind and help keep canes neat and easy to harvest. The ease of harvest makes for a more even, clean picking and lessens the attraction of pests attracted to overripe and rotted fruit. Trellis systems can also be used with a crop cover or without depending on the harshness of the climate. Overall, trellis systems can make plants easier to manage and adaptable for climate conditions.

### **Blackberry high tunnel production**

Another advancement in blackberry cultivation in diverse climates is the use of high tunnel that are semi-permanent structures made of arches and covered with polyethylene plastic

to create a protected environment for plants. In the United Kingdom, the use of polytunnels protect about 90% of the soft fruit sold (Barnett, 2007). Raspberries and blackberries benefit from growing in high tunnels, which can extend harvest seasons, improve fruit quality and yield, and enable growers to harvest fruit when raining. Studies in North Carolina and Arkansas (Fernandez et al., 2015) showed that primocane-fruiting raspberry and blackberry cultivars grown in tunnels resulted in high yields their first growing season. In comparison with field-grown raspberries, the tunnel-grown yields were 30% greater or more, depending upon location and growing conditions. Tunnels typically are between 3 to 5 m high and 6 to 10 m wide, but various sizes and upgrades are available. The tunnels can be assembled side-by-side to cover areas of several hectares. Placing tunnels close together gives more protection from possible wind damage. Plastic is typically used as a cover for tunnels and is replaced every few years as the plastic degrades or is damaged. In colder regions, plastic can be hung at the end of the tunnels to keep some heat inside the tunnel to protect the fruit from colder temperatures at night. In climates with cold winters, tunnels may also extend summer harvest seasons into the fall.

By using tunnels to extend the season, production of blackberries in colder climates is more viable, offering protection from extreme or erratic weather and decreased pest presence resulting in greater yield. Black et al. (2019) used tunnel-covered plots on primocane-fruiting raspberries at the Utah State University Greenville Research farm in North Logan, UT. The tunnel-covered plots were compared with field plantings for primocane growth rate, fruiting season, yield, and fruit quality. Use of high tunnels increased cane growth rate, advanced the harvest season by 18 to 26 days depending on season, and cultivar but did not consistently affect yield or fruit size. High elevation areas have challenging growing seasons including cold winter temperatures and short growing seasons. Tunnels protect fruit against cold as well as many

extreme or unusual weather events such as hail, wind, torrential rain, and unexpected cold snaps. They also provided shading from extreme sun and prevented deer damage. Based off a set of semi-structured interview questions, Conner and Demchak (2018) determined that tunnels extended the season for independent growers in eight states (Arkansas, Arizona, Kansas, Michigan, Minnesota, New York, West Virginia, and Wisconsin) where weather difficulties can impede the growing season. One of the drawbacks of high tunnels is nutrient management, because nutrients are cycled faster due to the warmer temperatures and higher productivity. Mettler and Hatterman-Valenti 2018 used a combination of the RCA trellis system and rowcovers for blackberry production in North Dakota. Their research indicated cultivar had a greater impact on plant growth and fruit quantity than temperature regulation.

### **Breeding blackberries**

Identification of marketability attributes can help guide fresh-market blackberry breeding programs, increasing consumer awareness, and enhancing profit obtained by growers.

Blackberry breeding initiatives occur on every continent with the exception of Antarctica (Strik et al., 2007). In the United States, blackberry breeding programs have existed for more than 100 years and continually work to enhance favored traits and reduce undesirable traits in plants and fruit. Blackberry breeders use existing cultivars and breeding selections (genotypes) to develop and release new cultivars.

The oldest currently active program is at the USDA Agricultural Research Service in Corvallis, OR, which was initiated in 1928 (Finn and Clark, 2011). Fresh-market blackberry cultivars released by USDA include ‘Obsidian’, ‘Metolius’ and the newest releases ‘Eclipse’, ‘Galaxy’ and ‘Twilight’ (USDA, 2020).

In 1964, the UA System blackberry breeding program was initiated by Dr. James N. Moore (Clark, 1999). The program is currently directed by Dr. John Clark and Dr. Margaret Worthington. The UA System blackberry breeding program is located at the Fruit Research Station, Clarksville, AR, and has prioritized development efforts on attributes including thornlessness, erect growth habit, mechanical harvesting capability, disease resistance, productivity, and environmental and geographic adaptation (Clark, 1999; Strik et al., 2007). The fruit improvement objectives for this program included large fruit size, desirable flavor, firmness, and high plant fertility (Clark, 1999). The UA System breeding program has developed and patented 43 fresh-market blackberry cultivars and is regarded as one of the leading public blackberry breeding programs in the world. In 2020, the UA System blackberry breeding program profited \$1.48 million dollars from blackberry royalties from plant patents (University of Arkansas System, Arkansas Agricultural Experiment Station, 2021). The UA System blackberry breeding program also produced advancements in thornless plants, erect cane structures, increased fruit firmness, and the development of primocane-fruiting, and cultivars to lengthen the harvest season (Clark and Moore, 1999; Clark, 2005; Moore, 1984; Moore and Clark, 1993). 'Ouachita' and 'Osage' are two of the most widely-grown cultivars released from the UA System (*personal communications, Dr. John Clark*). The most recent cultivars from the UA System are 'Sweet-Ark<sup>®</sup> Caddo', (Clark et al., 2019) released in 2018, 'Sweet-Ark<sup>®</sup> Ponca', released in 2019 and 'Prime-Ark<sup>®</sup> Horizon', released in 2020.

Mexico is the leading producer of blackberries worldwide, with most of the crop produced for export into the off-season fresh markets in North America and Europe (Perry, 2017). 'Tupy' is the primary cultivar grown in Mexico. 'Tupy' has the erect blackberry 'Comanche' and a wild Uruguayan blackberry as parents (Clark et al., 2012).

## **Blackberry harvesting**

Fresh-market blackberries are harvested when firm and shiny-black. Most fresh-market blackberries are hand-picked to ensure the fruit maintains quality from harvest to consumption. Depending on cultivar, blackberries can be harvested from either floricanes for two to three weeks or around eight weeks for double-cropped (primocane and floricanes fruiting) as fruit on the plants ripen at different times (Clark and Perkins-Veazie, 2011). However, hand harvesting has downsides, as berries must be harvested at peak ripeness. Lack of labor, weather disruptions, and abundance of fruit can lead to yield loss at harvest.

The method of harvesting blackberries impacts the potential for shipping and storage. Edgley et al. (2020) reported that mechanical injury caused anthocyanin degradation and increased red-drupelet reversion. Edgley et al. (2019) also evaluated the temperatures at time of harvest and showed that higher temperatures positively correlated with development and severity of red-drupelet reversion. Previous theories suggested time of day had an impact on postharvest qualities of fresh-market blackberries. Felts et al. (2020) showed there was minimal impact of harvest time (7:00am versus 12:00pm), but genotype and length of time in storage had the greatest impact. Most of the color change happened within 24 hours of the blackberries entering cool storage, but the number of reverted drupelets can increase for up to two weeks postharvest (Edgley et al., 2019; Yin, 2017). Edgley et al. (2019) found that ‘Ouachita’ berries exposed to impact damage at a warmer initial temperature ( $>25^{\circ}\text{C}$ ) before cooling to  $2^{\circ}\text{C}$  had increased rates of reversion compared to berries that were gradually cooled.

## **Automated harvesting of blackberries**

Automated machine harvesters have become common in agriculture to offset the decreasing harvest workforce and increasing costs. Fruit such as strawberries and plums have



shifted to mechanical harvesting. Robotic harvesters with infrared sensing have been used to harvest strawberries based on fruit ripeness by cutting the stem without touching the berry (Xiong et al., 2019). The caneberries, such as blackberries and raspberries, pose more challenges since the fruit is in a dense cluster of leaves and canes. In 1964, a prototype was developed at the University of Arkansas for one of the first mechanical harvesters for caneberries by mechanically shaking the canes (Morris et al., 1978). Harvest labor costs accounted for over two-thirds of total labor costs. One concern for the welfare of the workers, most of them migrant workers, spurred the development of mechanical harvesters to alleviate labor costs (Morris, 1999). Cavender et al. (2014) evaluated an over-the-row rotary harvester, however that was not ideal because of the damage caused to the berries. Pérez- Pérez et al. (2018) reported that vibration significantly increased incidences of red drupelet reversion versus control fruit. In addition, with a mechanical harvester, berries were harvested regardless of the ripeness resulting in yield loss. Fumiomi and Peterson (1999) showed existing bramble mechanical harvesters can detach blackberries from the plant more cheaply than hand harvesting, however the fruit did not maintain fresh-market quality. The development of a harvester that can optimize the quality and quantity of fruit harvested is important to the future of the fresh-market blackberry industry.

Soft-touch robots, made from rubber, silicone, or other flexible and durable materials driven by an actuation mechanism, have been introduced into the fruit harvest sector. Soft touch was initially used to preform minimally invasive surgery or to complete a surgical procedure safely and quickly, while minimizing damage to the tissue (Runciman et al., 2019). Much like surgery, the goal of fresh-market machine harvesting is to minimize damage to delicate fruit tissue. Soft-touch robotics are ideal for grasping and manipulating delicate objects. Actuation and control of pressure distribution can be programmed at the design stage reducing the need for

high-resolution sensory feedback (Venter and Dirven, 2017). Different thicknesses of cables inside the soft touch coating can mimic the force used exerted as compared to harvest the fruit by hand. The cable actuation gives dexterity to the robotic device.

Continuum robots, that do not have rigid links or joint but rather are able to bend continuously, are a good candidate for this type of work. Octopus tentacles are an inspiration since they can demonstrate dexterous control, bending and stiffening despite consisting of only soft tissue. The downfall is that they need some sort of base support, and it is more difficult to exert force over a longer length span (Runciman et al., 2019). Soft-touch robotics have to a balance of sensitivity and durability. Touch provides feedback to the controls relaying information such as the position of the object, the service curvature, friction, and force exerted by robot (Bartolozzi et al., 2016). Current advancements in the soft-touch robotics field emulate the movement of a human hand. Development of a gripper has the capacity to reduce labor-intensive harvesting (Venter and Dirven, 2017).

### **Blackberry grades and standards**

The USDA defines grades and standards for produce that are sold commercially to help maintain quality and guidance. The fruit is graded on appearance, texture, composition, and marketability attributes. Improvement in these categories is an important aspect for the fruit breeder since appearance enhances overall quality of the fruit and increases profits obtained by the grower. Fresh-market blackberry appearance is graded a Number 1 if they are firm, well-colored, well-developed, and if 99% of the packaged blackberries are free from any mold or decay (USDA, 2018). A blackberry is graded Number 2 if more than 90% of the berry is free from damage or two percent of the packaged berries are free from mold and decay (USDA, 2018). Research continues to determine cultivars that are best for fresh-market blackberries, and

public and private blackberry breeding programs continue to develop new cultivars to meet the needs of growers, packers, processors, and consumers (Clark and Finn, 2014).

In terms of red drupelet reversion, discoloration affecting individual drupelets that are reddish black or reddish blue and blend in color are not considered a defect; however, discoloration that is noticeably bluish red to bright red and do not blend in color will be scored as damage and serious damage (USDA, 2018). Red drupelet damage is noted when it detracts from the appearance, and serious damage when seriously detracts from the appearance, but USDA provides visual aids to help with identification of the level of damage. In terms of white drupelet disorder (tan or white color) damage is noted when impacting two or more drupelets, and serious damage if more than five drupelets (USDA, 2018).

White drupelets occur prior to harvest and may be caused by weather or ultra-violet (UV) radiation when there is hot, dry air with little humidity. Humidity absorbs and scatters the UV rays, so it is not directly penetrating the fruit (Bolda et al., 2009). Shade cloth covering the plants and fruit decreased white drupelet disorder in three cultivars, ‘Chickasaw’, ‘Kiowa’, and ‘Sweetie Pie’ grown in south Mississippi (Stafne et al., 2017). ‘Apache’ and ‘Kiowa’ were more susceptible to white drupelets (Bolda et al., 2009; Fernandez, 2012). Nitrogen application could also decrease occurrence of white drupelet disorder (Quezada et al., 2007).

### **Blackberry size, shape, and firmness**

Blackberries are an aggregate fruit comprised of drupelets surrounding a soft tissue receptacle (torus). Each drupelet has a thin exocarp, a fleshy mesocarp, and a hard-lignified endocarp, or pyrene, that encloses a single seed (Tomlik-Wyremblewska et al., 2010). The size (berry weight, length, and width) of a fully-ripened blackberry varies among cultivars. On average, the weight of each blackberry will range from 5-15 g with length of 15-30 mm

(Carvalho and Betancur, 2015). The berries can have different shapes, such as a round shape, or the berries can be long and oval shaped.

In addition, firmness, measured by the force to compress an individual blackberry can vary. Firmness is influenced by protopectin in the inter-cellular structures of blackberry drupelets, which act like cement to give blackberries a firm texture, but hydrolysis, large respiration rates, and warmer conditions during ripening decrease protopectin activity (Jennings, 2003). Evaluation of many genotypes of blackberries grown in Arkansas showed that the average firmness was 3-8 Newtons (Threlfall et al., 2016b; Segantini et al., 2018; Salgado and Clark, 2016).

### **Blackberry sugars and acids**

Another factor that makes up the unique attributes of fresh-market blackberries is the composition of sugars and acids. Sugars and organic acids are the main soluble constituents of berries that impact the sweet and sourness (Mikulic-Petkovsek et al., 2012). The primary sugars in blackberries are glucose, fructose, and sucrose, with glucose and fructose having higher concentrations and sucrose significantly less. Glucose values range from 1.8-4.4 g/100 g, fructose ranges from 1.7-4.5 g/100 g while sucrose was less than 0.1 g/100 g (Du et al., 2010; Segantini et al., 2018). Sugars are the major soluble solids in blackberries, but other soluble materials include organic and amino acids and soluble pectin. Soluble solids levels (% or °Brix) of the juice from blackberries can be measured using a refractometer. The soluble solids of commercially acceptable fresh-market blackberry ranges from 8-11% (Threlfall et al., 2016a).

Primary organic acids in blackberries are isocitric, lactone isocitric, and malic acid. Segantini et al. (2018) measured isocitric acid (0.8-1.1 g/100g), isocitric lactone (0.2-0.3 g/100 g), and malic acid (about 0.3g/100 g) in blackberries grown in Arkansas. The titratable acidity is

a measure of the predominant acid (usually citric) in the fruit and is inversely related to the pH of the berry.

Cultivated blackberries, in contrast to wild blackberries, have a greater size, but lower soluble solids, titratable acidity, and pH (Yilmaz et al., 2009). The pH of a commercially-acceptable fresh-market blackberry ranges from 3.0-3.6 and titratable acidity ranges from 0.7-1.4% (Threlfall et al., 2016a). Segantini et al. (2018) determined important attributes for quality, demonstrating fresh-market blackberries had a good balance of acidity and sugar content, as noted by sensory panelists. The balance of sugars and acids along with maintaining fruit quality during storage are important attributes for fresh-market blackberries.

### **Blackberry volatile aromatics**

Aromatic attributes, or volatiles perceived by the olfactory system while chewing a sample in the mouth, impact the flavor consumers experience when eating a blackberry. According to Wang et al. (2005), blackberries have a wide range of aroma profiles that can be quantified including acids, esters, alcohols, aldehydes, ketones, lactones, and terpenoids. Jacques et al. (2014) identified 45 volatile compounds in ‘Tupy’, and the majority of volatiles were comprised of terpenoids with limonene as the predominate individual compound. Quian and Wang (2005) summarize that there is no single compound that is the source for a typical blackberry odor, but the aroma of the blackberries in their study of ‘Marion’ and ‘Thornless Evergreen’ comes from a mixture of compounds in certain proportions.

Volatiles extracted with hexane and analyzed using gas chromatography mass spectrometry (GC-MS), were mainly hydrocarbons and those extracted with acetone were furans and pyrans. A flame ionization detector (FID) can be used in GC-MS analysis to quantitatively measure analytes in a gas stream. FID is a process where an analyte is injected, then filtered

through a GC column. After the sample leaves the GC column it enters the FID detector, consisting of a flame, fed usually with hydrogen. The analyte enters the flame, burning completely due to combustion, creating ions to be detected. The ions are detected by a collector electrode, the negative electrode, which induces a current. The induced current is proportional to the rate of ionization, which ultimately depends on the hydrocarbon concentration. The data is then converted into a peak. The resulting chromatogram shows the amount of volatile present in a sample (Skoog et al., 2018).

A technique known as Solid Phase Micro Extraction (SPME) is also used for volatile compounds. The SPME fiber is coated in specific material depending on the desired analytes captured for analysis. The SPME adsorbs the volatile sample, and the sample can then be injected directly into the GC for analysis. Wang et al. (2005) found that only 13% of the compounds in blackberries were aromatic. In a similar study, Du et al. (2010a, 2010b) quantified volatiles of eight different genotypes of blackberries. With a range of compounds, such as esters, terpenoids, aldehydes and ketones, alcohols, norisoprenoids, lactones, acids, and furanones. The compounds were quantified, but the values of each compound did not distribute uniformly across all genotypes.

GC-MS along with gas chromatography-olfactometry (GC-O) can also be used to evaluate the aroma of fresh-market blackberries. The GC-O separates compounds using GC, and as a peak is detected that odor is separated and delivered to a trained panelist to evaluate the intensity of the aroma detected (Wang et al., 2005). Barba et al. (2018) evaluated odorant compounds that enhanced sweet flavor in sugar-reduced juice using GC-O to isolate taste-enhancing compounds and showed that ethyl 2-methylbutonate enhanced flavor sweetness. This type of analysis could be helpful to target odorant compounds that enhance desired flavors.

Limited GC-O research has been conducted on blackberries, however research conducted on other food products can help identify and isolate desired compounds in blackberries.

Zhu et al. (2018) evaluated mulberry fruit and detected four compounds that could influence six aroma descriptors (sweet, sour, mellow, fruity, sulfur, and green). The four compounds identified indicated that volatile compounds present at sub-threshold concentrations might interact with other volatile compounds. More research needs to be conducted on blackberries to see if there is an interaction with lower-concentration compounds. Evaluation of volatile aroma profiles can help determine what consumers desire when purchasing fresh-market blackberries, as well as guide breeding decisions for more flavorful cultivars.

### **Blackberry postharvest storage and marketability**

Postharvest storage can impact the quality of berries that arrive to the market, remain on the shelf at the market, and are stored in the consumer's home. Roughly one-third of fresh produce is lost at various points in the distribution system (Kader, 2002). Blackberries can be held in postharvest cold storage for a week or more, but storability depends on many factors. Temperature and humidity are two of those factors. Temperature is one of the most important factors that influence the deterioration of harvested commodities. Most perishable horticultural commodities last longest at optimal temperatures and can deteriorate two to three-fold for each 10 °C rise in temperature (Kader, 2002). Blackberries are susceptible to water loss during storage and an effective method for reducing water loss is to increase the relative humidity in air. However, high humidity in storage can lead to bacterial and fungal growth. A relative humidity of 85 to 90 percent has proved satisfactory for storage (Willis et al., 1989).

Blackberries are one of the most perishable types of fruit because of their thin and fragile skin and large respiration and transpiration rates. Hence, rapid changes in blackberry

composition and sensory properties, along with decay can occur during postharvest storage. Temperature management, including rapid cooling after harvest and maintenance of low temperatures, is the most important factor in minimizing blackberry deterioration and maximizing quality and postharvest storage (Bolda et al., 2012). For blackberry storage, temperatures from 0-5 °C and modified atmosphere (5–10% oxygen/15–20% carbon dioxide) are recommended during shipping (Cia et al., 2007; Kader, 2002).

Storage time, storage temperature, and handling of blackberries can damage berries and make berries less appealing to consumers. Mold growth can occur on blackberries during postharvest storage, and the most predominant species of mold growth is *Botrytis cinerea* Pers. and *B. caroliniana*, also known as gray mold (Li et al., 2012). *B. cinerea* has an affinity for a high-pectin content host and destroys plant cell structure then colonizes on dead tissue on the fruit. Although optimum growth of *B. cinerea* is 20 °C, its ability to grow at colder temperatures (as low as 0 °C) leads to slow decay during storage of fresh-market fruit (Bautista-Baños, 2014). Other research has shown storage temperature of blackberries was directly related to degree of deterioration (Palharini et al., 2015; Perkins-Veazie et al., 1999; Perkins-Veazie and Clark, 2005; Segantini et al., 2018).

Blackberries can be affected by poor harvest and handling procedures, as well as improper storage temperatures, leading to fruit deterioration and decreased marketability (Kader, 2002). Kim et al. (2015) showed that blackberries stored at 1 °C had better postharvest quality compared to 20 °C, and room temperature storage reduced quality in all cultivars. Types of storage container, packing procedures, storage temperature, and humidity affect marketability of the fruit (Joo et al., 2011). Kim et al. (2015) reported that fruit stored at 1 °C retained consistent marketability, however, when removed from cold storage and placed in room temperature, fruit



deterioration rapidly increased. Kader (2002) also showed stage of picking (dull black versus shiny black) affected leakage, decay, and red-drupelet reversion. Edgley (2020) investigated how harvest and handling of fresh blackberries impacted fruit quality showing that in 85% of blackberries that were handled roughly (picked from cane and dropped into a container with no regard to how or where the berry lands in regard to other berries already in the container) during harvest had red drupelet reversion (one drupelet per berry). In comparison, only 6% of blackberries that were handled gently (picked from cane and placed into container with delicate regard, ensuring it did not damage berries already in the container) displayed red drupelet reversion.

Firmer berries have longer shelf life (Segantini et al., 2017; Kim et al., 2015) and have enhanced potential for shipping and postharvest storage. The UA System evaluated postharvest storage potential of “crispy” genotypes with a firmer texture, which might be better suited for fresh-market sales in contrast to softer berries that can be used for processing or local market sales. According to Salgado and Clark (2016), the crispy genotypes maintain cell-wall and cell-to-cell adhesion and have better storage potential compared to less-crispy genotypes. Softness was positively correlated to decay/leakage of blackberries (Kim et al., 2015).

Cultivar could also affect postharvest disorders with some cultivars better suited for early morning harvesting. Different cultivars can produce berries that have large yield and longer storage potential (Clark and Moore, 1999), therefore those berries will be more profitable for the grower, and the consumer will have a berry with a longer post-purchase life. Lawrence and Melgar (2018) reported leakiness was greater in ‘Chester’ and ‘Triple Crown’ when harvested later in the day compared to other cultivars in the study.

Edgley et al., (2019) and Kim et al. (2019) reported anthocyanins, cyanidin3-glucoside, cyanidin 3-malonylglucoside, cyanidin 3-dioxalyglucoside, and total anthocyanin were significantly lower in red drupelets versus black drupelets. With increased availability of blackberries in the market, the quality of fruit will be a major driver for consumer purchasing.

### **Blackberry sensory**

Different types of sensory analysis have been done on fresh-market blackberries to determine consumer-driven attributes. Sensory science is “a scientific discipline used to evoke, measure, analyze, and interpret reactions to those characteristics of food and other materials as they are perceived by the senses of sight, smell, touch, taste, and hearing” (Stone and Sidel, 1993). The basic tastes of foods include sweet, sour, salty, bitter, and umami, but sweet, sour, and bitter are the primary basic tastes of blackberries.

Descriptive sensory analyses have been conducted to determine attributes that are commercially acceptable, such as appearance, aroma, basic tastes, aromatics, and feeling factors. Descriptive sensory analysis involves a trained panel that uses a lexicon (terms to describe the product) and references to evaluate products on a line scale. Threlfall et al. (2016a) developed a fresh-market blackberry lexicon in an evaluation of UA System blackberries with eight appearance, three basic tastes, two feeling factors, and eight aromatics of the blackberries were evaluated. According to Threlfall et al. (2016b), the descriptive sensory panelists were not able to differentiate sweetness among five blackberry genotypes; however, the panelists could easily differentiate sourness and overall aromatic impact. Segantini et al. (2017) studied sensory attributes in postharvest storage and reported panelists could not perceive a significant difference in color, uniformity of color, glossiness, firmness, or sweetness after storage, but could identify blackberries as more astringent and less sour and bitter after storage.

A consumer sensory panel where panelists represent the average customer and are recruited based on consumption and purchasing habits of the product evaluated is a valuable tool. In consumer sensory studies, a large number of consumers (over 75 panelists) is needed to ensure a representative population. The consumer panels assess the acceptability of a sample usually in terms likeability or preference. Consumers want a fresh-market blackberry that is uniform in color, fresh, has a good shelf life, fair-priced, rich in nutraceuticals, and has unique flavors and aromas (Threlfall et al., 2020). Appearance is important because consumers make purchase decisions based on appearance. Since blackberries are typically sold in transparent, plastic clamshells, the consumers can see the berries and want a glossy, black berry with a uniform size and little to no blemishes (Threlfall et al., 2020, 2021). Studies have shown consumers want a balance of sweetness and sourness in blackberries without bitterness (Mikulic-Petkovsek et al., 2012, Segantini et al., 2017; Threlfall et al., 2016b).

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## Chapter 1

### Identifying Unique Attributes of Arkansas Fresh-market Blackberries

#### Abstract

Fresh-market blackberries (*Rubus* subgenus *Rubus*) have unique flavors that appeal to consumers. Although basic tastes (sweetness, sourness, and bitterness) impact flavor perception of blackberries, volatile aroma compounds (substances which vaporize at ambient temperature) also contribute to aroma and flavor complexity. Blackberry genotypes (cultivars and breeding selections) were harvested from the University of Arkansas System (UA System) Division of Agriculture Fruit Research Station in Clarksville, AR. Nineteen genotypes were harvested in 2020, and eight genotypes were harvested in 2021. The physical, composition and volatiles of these genotypes were evaluated in triplicate at the UA System Food Science Department, then the descriptive sensory attributes of six genotypes each year were evaluated in duplicate at the UA System Sensory Science Center. The descriptive sensory panel (n=9 in 2020; n=7 in 2021) evaluated aroma, aromatics, basic tastes, and feeling factors of a puree of blackberries on a 15-point scale (0=less of attribute; 15=more of attribute). The composition attributes of these genotypes differed each year but were within the typical ranges for fresh-market genotypes grown in Arkansas. For both years, berries were 6-15 g, 24-44 mm long, 21-26 mm wide, 2-13 N firm, 9-15% soluble solids, 3.0-4.2 pH, 0.4-1.4% titratable acidity, and 8.2-32.2 soluble solids/titratable acidity ratio. ‘Sweet-Ark<sup>®</sup> Ponca’ had highest soluble solids in both years (14-15%), There were 159 volatile aroma compounds identified in Arkansas blackberry genotypes in 2020 and 103 in 2021, mainly monoterpenes, esters, aldehydes, and alcohols. In 2020, ‘Sweet-Ark<sup>®</sup> Caddo’ (28,430 µg/kg) had the highest total volatiles followed by ‘Tupy’ (17,249 µg/kg), and A-2620 (16,265 µg/kg), whereas in 2021, A-2620T (5,886 µg/kg) had highest total volatiles,

followed by A-2658T (2,702 µg/kg) and ‘Sweet-Ark<sup>®</sup> Caddo’ (2,651 µg/kg). In both years, five impactful volatiles, ethyl butanoate (fruity, apple-like), linalool (floral, perfume), ethyl 2-methylbutanoate (fruity), 2-hexenal (green, leafy), and geraniol (sweet, rose-like) were identified in Arkansas fresh-market blackberries with linalool in the highest concentration. For descriptive sensory attributes in 2020, the genotypes differed in fruity aroma, green/unripe aromatics, and sour basic tastes. ‘Sweet-Ark<sup>®</sup> Caddo’ had the highest fruity aroma, and Prime-Ark<sup>®</sup> Horizon the least. ‘Ouachita’ had the highest green/unripe aromatics and sour basic taste, and A-2701T had the least green/unripe aromatics and sourness. In 2021, the genotypes differed in overall intensity of aromatics and basic tastes. ‘Sweet-Ark<sup>®</sup> Caddo’ and ‘Ouachita’ had the highest overall intensity. ‘Sweet-Ark<sup>®</sup> Ponca’ had the highest sweetness, but ‘Ouachita’ had the highest sourness and bitterness. The combination of physical, composition, volatile, and descriptive sensory analysis can be a useful tool to steer breeding decisions to help southern U.S. growers better market blackberries, and determine commercial potential of Arkansas-grown, fresh-market blackberries.

## Introduction

Blackberry (*Rubus* subgenus *Rubus*) plants are native to several continents with many cultivars that are grown commercially. Blackberries are generally referred to as caneberries, with most cultivars that are floricanes fruiting, producing berries on the second-year canes (floricanes). However, there are cultivars that produce fruit on first-year canes (primocane). Blackberries are grown domestically and internationally for both fresh and processing industries. Fresh-market blackberries are harvested for direct sale to consumers, while processing blackberries are harvested for other uses, such as freezing, canning, and beverages. Fresh-market blackberries are hand-picked to ensure the fruit maintains quality from harvest to consumption.

Blackberry is one of the best examples of a wild-harvested specialty crop that moved to commercial use through increased consumer demand, new cultivars, advanced production methods, and year-round availability. Over the last 20 years, blackberry cultivation has increased worldwide. Cultivated blackberries are grown in excess of 25,000 hectares (61,000 acres) worldwide, with Mexico, Spain, and Italy as the top producers (Clark and Finn, 2014). Mexico is the leading producer of blackberries worldwide, with most of the crop produced for export into the off-season fresh markets in North America and Europe (Perry, 2017). 'Tupy', the primary cultivar grown in Mexico, has the erect blackberry 'Comanche' and a wild Uruguayan blackberry as parents (Clark et al., 2012).

The U.S. berry industry has experienced rapid growth in the past decades and accounted for 22.1% of the fruit market with a total value of \$7.5 billion in 2019. Blackberries account for \$697 million of the market value, and the total blackberry market value has grown by 7.3% between 2019 and 2020 (California Strawberry Commission, 2020). Data from the United States Department of Agriculture USDA, National Agricultural Statistics Service (NASS) (USDA

NASS, 2017) showed that North America was the largest producer by weight of fresh-market blackberries, with 7,200 hectares (17,700 acres) of commercially cultivated blackberries primarily grown in Oregon, Washington, and California. In 2017, Oregon produced 18,000 metric tons (20,100 tons) on 2,500 hectares (6,300 acres). Blackberry yields per 0.40 hectare (one acre) were 2.9 metric tons (3.19 tons) with 1,360 metric tons (3 million pounds) sold as fresh berries and 16,783 metric tons (37 million pounds) sold as processed product (USDA NASS, 2017).

Blackberries are a nutraceutical-rich fruit, meaning that the berries provide additional human health benefits, like antioxidants, other than the basic nutritional value when consumed. Blackberries have become popular among consumers because of their increased availability, reported health benefits, and unique flavors (Carvalho and Betancur, 2015; Koca and Karadeniz, 2009; Robinson et al., 2020; Souza et al., 2014). Consumers that once only had fresh blackberries seasonally, either harvested from a wild plant or pick-your-own operations, now expect to have year-round availability of fresh-market blackberries at retail commercial markets. Blackberries have established a more prominent place in the market due to enhanced shipping capability, prolonged postharvest storage, shelf-life, off-season availability, and double blossom/rosette disease resistance (Clark, 2005; Strik et al., 2007). To meet demand, both public and private blackberry breeding programs have focused on enhancing blackberry plant and fruit attributes. Fresh-market blackberries have unique attributes that impact consumer perception, harvest-handling, postharvest storage, and marketability.

Identification of marketability attributes can help guide fresh-market blackberry breeding programs, increase consumer awareness, and enhance profit obtained by growers. In the United States, blackberry breeding programs have existed for more than 100 years and continually work



to enhance favored traits and reduce undesirable traits in plants and fruit. Blackberry breeders use existing cultivars and breeding selections to develop and release new cultivars. The oldest currently active program is at the USDA Agricultural Research Service in Corvallis, OR and was initiated in 1928 (Finn and Clark, 2011). Fresh-market blackberry cultivars released by USDA include ‘Obsidian’, ‘Metolius’ and the newest releases ‘Eclipse’, ‘Galaxy’ and ‘Twilight’ (USDA, 2020).

In 1964, the University of Arkansas System Division of Agriculture (UA System) Blackberry Breeding Program was initiated by Dr. James N. Moore (Clark, 1999). The program is currently directed by Dr. John Clark and Dr. Margaret Worthington. The UA System blackberry breeding program is located at the Fruit Research Station, Clarksville, AR and has prioritized development efforts on attributes including thornlessness, erect growth habit, mechanical harvesting capability, disease resistance, productivity, and environmental and geographic adaptation (Clark, 1999; Strik et al., 2007). The fruit improvement objectives for this program included large fruit size, desirable flavor, firmness, and high plant fertility (Clark, 1999). The UA System Blackberry Breeding Program has developed and patented 43 fresh-market blackberry cultivars and is regarded as one of the leading public blackberry breeding programs in the world. In 2020, the UA System blackberry breeding program profited \$1.48 million dollars from blackberry royalties from plant patents (UA System, Arkansas Agricultural Experiment Station, 2021). The UA System blackberry breeding program also produced advancements in thornless plants, erect cane structures, increased fruit firmness, the development of primocane fruiting, and released cultivars to lengthen the harvest season (Clark and Moore, 1999; Clark, 2005; Moore, 1984; Moore and Clark, 1993). ‘Ouachita’ and ‘Osage’ are two of the most widely-grown cultivars released from the UA System (*personal communications, Dr. John*

*Clark*). The most recent cultivars from the UA System are ‘Sweet-Ark<sup>®</sup> Caddo’, (*Clark et al.*, 2019) released in 2018, ‘Sweet-Ark<sup>®</sup> Ponca’, released in 2019, and ‘Prime-Ark<sup>®</sup> Horizon’, released in 2020.

Blackberries are an aggregate fruit comprised of drupelets surrounding a soft tissue receptacle (torus). Each drupelet has a thin exocarp, a fleshy mesocarp, and a hard-lignified endocarp, or pyrene, that encloses a single seed (*Tomlik-Wyremblewska et al.*, 2010). The size (berry weight, length, and width) of a fully ripened blackberry varies among cultivars. On average, the weight of each blackberry will range from 5-15 g with length of 15-30 mm (*Carvalho and Betancur*, 2015). The berries can have different shapes, such as a round shape, or the berries can be long and oval shaped. In addition, firmness, measured by the force to compress an individual blackberry can vary.

Sugars and organic acids are the main soluble constituents of berries that impact the sweet and sourness (*Mikulic-Petkovsek et al.*, 2012). The primary sugars in blackberries are glucose, fructose, and sucrose, with glucose and fructose having higher concentrations and sucrose significantly less. Glucose values range from 1.8-4.4 g/100 g, fructose ranges from 1.7-4.5 g/100 g while sucrose was less than 0.1 g/100 g (*Du et al.*, 2010b; *Segantini et al.*, 2018). Sugars are the major soluble solids in blackberries, but other soluble materials include organic and amino acids and soluble pectin. Soluble solids levels (% or °Brix) of the juice from blackberries can be measured using a refractometer. The soluble solids of commercially acceptable fresh-market blackberry ranges from 8-11% (*Threlfall et al.*, 2016a). Primary organic acids in blackberries are isocitric, malic acid, and lactone isocitric. *Segantini et al.* (2018) measured isocitric acid (0.8-1.1 g/100g), isocitric lactone (0.2-0.3 g/100 g), and malic acid (about 0.3g/100 g) in blackberries grown in Arkansas. The titratable acidity is a measure of the

predominant acid (usually citric) in the fruit and is inversely related to the pH of the berry. Cultivated blackberries, in contrast to wild blackberries, have a greater size, but lower soluble solids, titratable acidity, and pH (Yilmaz et al., 2009). The pH of a commercially acceptable fresh-market blackberry ranges from 3.0-3.6 and titratable acidity ranges from 0.7-1.4% (Threlfall et al., 2016a). Segantini et al. (2018) determined important attributes for quality, demonstrating fresh-market blackberries had a good balance of acidity and sugar content, as noted by sensory panelists. The balance of sugars and acids along with maintaining fruit quality during storage are important attributes for fresh-market blackberries.

The aroma, appearance, flavor, and texture of blackberries varies by cultivar. Although the basic tastes (sweetness, sourness, and bitterness) impact the flavor of blackberries, volatile aroma compounds (substances in fruit which vaporize easily at ambient temperature) are also responsible for typical aromas and aromatic flavors of blackberries. Volatiles in blackberries include acids, esters, alcohols, aldehydes, ketones, lactones, phenols, and terpenoids but vary by cultivar, ripeness, and harvest and storage conditions (Du et al. 2010a; El Had et al. 2013; Qian and Wang 2005). Some published research on blackberry volatile composition has been done in the United States (Du et al. 2010; Qian and Wang 2005), Poland (Wajs-Bonikowska et al. 2017), Italy and Spain (D'Agostino et al. 2015), and Brazil (Jacques et al. 2014). Although Morin (2020) profiled the phenolic and volatile composition of 16 blackberry genotypes (cultivars and selections) harvested at the UA System Fruit Research Center and evaluated antiinflammatory capacities of three genotypes on inflamed cells.

According to Wang et al. (2005), blackberries have a wide range of aroma profiles that can be quantified including acids, esters, alcohols, aldehydes, ketones, lactones, and terpenoids. Jacques et al. (2014) identified 45 volatile compounds in 'Tupy', and the majority of volatiles

were comprised of terpenoids with limonene as the predominate individual compound. Qian and Wang (2005) concluded that no single compound is the source for a typical blackberry aroma, but the aroma of the blackberries in their study of 'Marion' and 'Thornless Evergreen' comes from a mixture of compounds in certain proportions. Research on Oregon-grown blackberries showed that 'Marion' and 'Black Diamond' (Du et al. 2010a) and 'Marion' and 'Thornless Evergreen' (Qian and Wang 2005) had volatiles including alcohols (32%), acids (32%), and monoterpenes (24%). Du et al. (2010b) and Qian and Wang (2005) showed hexanoic acid, 2-heptanol, linalool, butanoic acid, octanol, hexanol, benzyl alcohol,  $\alpha$ -pinene, acetic acid,  $\alpha$ -terpineol, and p-cymen-8-ol as the 10 major volatile compounds.

Volatiles are identified using gas chromatography mass spectrometry (GC-MS) then quantified using flame ionization detector (FID). Solid Phase Micro Extraction (SPME) fiber can be used to capture analytes in the headspace of a sample for analysis. Wang et al. (2005) found that only 13% of the compounds in blackberries were aromatic. In a similar study, Du et al. (2010a, 2010b) quantified volatiles of eight genotypes of blackberries and reported a range of compounds, such as esters, terpenoids, aldehydes and ketones, alcohols, norisoprenoids, lactones, acids, and furanones. The compounds were quantified, but the values of each compound did not distribute uniformly across all genotypes.

The measurement of total volatiles may give a partial profile of the aromas of blackberry genotypes. Wang et al (2005) used aroma extraction dilution analysis to characterize the aroma profile of 'Chickasaw' blackberries grown in Oregon and Arkansas. 'Chickasaw' was released from the UA System Blackberry Breeding Program and shares some genetics with current selections and cultivars from the UA System program. According to Wang et al. (2005) the compounds with the most impactful aromas in 'Chickasaw' grown in Arkansas were ethyl

butanoate (fruity, apple-like), linalool (floral, perfume), methional (cooked potato), ethyl 2-methylbutanoate (fruity), allo-ocimene (Chinese medicine, herbaceous), trans-2-hexenal (green, leafy),  $\beta$ -damascenone (rose-like, berry), geraniol (sweet, rose-like), and 2,5-dimethyl-4-hydroxy-3(2H)-furanone (sweet, caramel). Du et al. (2010b) found that furaneol (fruity, strawberry), linalool,  $\beta$ -ionone (sweet, floral, woody), 2-heptanol (citrus), and carvone (caraway seeds) were potent aromas of blackberries grown in the Pacific Northwest.

The variation in total quantified volatiles from year to year could be due to different factors. An eight-year study on blackcurrant berries (Marsol-Vall et al., 2018 and Severo et al. 2017) found the volatile concentration, especially esters, increased when berries received more ultraviolet light and when the average temperature the week before harvest was higher. Another factor could be the sample preparation and detection methods. According to Kraujalyte et al., 2013 SPME is a sensitive and fast technique that does not require solvents, but other parameters such as heating temperature, volume of sample, extraction time and SPME fiber could substantially affect results.

Different types of sensory analysis of blackberries have been done on fresh-market blackberries to determine consumer-driven attributes. Sensory science is “a scientific discipline used to evoke, measure, analyze, and interpret reactions to those characteristics of food and other materials as they are perceived by the senses of sight, smell, touch, taste, and hearing” (Stone and Sidel, 1993). The basic tastes of foods include sweet, sour, salty, bitter, and umami, but sweet, sour, and bitter are the primary basic tastes of blackberries.

Descriptive sensory analyses have been conducted to determine attributes that are commercially acceptable, such as appearance, aroma, basic tastes, aromatics, and feeling factors. Descriptive sensory analysis involves a trained panel that uses a lexicon (terms to describe the

product) and references to evaluate products on a line scale. Threlfall et al. (2016a) developed a fresh-market blackberry lexicon in an evaluation of UA System blackberries with eight appearance, three basic tastes, two feeling factors, and eight aromatics of the blackberries evaluated. According to Threlfall et al. (2016b), the descriptive sensory panelists were not able to differentiate sweetness among five blackberry genotypes; however, the panelists could easily differentiate sourness and overall aromatic impact. Segantini et al. (2017) studied sensory attributes in postharvest storage and reported panelists could not perceive a significant difference in color, uniformity of color, glossiness, firmness, or sweetness after storage, but could identify blackberries as more astringent and less sour and bitter after storage.

A consumer sensory panel where panelists are recruited based on consumption and purchasing habits of the product evaluated is an important sensory test. In consumer sensory studies, a large number of consumers (over 75 panelists) is needed to ensure a representative population. The consumer panels assess the acceptability of a sample usually in terms likeability or preference. Consumers want a fresh-market blackberry that is uniform in color, fresh, has a good shelf life, fair-priced, rich in nutraceuticals, and has unique flavors and aromas (Threlfall et al., 2020). Appearance is important because consumers make purchase decisions based on appearance. Since blackberries are typically sold in transparent, plastic clamshells, the consumers can see the berries and want a glossy, blackberry with a uniform size and little to no blemishes (Threlfall et al., 2020, 2021). Studies have shown consumers want a balance of sweetness and sourness in blackberries without bitterness (Mikulic-Petkovsek et al., 2012, Segantini et al., 2017; Threlfall et al., 2016b).

There is a critical need to determine the key aroma and flavor attributes that impact consumer preference and can be used to steer breeding decisions and help southern U.S. growers better

market blackberries. Since the UA System Blackberry Breeding Program contributes to the global blackberry industry, the objectives of the research were to identify unique attributes of Arkansas-grown fresh-market blackberries with a focus on the physical, volatile, and sensory attributes.

## **Methods and Materials**

### **Blackberry plants and culture**

Nineteen genotypes were evaluated in 2020 (A-2526T, A-2528T, A-2547T, A-2587T, A-2610T, A-2620T, A-2625T, A-2658T, A-2701T, APF-409T, ‘Natchez’, ‘Osage’, ‘Ouachita’, ‘Prime-Ark<sup>®</sup> 45’, ‘Prime-Ark<sup>®</sup> Horizon’ ‘Prime-Ark<sup>®</sup> Traveler’, ‘Sweet-Ark<sup>®</sup> Caddo’, ‘Sweet-Ark<sup>®</sup> Ponca’, and ‘Tupy’). Eight genotypes were evaluated in 2021 (A-2547T, A-2610T, A-2620T, A-2658T, A-2701T, ‘Ouachita’, ‘Sweet-Ark<sup>®</sup> Caddo’, and ‘Sweet-Ark<sup>®</sup> Ponca’). In 2021, a record freeze (-5 °C) in February and a late freeze in April impacted the survival and availability of fruit from the blackberry plants. The plants were grown at the UA System Fruit Research Station in Clarksville, AR (West Central Arkansas, lat. 35 °31’58” N and long. 93 °24’12” W). Plants were trained to a T-trellis with two lower wires ~0.5 m from the soil surface spaced 0.5 m apart and two upper wires ~1.0 m high spaced 0.8 m apart. The blackberry plants that were harvested for this project were in three plots with five plants per plot, and the plots were established in 2017, 2018, and 2019. Standard cultural practices for erect blackberry production were used including annual spring nitrogen fertilization (56 kg/ha N) using ammonium nitrate. The plants were irrigated as needed using trickle irrigation. Dormant pruning consisted of removing dead floricanes and removing primocane tissue to a point below the flowering area on the primocanes. The plants received a single application of liquid lime sulfur (94 L/ha) at budbreak for control of anthracnose (*Elsinoë veneta* [Burkholder] Jenk.). Raspberry

crown borer (*Pennisetia marginata* [Harris]) was controlled by a single application of a labeled insecticide with bifenthrin as the active ingredient in October of each year. Insecticides labeled for commercial use in Arkansas were used for spotted wing drosophila (*Drosophila suzukii* Matsumura) control.

### **Blackberry harvest**

Blackberries were hand harvested from the floricanes from 7:00<sub>AM</sub> to 10:00<sub>AM</sub>. The fruit was harvested at the shiny-black stage of ripeness and were free of major blemishes, flaws, or damage. About 2 kg of blackberries were harvested in June 2020 and 2021 in triplicate for each genotype and placed directly into 312 g (11oz) vented clamshells. After harvest, the clamshells were placed in chilled coolers and transported to the UA System Department of Food Science, Fayetteville for evaluation of physical attributes, composition attributes, volatile attributes, and descriptive sensory attributes.

### **Physical attribute analysis**

Five berries per genotype and replication were used for physical attributes then frozen (-10 °C) for composition analysis.

**Berry size.** Each berry was weighed (g) using a precision digital scale (PA224 Analytic Balance, Ohaus Corporation, Parsippany, NJ), then the length and width (mm) were measured using digital calipers.

**Berry firmness.** Firmness of each berry was measured by a Stable Micro Systems TA.TX. XT plus Texture Analyzer (Texture Technologies Corporation, Hamilton, MA). Firmness was measured using a 7.6-cm diameter cylindrical probe to compress with a trigger force of 0.02 N, an individual berry placed horizontally on a flat surface. The force needed to compress the berry was measured in Newtons (N).



## **Composition attribute analysis**

Composition of the juice from five berries per genotype and replication were measured for soluble solids, pH, titratable acidity, organic acids, and sugars. The five berries were thawed at room temperature (21 °C) and squeezed through cheesecloth to extract the juice for analysis.

**Soluble solids.** Soluble solids of the juice were measured and expressed as percent (%) using an Abbe Mark II refractometer (Bausch and Lomb, Scientific Instrument, Keene, NH).

**pH.** The pH of juice was measured using a pH700 Benchtop pH meter (APERNA Instruments, Columbus, OH).

**Titratable acidity.** The titratable acidity of the juice was measured using a Metrohm 862 Compact Titrosampler (Metrohm AG, Herisau, Switzerland) fitted with a pH meter. Three grams of sample was added to 50 mL degassed, deionized water and titrated with 0.1 N sodium hydroxide to an endpoint of pH 8.2. The titratable acidity of juice was expressed as % w/v (g/100 mL) citric acid.

**Soluble solids/titratable acidity ratio.** The soluble solids/titratable acidity ratio was calculated as the soluble solids divided by the titratable acidity.

**Organic acids and sugars.** Organic acids and sugars were determined using high performance liquid chromatography (HPLC). The juice for compositional analysis was filtered through a 0.45  $\mu\text{m}$  nylon filter (VWR International, Radnor, PA) and was analyzed using HPLC. Glucose, fructose, isocitric, and malic acids of blackberries were measured using previously established HPLC procedures (Walker et al., 2003; Segatini et al., 2018). The HPLC was equipped with a Bio-Rad HPLC Organic Acid Analysis Aminex HPX-87H ion exclusion column (300  $\times$  7.8 mm), Bio-Rad HPLC Fast Acid Analysis column (100  $\times$  7.8 mm), and a Bio-Rad HPLC column for fermentation monitoring (150  $\times$  7.8 mm) in series. A Bio-Rad Micro-Guard Cation-H refill cartridge (30  $\times$  4.5 mm) was used for a guard column (Bio-Rad, Hercules, CA). Columns were

maintained at 65 °C by a temperature control unit. Mobile phase consisted of a pH 2.28 solution of sulfuric acid and water with a resistivity of 18 M obtained from a Millipore Milli-Q reagent water system. The sulfuric acid solution was used as the isocratic solvent with 0.35 mL/min flow rate. The solvent delivery system was a Waters 515 HPLC pump equipped with a Waters 717 plus autosampler (Waters Corporation, Milford, MA). Injection volumes were 10  $\mu$ L for all samples, and run time for completion was 45 min. A Waters 410 differential refractometer to measure refractive index connected in series with a Waters 996 photodiode array detector monitored the eluting compounds. Isocitric and malic acids were detected by photodiode array at 210 nm and glucose and fructose were detected by the differential refractometer. The peaks were quantified using external standard calibration based on peak height estimation with baseline integration. Individual sugars, individual organic acids, total sugars (glucose + fructose), and total organic acids (isocitric + malic acid) were expressed as g/100 g.

### **Volatile aroma attribute analysis**

Ten berries per genotype and replication were frozen (-10 °C) after harvest and used for volatile aroma attribute analysis. Gas chromatography analysis was performed using a Shimadzu GC-2010 Plus Gas Chromatograph equipped with a Flame Ionization Detector (GC-FID) and a GCMS-QP2010 SE Mass Spectrometer (GC-MS). The analysis includes identification and quantitation of volatile compounds. For the analysis of blackberry volatiles, the weight of 10 frozen blackberries, deionized water and NaCl were mixed using a ratio of 1:2:0.1 (w/v/w). Two samples of 2mL berry/deionized water/NaCl solution were added to 2mL of deionized water (4mL total) were placed in 20mL headspace vials. The vials were incubated for 15 minutes with agitation at 65 °C, and then the volatiles were absorbed using an 85  $\mu$ m DVB/CAR/PDMS Solid Phase Microextraction (SPME) fiber was placed in the headspace above the sample for an

additional 20 minutes. The SPME fiber was then removed from the vial and placed into GC injection ports.

Samples were analyzed on both GC-FID and GC-MS and separation was performed on each using a HP-5 (30 m × 0.25 mm inner diameter, 5% phenyl-methylpolysiloxane, 1.0 μm film thickness) capillary column. For both GC-MS and GC-FID analysis, the injector temperature was 250 °C. Helium was used as the carrier gas and column flow rate was 1.92 mL/min for GC-FID and 1.20 mL/min for GC-MS. The oven temperature was programmed for a 4 min hold at 30 °C, then 30 °C to 180 °C at 6 °C/min, then from 180 °C to 280 °C at 8 °C/min, and with a 3 min hold at 280 °C. The GC-FID detector temperature was 280 °C, and the interface temperature for the GC-MS had an ion source temperature of 230 °C and an interface temperature of 250 °C. GC-MS was performed in full scan mode, with a scan range of 20-300 *m/z*. The volatiles were identified by comparison of their mass spectra with the spectral library, literature data, and retention indices, and expressed as μg/kg.

### **Descriptive sensory attribute analysis**

The descriptive sensory analysis was done at the UA System Sensory Science Center at the Food Science Department, Fayetteville in 2020 and 2021. The Covid-19 pandemic impacted the implementation of the sensory in both years so the fruit was frozen until evaluation, and purees of samples were evaluated. The descriptive sensory panelists (n=9 in 2020 and n=7 in 2021) evaluated the aroma, basic tastes, aromatics, and feeling factors of pureed blackberries in duplicate. Panelists were trained to use the Sensory Spectrum (New Providence, NJ) method, an objective method for describing the intensity of attributes in products using references for the attributes. Intensities of the attributes were based on the Universal Scale, a saltine cracker equal to 2.0, applesauce equal to 5.0, orange juice equal to 7.5, grape juice equal to 10.0, and Big Red

Gum® (Mars, Inc., McLean, VA) equal to 15.0. The panelists used a lexicon of descriptive sensory terms previously developed through consensus during orientation and practice sessions for fresh-market blackberries.

The blackberries used for this analysis were frozen after harvest, then 300 g of each genotype was placed in a sanitized Erlenmeyer flask, slightly thawed, then pureed using with a Magic Bullet blender (MBR-1101, Los Angeles, CA) with cross blades in a 473-mL container. The blackberries were served to the panelists one at a time at room temperature (25 °C) in Snap-Seal™ translucent polypropylene containers (45 mL) labeled with three-digit codes. Each container had 10 g of sample. Serving order was randomized across each replication to prevent presentation order bias. The descriptive panel evaluated the blackberry puree for 17 attributes using 0 = less of an attribute and 15 = more of an attribute. Four aromas (jam, berry, fruity, and vegetative), three basic tastes (sweet, sour, and bitter), eight aromatics (overall intensity, blackberry, earthy/dirty, green/unripe, overripe/fermented, chemical, mold/mildew, and metallic) and two feeling factors (astringent and metallic) were evaluated.

The genotypes evaluated in 2020 were A-2547T, A-2625T, A-2701T, ‘Ouachita’, ‘Sweet Ark® Caddo’, and ‘Sweet Ark® Horizon’. The 2020 blackberry genotypes were selected based on total levels of volatile compounds (two low, two middle, and two high). The genotypes evaluated in 2021 were A-2547T, A-2610T, and A-2701T, ‘Ouachita’, ‘Sweet Ark® Caddo’, and ‘Sweet Ark® Ponca’.

### **Statistical design and analysis**

For physical, composition, and volatile attributes, all genotypes were evaluated in triplicate by year. The descriptive sensory attributes of the genotypes were evaluated by year in duplicate. The data was analyzed by analysis of variance (ANOVA) using JMP® (version 16.0.0;

SAS Institute Inc., Cary, NC). Tukey's Honestly Significant Difference was used for mean separations ( $p = 0.05$ ). For the descriptive sensory evaluation, panelist main effect and genotype x panelist interaction were included in the model to account for the error explained by between-panelist variation. The genotype x panelist interaction was not significant for any descriptive sensory attributes in 2020 and only significant for overall intensity and chemical attributes in 2021 (data not shown), showing that panelists were consistent in their ratings of each genotype. Associations among all dependent variables were determined using multivariate pairwise correlation coefficients of the mean values using JMP (version 16.0.0; SAS Institute Inc., Cary, NC). Principle component analysis was done using XLStat (Addinsoft Inc., New York, NY).

## **Results and Discussion**

Average monthly temperature and rainfall were tracked, recorded, and reported from January to June, the end of blackberry harvest (Fig. 1.) The 2020 blackberry season in Clarksville, AR was relatively mild in terms of temperature and rainfall. The 2021 season had notable weather events in February and April. In 2020, the high temperatures in June were 33 °C and low temperatures of were 14 °C, while in 2021 the high temperatures in June were 33 °C and low temperatures were 11 °C. There was record cold temperatures (-5 °C) with 178 mm of snow in February of 2021 at the Fruit Research Station followed by a freeze after budbreak in late April (-1 °C overnight). The cultivars available for harvest were impacted by both low temperature events in 2021. Total rainfall in 2021 (765 mm) was less than rainfall in 2020 (843 mm). Rainfall in June 2021 (142 mm) was triple the rainfall in June 2020 (41 mm).

### **Physical attributes**

The cultivars harvested in 2020 and 2021 significantly impacted the physical attributes (berry weight, length, width, and firmness) evaluated at harvest (Table 1 and 2). For both years, berries

were 6-15 g, 24-44 mm long, 21-26 mm wide, and 2-13 N firm. The physical attributes were within ranges established by previous research on Arkansas fresh-market blackberries. Segantini et al. (2017) harvested 11 Arkansas genotypes in 2015 with firmness 5-9 N. Felts et al. (2020) harvested nine Arkansas genotypes in 2017 with berry weights 4-9 g, and 5-9 N firm. Carvalho and Betancur (2015) found the average weight of a blackberry ranges from 5-15 g and 15-30mm in length. Firmness is influenced by protopectin in the inter-cellular structures of blackberry drupelets, which act like cement to give blackberries a firm texture, but hydrolysis, large respiration rates, and warmer conditions during ripening decrease protopectin activity (Jennings, 2003). Evaluation of many genotypes of blackberries grown in Arkansas showed that the average firmness was 3-8 Newtons (Threlfall et al., 2016b; Segantini et al., 2018; Salgado and Clark, 2016).

**2020.** A-2620T had the highest berry weight (15.15 g) and berry length (43.75 mm) but had the lowest firmness (1.78 N). Osage had the lowest berry weight (6.27 g). Sweet-Ark<sup>®</sup> Ponca had the lowest berry length (25.90 mm). A-2658T had the highest berry width (25.94 mm) and APF-409T had the lowest (20.99 mm). A-2701T had the highest firmness (13.13 N). Tupy, the commercial standard, had a berry weight of 10.02 g, berry length of 36.13 mm, berry width of 24.18 mm, and berry firmness of 4.88 N.

**2021.** A-2701T had the highest berry weight (13.30 g) and firmness (11.34 N). ‘Sweet-Ark<sup>®</sup> Ponca’ had the lowest berry weight (6.07 g). A-2620T had the largest berry length (40.90 mm), ‘Sweet-Ark<sup>®</sup> Ponca’ had the lowest berry length (24.81 mm). A-2658T was the widest berry (25.96 mm) and A-2547T (20.92 mm) had the narrowest width. ‘Ouachita’ was the least firm (6.77 N).

### **Composition attributes**

The cultivars harvested in 2020 and 2021 significantly impacted the composition attributes (soluble solids, pH, titratable acidity, soluble solids/titratable acidity ratio, sugars, and organic acids) at harvest (Table 1, 2, 3, and 4). The primary sugars identified were glucose and fructose and the primary acids were citric and malic (Tables 3 and 4). For both years, berries had 9-15% soluble solids, 3.0-4.2 pH, 0.4-1.4% titratable acidity, and 8.2-32.2 soluble solids/titratable acidity ratio. The individual and total sugars in both years had a range of values for glucose (1.8-3.9 g/100 mL), fructose (1.9-3.8 g/100 mL), and total sugars (3.6-7.7 g/100 mL). The individual and total organic acids in both years had a range of values for citric (0.4-1.3 g/100 mL), malic (0.04-0.54 g/100 mL), and total organic acids (0.4-1.8 g/100 mL). The composition attributes were within ranges established by previous research on Arkansas fresh-market blackberries. Segantini et al. (2017) harvested 11 genotypes in 2015 with soluble solids 4.7-19.5%, pH 3.0-3.4, and titratable acidity 0.5-1.5%. In a consumer study, Threlfall et al. (2016) found that fresh-market blackberries should have soluble solids of 9-11%, titratable acidity of 0.9-1.0%, and a soluble solids/titratable acidity ratio of 10-13.

**2020.** ‘Sweet-Ark<sup>®</sup> Ponca’ had the highest soluble solids (14.60%), and A-2587T had the lowest soluble solids (9.80%). ‘Osage’ had the highest pH (3.91) and lowest titratable acidity (0.64%), conversely, ‘Ouachita’ had the lowest pH (3.01) and highest titratable acidity (1.37%). A-2658T (20.82) had the highest soluble solids/titratable acidity ratio, while APF-409T (8.20) had the lowest. ‘Tupy’ had a soluble solids level of 12.10%, pH of 3.58, titratable acidity of 0.93%, and a soluble solids/titratable ratio of 13.13

The genotypes that had the soluble solids/titratable acidity ratio of 10-13 included A-2526T, A-2528T, A-2587T, ‘Prime-Ark<sup>®</sup> 45’, ‘Prime-Ark<sup>®</sup> Horizon’, ‘Prime-Ark<sup>®</sup> Traveler’, Sweet-Ark<sup>®</sup> Caddo’, and ‘Tupy’.

In terms of sugars, 'Sweet-Ark<sup>®</sup> Ponca' had the highest glucose (3.66 g/100 mL), fructose (3.64 g/100 mL), and total sugars (7.30 g/100 mL), while A-2587T had the lowest glucose (1.79 g/100 mL), fructose (1.85 g/100 mL), and total sugars (3.63g/100 mL). APF-409T (0.54 g/100 mL) had the highest malic acid. 'Ouachita' had the highest citric acid (1.29 g/100 mL) and total organic acids, (1.78 g/100 mL). 'Osage' (0.44 g/100 mL) had the lowest citric acid, and A-2658T had the lowest malic (0.14 g/100 mL) and total organic acids (0.66 g/100 mL). Previous research in blackberry sugars (Ali et al. 2011, Du et al. 2010b, Felts et al. 2020, Kafkas et al. 2006, and Segantini et al. 2018) found glucose from 1.58-4.55 g/100 mL and fructose from 1.42-4.49 g/100 mL in blackberries. Felts et al. (2020), Mikulic-Petkovsek et al. (2012), and Segantini et al. (2018) measured citric acid that ranged from 0.32-1.06 g/100 mL. Felts et al. (2020), Kafkas et al. (2006), Mikulic-Petkovsek et al. (2012), and Segantini et al. (2018) measured malic acid and that ranged from 0.06-0.43 g/100 mL. All sugars and organic acids measured were within ranges of the previous research.

**2021.** 'Sweet-Ark<sup>®</sup> Ponca' had the highest soluble solids (13.70%), and A-2658T had the lowest soluble solids (9.03%). A-2701 had the highest pH (4.16) and lowest titratable acidity (0.36%), conversely, A-2620T had the lowest pH (3.13) and highest titratable acidity (0.93%). 'Sweet-Ark<sup>®</sup> Ponca' (32.24) had the highest soluble solids/titratable acidity ratio, while 'Ouachita' (11.32) had the lowest. The genotypes that had the soluble solids/titratable acidity ratio of 10-13 included A-2610T, A-2620T, and 'Ouachita'.

A-2620T had the highest glucose (3.93 g/100 mL), A-2701T had the highest fructose (3.77 g/100 mL) and total sugars (7.69 g/100 mL), while A-2658T had the lowest glucose (2.38 g/100 mL), fructose (2.32 g/100 mL), and total sugars (4.70 g/100 mL). A-2620T had the highest citric acid (0.87 g/100 mL), malic acid (0.09 g/100mL), and total acids (0.96 g/100mL). A-



2701T had the lowest malic acid (0.04 g/100mL). ‘Sweet-Ark<sup>®</sup> Caddo’ had the lowest citric acid (0.38g/100mL) and total acids (0.42 g/100mL).

### **Volatile aroma attributes**

The compounds identified in blackberries, their compound class, the measured retention index, the aroma category each was grouped into, more detailed aroma descriptors, and the total ion chromatogram (TIC) relative peak area in percent (Tables 5 and 6). Across all genotypes and both years, monoterpenes, esters, aldehydes, and alcohols were the major classes of volatiles found in the blackberries, accounting for 27, 22, 20, and 15% of total volatiles, respectively. Monoterpenes are a class of terpenes that contain two isoprene molecules and are predominantly the product of secondary metabolism of plants known for their biological activities such as antimicrobial, anti-inflammatory, and anti-plasmodial properties and have been used in flavorings and fragrances (Tchimene et al., 2013). Esters with a fruity aroma were the second largest class of compounds in all blackberries. Sesquiterpenes, ketones, aromatic hydrocarbons, norisoprenoids, and lactones were present in low amounts.

These results varied from those of Du et al. (2010a) and Qian and Wang (2005) for berries grown in the Pacific Northwest where alcohols, acids, and monoterpenes (32, 32 and 24% respectively) were the major classes, with other classes ranging between 0.1 and 3%. In another study involving blackberries (*Rubus ulmifolius Schott*) grown in Spain and Italy, esters and alcohols were the predominant class of volatiles followed by monoterpenes, aldehydes, and ketones (D’Agostino et al., 2015). The discrepancy among our results and other studies was not surprising as the volatile composition of blackberries varies due to genetics, ripening stage, harvest, and storage conditions as well as sample preparation and gas chromatography conditions (El Hadi et al., 2013; Qian & Wang, 2005). Although 2020 and 2021 had comparable number of

rain events (15 versus 16), the amount of rain was triple in 2021 as compared to 2020 indicating there was less sunny, dry days in 2021. In addition, extraction method can impact volatile identification and quantification when comparing values from different research. However, in our study conditions were optimized to achieve ideal results with all samples prepared the same using a DVB/CAR/PDMS SPME fiber (preferable for berry volatiles), 4 mL sample amount, 15 min pre-equilibrium time, 20 min extraction time, and 65 °C extraction temperature.

PCA was used to reduce the dimensionality of the data and to clarify relationships between compound classes and genotypes. The relative TIC peak areas (%) were summed for compounds within each compound class and aroma category. In terms of PCA in both years, PC1 had the most variation in the data followed by PC2. The PCA showed distinctions in blackberry genotypes and for compound classes and aroma categories.

The compounds with the most impactful aromas found by Wang et al. (2005) in ‘Chickasaw’ grown in Arkansas determined by their flavor dilution (FD) were ethyl butanoate, linalool, methional, ethyl 2-methylbutanoate,  $\beta$ -damascenone, geraniol, *allo*-ocimene, trans-2-hexenal, and 2,5-dimethyl-4-hydroxy-3(2H)-furanone; all of which had a FD  $\geq$  512. Du et al., calculated odour activity values (OAVs) and found furaneol, linalool,  $\beta$ -ionone, 2-heptanol, and carvone could be the compounds that contributed to the major aroma contributing compounds in blackberries grown in the Pacific Northwest. In contrast to Wang et al. (2005), methional,  $\beta$ -damascenone, *allo*-ocimene, ethyl 2-methylbutanoate, and 2,5-dimethyl-4-hydroxy-3(2H)-furanone were not detected in our Arkansas-grown blackberries nor by Morin et al. (2020) who also investigated volatiles in Arkansas-grown blackberries. These findings indicated that measurement of potent volatiles may be a better approach to screen blackberry genotypes for

improved aroma. In 2020 ‘Sweet-Ark<sup>®</sup> Ponca’ had the highest accumulative amount of these impactful compounds and in 2021 ‘Sweet-Ark<sup>®</sup> Caddo’ had the highest amount.

**2020.** There were 159 volatile aroma compounds identified across nine different compound classes in Arkansas blackberry genotypes in 2020 (Table 5). Compound categories included chemical, floral, fruity, green/fat, roasted/caramelized, vegetal alcohols, floral, green/fat, vegetal, and roasted/caramelized aldehydes, fruity and vegetal aromatic hydrocarbons, fruity esters, vegetal and fruity ketones, vegetal, fruity, floral, green/fat monoterpenes, floral norisoprenoids, and green/fat, and fruity sesquiterpenes.

Figures 2 shows the cumulative concentration of each class of volatile compounds for each Arkansas blackberry genotypes in 2020. In terms of total volatile compounds, ‘Ouachita’ (1,401 µg/kg) had the lowest and ‘Sweet-Ark<sup>®</sup> Caddo’ (28,430 µg/kg) had the highest. ‘Sweet-Ark<sup>®</sup> Caddo’, ‘Tupy’, and A-2620T had the three highest levels of total volatile compounds. Genotypes with less than 3,000 µg/kg total volatile compounds included ‘Natchez’, APF-409T, A-2528T, ‘Prime-Ark<sup>®</sup> Traveler’, and ‘Ouachita’.

The highest amounts of aroma compounds in the blackberries were mostly ethyl acetate in the aroma category fruity with an aroma descriptor of fruity, pineapple, and anise. ‘Sweet-Ark<sup>®</sup> Caddo’ (23,135 µg/kg), ‘Tupy’ (12,990 µg/kg), and A-2620T (9,062 µg /kg) had high ethyl acetate levels. There were blackberries with high levels of D-limonene, a monoterpene in the fruity aroma category with a citrus and mint aroma descriptor. A-2620T (1,504 µg/kg) and A-2625T (1,474 µg/kg) had the highest levels of D-limonene. There were blackberries with high levels of hexanal, an aldehyde in the green/fat aroma category with a green and herbal aroma descriptor. ‘Sweet-Ark<sup>®</sup> Caddo’ (2,602 µg/kg) had the highest levels of hexanal.

When a PCA was conducted on the compound class variables in 2020 (Figure 3), two components explained 54% of the variation in the data. PC1 (31.44%) had positive loadings for alcohols, ketones, sesquiterpenes, lactones, norisoprenoids, and aldehydes. Genotypes positively loaded for PC1 included ‘Osage’, A-2658T, ‘Ouachita’, A-2526T, A-2701T, and ‘Natchez’. Esters, monoterpenes, and aromatic hydrocarbons were all loaded negatively on PC1 along with genotypes ‘Sweet-Ark<sup>®</sup> Caddo’, APF-409T, ‘Sweet-Ark<sup>®</sup> Ponca’, A-2528T, A-2625T, A-2587T, A-2547T, ‘Prime-Ark<sup>®</sup> 45’, A-2610T, A-2620T, A-2701T, ‘Tupy’, ‘Prime-Ark<sup>®</sup> Traveler’, and ‘Prime-Ark<sup>®</sup> Horizon’. PC2 (25.24%) had positive loadings for aldehydes, monoterpenes, aromatic hydrocarbons, sesquiterpenes, alcohols, ketones, and lactones. Norisoprenoids and esters were negatively loaded for PC2. Most of the blackberry genotypes were clustered around the center showing little variation except ‘Osage’ which was at the far positive side of PC1, but around neutral of PC2.

Figure 4 shows the total concentration of the impactful volatile aroma compounds in 2020. Five of the ten impactful volatiles from Wang et al., (2005) were identified in Arkansas grown fresh-market blackberries in 2020. The compounds not found were methional,  $\beta$ -damascenone, ethyl 2-methylpropanoate, *allo*-ocimene, and 2,5-dimethyl-4-hydroxy-3(2H)-furanone. 2-hexenal a floral aldehyde and linalool, a floral monoterpene had the highest levels of the five impactful compounds that were quantified. The next highest impactful compound was ethyl 2-methylbutanoate, an ester with a fruity aroma.

**2021.** There were 103 volatile aroma compounds identified in Arkansas blackberry genotypes in 2021 (Table 6). Compound categories included chemical, floral, fruity, green/fat, roasted/caramelized, and vegetal alcohols, floral, green/fat, vegetal, and roasted/caramelized aldehydes, fruity and vegetal aromatic hydrocarbons, fruity esters, vegetal and fruity ketones,

vegetal, fruity, floral, and green/fat monoterpenes, floral norisoprenoids, and green/fat, and fruity sesquiterpenes. Figure 5 show the cumulative concentration of each class of volatile compounds for each blackberry genotype in 2021. In terms of total volatile compounds, 'A-2547' (1,273 µg/kg) had the lowest and A-2620T (5,886 µg/kg) had the highest. A-2658T (2,702 µg/kg) and 'Sweet-Ark<sup>®</sup> Caddo' (2,651 µg/kg) had the next highest levels of total volatile compounds. These volatiles were much lower values in 2021 as compared to 2020, possibly due to the amount of rain.

The highest quantified aroma compounds in the 2021 blackberries was D-limonene, a monoterpene with a fruity aroma category and citrus, mint aroma description. A-2620T (2,166 µg/kg) had the highest level of D-limonene followed by A-2658 (749 µg/kg). Ethyl acetate was the second most abundant volatile, detected in the 2021 blackberries. Ethyl acetate is in the aroma category fruity with an aroma descriptor of fruity, pineapple, and anise. A-2610T (498 µg/kg), A-2620T (467 µg/kg), and 'Ouachita' (258 µg/kg) had highest ethyl acetate levels. Overall, in both 2020 and 2021 blackberries, monoterpenes and esters were the largest classes of compounds in all blackberries, which characteristically have a fruity smell.

When a PCA was conducted on the compound class variables in 2021 (Figure 6), two components explained 64% of the variation in the data. PC1 (39.04%) had positive loadings for norisoprenoids, aromatic hydrocarbons, monoterpenes, and esters. Genotypes positively loaded for PC1 included A-2620T, A-2610T, A-2547T, 'Ouachita' and 'Sweet-Ark<sup>®</sup> Caddo'. Alcohols, aldehydes, sesquiterpenes, and ketones were negatively loaded on PC1 along with genotypes A-2701T, A-2658T, and 'Sweet-Ark<sup>®</sup> Ponca'. PC2 (25.41%) had positive loadings for all the compound classes along with selection A-2620T, A-2658T, and 'Sweet-Ark<sup>®</sup> Ponca'. Genotypes

A-2701T, A-2610T, A-2547T, 'Sweet-Ark<sup>®</sup> Caddo', and 'Ouachita' were negatively loaded for PC2.

Figure 7 shows the total concentration of impactful volatile aroma compounds in 2021. The same five of the ten impactful volatiles from the Wang et al., (2005) study in 2020 were identified in 2021. 2-hexenal, a floral aldehyde and linalool, a floral monoterpene, had the highest levels of the five impactful compounds. The next highest impactful compound was ethyl butanoate, an ester with a fruity aroma.

### **Descriptive sensory attributes**

The descriptive panel evaluated the blackberry purees for 17 attributes including four aromas (jam, berry, fruity, and vegetative), three basic tastes (sweet, sour, and bitter), eight aromatics (overall intensity, blackberry, earthy/dirty, green/unripe, overripe/fermented, chemical, mold/mildew, and metallic), and two feeling factors (astringent and metallic) (Table 7). The genotypes evaluated in 2020 were A-2547T, A-2625T, A-2701T, 'Ouachita', 'Sweet Ark<sup>®</sup> Caddo', and 'Sweet Ark<sup>®</sup> Horizon'. The genotypes evaluated in 2021 were A-2547T, A-2610T, A-2701T, 'Ouachita', 'Sweet Ark<sup>®</sup> Caddo', and 'Sweet Ark<sup>®</sup> Ponca', Four of the same genotypes were evaluated in both years. The lexicon developed by the descriptive panel included the term, definition, technique, and references used by the panelist to evaluate the aroma, aromatics, basic tastes, and feeling factors (Table 7). Descriptive sensory analysis involves a trained panel that uses a lexicon (terms to describe the product) and references to evaluate products on a line scale. Threlfall et al. (2016a) developed a fresh-market blackberry lexicon in an evaluation of UA System blackberries with eight appearance, three basic tastes, two feeling factors, and eight aromatics of the blackberries were evaluated. According to Threlfall et al. (2016b), the descriptive sensory panelists were not able to differentiate sweetness among five

blackberry genotypes; however, the panelists could easily differentiate sourness and overall aromatic impact. One of the issues with sensory of fresh-market blackberries is the variability from berry to berry. So, if each panelist only has a few berries to sample, then the variability between the panelists will be high. In our study, we used a puree of blackberry sample which provided the descriptive panel with a more unified sample for evaluation.

**2020.** The genotypes only differed in fruity aroma, green/unripe aromatics, and sourness (Table 8). For aroma, fruity aroma of ‘Sweet-Ark<sup>®</sup> Caddo’ (4.33) had the highest and ‘Prime-Ark<sup>®</sup> Horizon’ (2.59) the lowest, with jam (4.08), berry (5.55), and vegetative (2.03) aromas. The aromatic attributes (volatiles perceived by the olfactory system while a sample is in the mouth) of the blackberry purees included overall intensity, blackberries, earthy/dirty, green/unripe, overripe/fermented, chemical, mold/mildew, and metallic. The intensity of green/unripe was different between genotypes with ‘Ouachita’ (3.18) the highest and A-2701T (1.48) the lowest. There were no significant differences in the genotypes for overall intensity (5.82), blackberries (5.42), earthy/dirty (1.76), overripe/fermented (0.92), chemical (0.64), mold/mildew (0.17), or metallic (1.43) aromatics. Genotypes impacted sourness, but not sweetness (4.36) or bitterness (3.37). ‘Ouachita’ (7.49) was the sourest and A-2701T (4.05) the least sour. The panelists found the sweetness of the blackberries close to the reference with the value 5 = 5% sucrose solution and bitterness of 2 = 0.05% solution of caffeine in water. There was not a difference in genotypes for astringency (5.99) when compared to the standard of 6=0.53 g alum/500mL or metallic feeling factor (1.64), biting into tin foil for reference.

A PCA provided an indication of associations among the genotypes and the 17 sensory attributes of the pureed blackberry samples (Fig. 8). PC 1 (48.47%) and PC 2 (19.82%) combined accounted for 68% of variations in the data. ‘Ouachita’, ‘Prime-Ark<sup>®</sup> Horizon’, A-

2547T, and ‘Sweet-Ark<sup>®</sup> Caddo’ were positively loaded for PC1 with attributes of green/unripe aromatic, overall intensity, aromatic intensity, sour, bitter, chemical aromatic, metallic feeling factor, blackberry aromatic, astringency and metallic. A-2625T and A-2701T were negatively loaded for PC1 along with attributes vegetative aroma, mold/mildew aromatic, fruity aroma, earthy/dirty aromatic, overripe/fermented aromatic, jam aroma, sweet, and berry aroma.

**2021.** Panelists did not detect differences between genotypes for any aroma attributes with jam (5.21), berry (5.69), fruity (3.26), and vegetative (2.23) (Table 9). The aromatic attribute of overall intensity was significantly different between genotypes with ‘Ouachita’ (7.55) the highest and A-2701T (5.79) the lowest. There was no significant difference in the genotypes for blackberries (6.0), earthy/dirty (1.9), green/unripe (2.6), overripe/fermented (1.0), chemical (0.6), mold/mildew (0.1), or metallic (2.5) aromatics. The panelists found a differences between genotypes for all basic tastes. The sweetness of the blackberries ranged from 1.89 (A-2547T) to 3.54 (‘Sweet-Ark<sup>®</sup> Ponca’) with 2 = 2% sucrose solution and 5 = 5% sucrose solution. For sourness the blackberries ranged from 2.42 (‘Sweet-Ark<sup>®</sup> Ponca’) to 3.97 (‘Ouachita’) with 2 = 0.05% citric acid solution and 5 = 0.08% citric acid solution. Bitterness ranged from 1.56 (‘Sweet-Ark<sup>®</sup> Ponca’) to 2.53 (‘Ouachita’) where 2 = 0.05% solution of caffeine in water. There was not a difference in genotypes for astringency (4.09) when compared to the standard of 0.53 g alum/500mL water=6.0. or metallic felling factor (2016), biting into tin foil for reference.

A PCA provided an indication of associations among the genotypes and the 17 sensory attributes of the pureed blackberry samples (Fig. 9). PC 1 (50.38%) and PC 2 (23.39%) combined accounted for 73.77% of variations in the data. ‘Ouachita’, A-2610T, and A-2547T were positively loaded for PC1 and long with attributes of green/unripe, chemical, sour, bitter, overall aromatic intensity, metallic feeling factor, blackberries, mold/mildew, astringency,



vegetative, and metallic. ‘Sweet-Ark<sup>®</sup> Caddo’, A-2701, and ‘Sweet-Ark<sup>®</sup> Ponca’ were negatively loaded for PC1 along with attributes fruity aroma, earthy/dirty, overripe/fermented, jam aroma, sweet, and berry aroma.

## **Conclusions**

The physical, composition, volatile, and descriptive sensory attributes of Arkansas-grown fresh-market blackberries were evaluated. Nineteen genotypes (eight cultivars and 11 breeding selections) were harvested from the UA System Fruit Research Station in Clarksville, AR in 2020 and eight genotypes (three cultivars and five breeding selections) in 2021. The descriptive sensory attributes of six genotypes each year were evaluated. Genotype significantly impacted the physical, composition, and volatile aroma attributes of these blackberries at harvest. Although the physical and composition attributes varied, they were typical of previously reported values from other research done on these cultivars and selections. ‘Sweet-Ark<sup>®</sup> Ponca’ had highest soluble solids in both years (14-15%). There were 159 volatile aroma compounds identified in Arkansas blackberry genotypes in 2020 and 103 in 2021, mainly monoterpenes, esters, aldehydes, and alcohols. Total volatiles levels in 2020 were higher than values in 2021. ‘Sweet-Ark<sup>®</sup> Caddo’ (28,430 µg/kg) had the highest total volatiles in 2020 and A-2620T (5,886 µg/kg) had highest in 2021. In both years, five of the six impactful volatiles identified by Wang et al. (2005) were found in Arkansas-grown fresh-market blackberries, with linalool (floral aroma) in the highest concentration. In term of descriptive sensory, genotypes differed in fruity aroma, green/unripe aromatics, and sour basic tastes in 2020. ‘Sweet-Ark<sup>®</sup> Caddo’ had the highest fruity aroma. In 2021, the genotypes differed in overall intensity of aromatics and basic tastes. ‘Sweet-Ark<sup>®</sup> Caddo’ and ‘Ouachita’ had the highest overall intensity and ‘Sweet-Ark<sup>®</sup> Ponca’ had the highest sweetness. The combination of physical, composition, volatile, and

descriptive sensory analyses can be a useful tool to steer breeding decisions, help southern U.S. growers better market blackberries, and determine commercial potential of Arkansas-grown, fresh-market blackberries.

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**Table 1.** Physical and composition attributes of fresh-market blackberry genotypes, Clarksville, AR (2020).

Genotype <sup>z</sup>	Berry weight (g)	Berry length (mm)	Berry width (mm)	Firmness (N)	Soluble solids (%)	pH	Titrateable acidity (%) <sup>y</sup>	Soluble solids/titrateable acidity ratio
A-2526T	7.78 e-h	30.98 f-i	22.56 cd	5.10 ef	12.07 a-d	3.27 b-e	1.00 a-d	12.06 a-c
A-2528T	8.47 d-h	31.62 e-h	22.11 cd	5.75 ef	11.47 b-d	3.55 a-d	0.75 d	13.32 a-c
A-2547T	7.82 e-h	30.22 g-j	22.56 cd	6.23 ef	12.43 a-d	3.49 a-e	0.87 b-d	14.73 a-c
A-2587T	13.50 ab	41.56 ab	25.01 a-c	4.88 fg	<u>9.80 d</u>	3.58 a-c	0.84 c-d	11.83 a-c
A-2610T	8.88 d-h	32.11 e-g	22.60 cd	5.93 ef	13.20 ab	3.39 b-e	0.96 b-d	14.07 a-c
A-2620T	<b>15.15 a</b>	<b>43.75 a</b>	25.58 ab	<u>1.78 g</u>	11.90 b-d	3.55 a-d	0.75 d	15.94 a-c
A-2625T	8.86 d-h	32.95 d-g	23.82 a-d	8.10 bf	12.27 a-d	3.77 ab	0.79 d	15.95 a-c
A-2658T	13.01 a-c	38.78 a-c	<b>25.94 a</b>	5.84 ef	13.10 a-c	3.61 a-c	0.68 d	<b>20.82 a</b>
A-2701T	10.46 c-e	40.20 a-c	22.32 cd	<b>13.13 a</b>	13.17 ab	3.74 ab	0.73 d	18.36 ab
APF-409T	7.31 gh	30.61 f-j	<u>20.99 d</u>	11.22 ab	9.83 d	3.17 c-e	1.20 a-c	<u>8.20 c</u>
Natchez	10.07 d-f	37.76 b-d	22.07 cd	7.63 c-f	10.50 cd	3.05 de	1.24 a-b	8.55 c
Osage	<u>6.27 h</u>	26.01 ij	22.71 b-d	6.71 ef	12.87 a-c	<b>3.91 a</b>	<u>0.64 d</u>	20.06 a
Ouachita	7.04 h	26.73 h-j	22.18 cd	5.46 ef	12.50 a-c	<u>3.01 e</u>	<b>1.37 a</b>	9.33 bc
Prime-Ark <sup>®</sup> 45	7.66 f-h	32.76 d-g	21.04 d	7.68 c-f	10.63 b-d	3.37 b-e	0.87 b-d	12.43 a-c
Prime-Ark <sup>®</sup> Horizon	9.99 d-g	39.72 a-c	22.23 cd	10.07 a-d	11.70 b-d	3.21 c-e	0.87 b-d	13.52 a-c
Prime-Ark <sup>®</sup> Traveler	7.42 f-h	31.12 e-h	21.00 d	10.71 a-c	11.13 b-d	3.38 b-e	0.91 b-d	12.30 a-c
Sweet-Ark <sup>®</sup> Caddo	10.98 b-d	35.63 ef	24.01 a-c	8.18 b-e	12.03 a-d	3.49 a-e	0.90 b-d	13.64 a-c
Sweet-Ark <sup>®</sup> Ponca	6.87 h	<u>25.90 j</u>	23.33 a-d	7.11 d-f	<b>14.60 a</b>	3.68 a-c	0.76 d	19.95 a
Tupy	10.02 d-g	36.13 c-e	24.18 a-c	4.88 fg	12.10 a-d	3.58 a-c	0.93 b-d	13.13 a-c
<i>P-value</i>	<i>&lt;0.0001</i>	<i>&lt;0.0001</i>	<i>&lt;0.0001</i>	<i>&lt;0.0001</i>	<i>&lt;0.0001</i>	<i>&lt;0.0001</i>	<i>&lt;0.0001</i>	<i>&lt;0.0001</i>

<sup>z</sup> Genotypes were evaluated in triplicate. Means highlighted are highest value and means underlined are lowest. Means with different letters for each attribute are significantly different (p<0.05) using Tukey's Honestly Significant Difference test.

<sup>y</sup> Titrateable acidity expressed as % citric acid.



**Table 2.** Physical and composition attributes of fresh-market blackberry genotypes, Clarksville, AR (2021).

<sup>z</sup> Genotypes were evaluated in triplicate. Means highlighted are highest value and means underlined are lowest. Means with different

Genotype <sup>z</sup>	Berry weight (g)	Berry length (mm)	Berry width (mm)	Firmness (N)	Soluble solids (%)	pH	Titratable acidity (%) <sup>y</sup>	Soluble solids/titratable acidity ratio
A-2547T	6.25 e	26.93 ef	<u>20.92 d</u>	8.72 ab	10.53 bc	3.37 cd	0.74 ab	14.25 bc
A-2610T	7.99 d	30.88 de	21.61 cd	9.42 ab	11.60 b	3.39 cd	0.92 a	12.75 c
A-2620T	13.07 ab	<b>40.90 a</b>	23.87 ab	10.66 a	11.17 b	<u>3.13 d</u>	<b>0.93 a</b>	12.05 c
A-2658T	11.86 abc	35.62 bc	<b>25.96 a</b>	10.48 a	<u>9.03 c</u>	3.58 bc	0.64 abc	14.08 bc
A-2701T	<b>13.30 a</b>	39.96 ab	23.76 abc	<b>11.34 a</b>	10.50 bc	<b>4.16 a</b>	<u>0.36 c</u>	30.19 ab
Ouachita	9.72 cd	28.52 def	25.48 ab	<u>6.77 b</u>	9.60 bc	3.51 cd	0.86 a	<u>11.32 c</u>
Sweet-Ark <sup>®</sup> Caddo	10.42 bcd	32.78 cd	23.54 bc	7.48 b	11.53 b	3.98 ab	0.49 bc	24.27 abc
Sweet-Ark <sup>®</sup> Ponca	<u>6.07 e</u>	<u>24.81 f</u>	21.61 cd	7.64 b	<b>13.70 a</b>	4.02 a	0.48 bc	<b>32.24 a</b>
<i>P-value</i>	<i>&lt;0.0001</i>	<i>&lt;0.0001</i>	<i>&lt;0.0001</i>	<i>0.0002</i>	<i>&lt;0.0001</i>	<i>&lt;0.0001</i>	<i>&lt;0.0001</i>	<i>0.0009</i>

letters for each attribute are significantly different (p<0.05) using Tukey's Honestly Significant Difference test.

<sup>y</sup> Titratable acidity expressed as % citric acid.

**Table 3.** Individual and total sugars and organic acids of fresh-market blackberry genotypes, Clarksville, AR (2020).

<b>Genotype<sup>z</sup></b>	<b>Glucose (g/100 mL)</b>	<b>Fructose (g/100 mL)</b>	<b>Total sugars (g/100 mL)</b>	<b>Citric acid (g/100 mL)</b>	<b>Malic acid (g/100 mL)</b>	<b>Total organic acids (g/100 mL)</b>
A-2526T	2.66 b	2.08 b-d	4.75 b-f	0.77 bcd	0.32 b-d	1.09 b-d
A-2528T	2.47 b-e	2.43 b-d	4.91b-e	0.53 d-f	0.23 d-g	0.76 d-f
A-2547T	2.53 b-d	2.49 b-d	5.03 b-d	0.71 c-f	0.24 d-g	0.95 c-f
A-2587T	<u>1.79 f</u>	<u>1.85 d</u>	<u>3.63 f</u>	0.49 ef	0.32 b-d	0.82 d-f
A-2610T	2.77 b	2.72 bc	5.48 bc	0.73 b-e	0.35 bc	1.08 b-d
A-2620T	2.28 b-f	2.28 b-d	4.55 b-f	0.52 d-f	0.19 e-g	0.71 ef
A-2625T	2.50 b-d	2.42 b-d	4.92 b-e	0.64 c-f	0.17 fg	0.80 d-f
A-2658T	2.63 bc	2.57 b-d	5.21 bc	0.52 d-f	<u>0.14 g</u>	<u>0.66 f</u>
A-2701T	2.83 b	2.82 b	5.66 b	0.58 c-f	0.15 g	0.72 ef
APF-409T	1.81 ef	1.97 cd	3.78 ef	0.83 bc	<b>0.54 a</b>	1.37 b
Natchez	1.97 c-f	2.00 cd	3.97 d-f	1.00 b	0.28 b-e	1.28 bc
Osage	2.52 b-d	2.51 b-d	5.02 b-d	<u>0.44 f</u>	0.23 d-g	0.67 f
Ouachita	2.36 b-f	2.40 b-d	4.76 b-f	<b>1.29 a</b>	0.48 a	<b>1.78 a</b>
Prime-Ark <sup>®</sup> 45	2.21 b-f	2.22 b-d	4.42 c-f	0.64 c-f	0.22 d-g	0.86 d-f
Prime-Ark <sup>®</sup> Horizon	2.34 b-f	2.37 b-d	4.71 b-f	0.67 c-f	0.15 g	0.82 d-f
Prime-Ark <sup>®</sup> Traveler	2.29 b-f	2.35 b-d	4.64 b-f	0.67 c-f	0.38 b	1.04 b-e
Sweet-Ark <sup>®</sup> Caddo	2.18 b-f	2.22 b-d	4.40 c-f	0.59 c-f	0.23 d-g	0.81 d-f
Sweet-Ark <sup>®</sup> Ponca	<b>3.66 a</b>	<b>3.64 a</b>	<b>7.30 a</b>	0.71 c-f	0.21 e-g	0.91 d-f
Tupy	1.91 d-f	1.99 cd	3.91 d-f	0.76 b-e	0.27 c-f	1.03 c-e
<i>P-value</i>	<i>&lt;0.0001</i>	<i>&lt;0.0001</i>	<i>&lt;0.0001</i>	<i>&lt;0.0001</i>	<i>&lt;0.0001</i>	<i>&lt;0.0001</i>

<sup>z</sup> Genotypes were evaluated in triplicate. Means highlighted are highest value and means underlined are lowest. Means with different letters for each attribute are significantly different ( $p < 0.05$ ) using Tukey's Honestly Significant Difference test.

**Table 4.** Individual and total sugars and organic acids of fresh-market blackberry genotypes, Clarksville, AR (2021).

<b>Genotype<sup>z</sup></b>	<b>Glucose (g/100 mL)</b>	<b>Fructose (g/100 mL)</b>	<b>Total sugars (g/100 mL)</b>	<b>Citric acid (g/100 mL)</b>	<b>Malic acid (g/100 mL)</b>	<b>Total organic acids (g/100 mL)</b>
A-2547T	2.79 bc	2.65 bc	5.45 bc	0.60 abc	0.07 ab	0.67 abc
A-2610T	3.72 ab	3.51 ab	7.23 ab	0.42 bc	0.05 ab	0.47 bc
A-2620T	<b>3.93 a</b>	3.72 a	7.65 a	<b>0.87 a</b>	<b>0.09 a</b>	<b>0.96 a</b>
A-2658T	<u>2.38 c</u>	<u>2.32 c</u>	<u>4.70 c</u>	0.54 abc	0.04 b	0.58 abc
A-2701T	3.92 a	<b>3.77 a</b>	<b>7.69 a</b>	0.44 bc	<u>0.04 b</u>	0.48 bc
Ouachita	2.54 c	2.45 c	4.99 c	0.79 ab	0.07 ab	0.86 ab
Sweet-Ark <sup>®</sup> Caddo	2.93 abc	2.84 a-c	5.77 abc	<u>0.38 c</u>	0.04 b	<u>0.42 c</u>
Sweet-Ark <sup>®</sup> Ponca	2.73 bc	2.60 bc	5.33 bc	0.51 abc	0.06 ab	0.57 abc
<i>P-value</i>	<i>&lt;0.0001</i>	<i>&lt;0.0001</i>	<i>&lt;0.0001</i>	<i>0.0027</i>	<i>0.0046</i>	<i>0.0024</i>

<sup>z</sup> Genotypes were evaluated in triplicate. Means highlighted are highest value and means underlined are lowest. Means with different letters for each attribute are significantly different ( $p < 0.05$ ) using Tukey's Honestly Significant Difference test.

**Table 5.** Volatile aroma compounds identified in fresh-market blackberries grown at the University of Arkansas System Division of Agriculture Fruit Research Station, Clarksville, AR (2020).

Compound	Compound class	Measured retention index	Aroma category	Aroma discription	A-2526T	A-2528T	A-2547T	A-2587T	A-2610T	A-2620T	A-2625T	A-2658T	A-2701T	APF-409T	Natchez	Osage	Ouachita	Prime-Ark® 45	Prime-Ark® Horizon	Prime-Ark® Traveler	Sweet-Ark® Caddo	Sweet-Ark® Ponca	Tupy
1-Butanol	alcohol	642	fruity	banana, alcohol, sweet	0.00	0.00	0.00	0.00	0.00	0.00	0.00	19.44	0.00	0.00	20.53	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
1-Penten-3-ol	alcohol	665	green/fat	butter, fish, green cherry, herbal, spices	11.51	4.42	2.51	0.00	0.00	29.72	1.24	1.40	4.63	3.06	28.04	2.92	5.43	4.09	2.11	2.28	0.00	4.06	23.34
3-Buten-1-ol, 3-methyl	alcohol	717	fruity	roasted/carmelized	0.00	0.00	0.00	0.00	0.00	0.00	6.59	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	8.19	0.00
1-Butanol, 3-methyl	alcohol	720	green/fat	burnt, cocoa, floral, malt	5.80	0.00	19.54	0.00	0.00	0.00	4.70	5.33	11.21	9.07	0.00	13.89	4.97	11.93	11.99	0.00	35.56	18.69	25.32
1-Butanol, 2-methyl	alcohol	723	green/fat	fish oil, green, malt, onion, wine	3.95	0.00	6.14	0.00	0.00	0.00	3.35	6.55	5.91	3.30	0.00	5.83	5.78	11.76	6.75	0.00	13.29	6.27	5.82
2-Penten-1-ol	alcohol	769	-	-	3.90	0.00	0.00	0.00	0.00	0.00	6.00	0.00	0.00	0.00	0.00	10.41	0.00	0.00	0.00	0.00	0.00	3.36	0.00
3-Hexen-1-ol	alcohol	851	vegetal	burdock	43.82	0.00	0.00	48.19	0.00	0.00	48.25	0.00	0.00	36.96	41.85	0.00	34.01	20.99	13.77	0.00	30.56	0.00	19.71
2-Hexen-1-ol	alcohol	861	green/fat	blue cheese, vegetable	5.22	15.40	5.25	3.00	19.30	3.33	6.24	13.70	0.00	40.02	13.38	56.12	6.22	5.14	2.38	24.53	3.27	15.72	4.45
1-Hexanol	alcohol	863	green/fat	grass, herbal, banana	141.61	78.96	95.47	26.77	74.66	28.18	104.06	102.47	70.86	82.80	126.86	212.95	64.61	21.56	14.13	54.34	38.47	174.43	38.97
2-Heptanol	alcohol	895	vegetal	mushroom, herbal	391.78	30.66	26.69	26.69	13.64	54.53	135.97	652.39	159.30	69.63	593.08	51.88	49.26	59.46	29.00	2.30	278.66	75.36	83.01
1-Heptanol	alcohol	968	green/fat	chemical, green, fresh	6.66	5.78	2.32	4.03	6.50	3.67	12.99	0.00	5.10	5.24	7.48	6.99	6.39	5.79	5.68	4.73	9.33	8.70	6.47
1-Octen-3-ol	alcohol	978	green/fat	fat, floral, mushroom	2.28	2.71	8.26	2.57	1.90	0.00	8.16	2.21	2.88	4.00	4.76	2.96	4.17	1.52	0.00	2.54	3.78	5.61	3.75
1-Pentanol, 3-ethyl-4-methyl	alcohol	1019	-	-	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	8.11	0.00	0.00	4.77	0.00	0.00	0.00	0.00
1-Hexanol, 2-ethyl	alcohol	1028	green/fat	green, rose	53.34	102.32	72.88	58.91	49.14	69.02	10.50	54.41	80.91	63.91	100.03	10.35	37.00	55.95	47.17	48.63	16.77	44.95	29.69
1-Octanol	alcohol	1069	chemical	chemical, metal	9.22	10.46	8.67	6.73	8.43	4.20	14.53	16.68	30.39	6.66	19.83	28.32	8.27	9.28	6.39	3.93	17.01	23.76	14.56
Dihydro myrcenol	alcohol	1072	-	-	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.79
1-Nonanol	alcohol	1169	green/fat	fat, green	4.98	3.41	1.73	3.27	3.04	3.45	4.79	2.06	4.50	3.99	3.31	7.81	3.24	4.26	2.59	2.55	5.83	6.63	5.07
Camphenol, 6	alcohol	1177	-	-	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	19.28	43.53	24.92	0.00	17.40	24.15	0.00	6.79	0.00	0.00	0.00
1-Decanol	alcohol	1271	green/fat	fat	2.83	3.06	1.44	4.47	3.84	0.92	8.07	8.05	6.57	1.28	0.65	6.71	1.08	2.04	1.47	0.48	5.08	2.69	2.62
1-Undecanol	alcohol	1374	fruity	mandarin	2.70	5.77	0.00	0.00	0.00	0.00	0.00	3.93	1.31	0.00	0.00	2.65	0.00	0.00	0.00	4.56	0.00	1.41	0.00
Dodecanol	alcohol	1474	green/fat	fat	0.85	0.56	0.38	0.87	0.00	3.15	0.90	0.64	1.29	0.00	1.51	3.09	2.36	0.57	0.19	0.29	0.00	2.74	0.00

\*Realitive peak area percent Compounds were identified by comparison of mass spectra with NIST14 (National Institute of Standards and Technology, Gaithersburg, MD, USA), Flavors and Fragrances of Natural and Synthetic Compounds (FFNSC3, John Wiley & Sons, Inc., Hoboken, NJ, USA), and Adams Essential Oils (Adams 2007) mass spectral libraries and comparison of calculated Kovats retention indices ( Kovats 1958) with previously reported values

Table 5. continued

Compound	Compound class	Measured retention index	Aroma category	Aroma description	A-2526T	A-2528T	A-2547T	A-2587T	A-2610T	A-2620T	A-2625T	A-2658T	A-2701T	APF-409T	Natchez	Osage	Orachita	Prime-Ark® 45	Prime-Ark® Horizon	Prime-Ark® Traveler	Sweet-Ark® Caddo	Sweet-Ark® Ponca	Tupay
Butanal, 3-methyl	aldehyde	629	-	-	46.79	16.83	30.68	27.99	86.02	29.62	39.50	0.00	40.95	28.60	35.94	87.68	15.99	72.18	27.02	45.30	37.02	64.35	15.80
2-Butenal	aldehyde	622	roasted/caramelized	malt, chocolate	0.00	0.00	0.00	0.00	0.00	39.05	4.65	15.53	1.68	0.00	4.47	10.15	8.15	11.26	0.00	6.53	32.09	5.46	7.06
Butanal, 2-methyl	aldehyde	636	roasted/caramelized	almond, cocoa	27.39	14.15	24.65	15.45	34.03	31.45	31.49	0.00	38.07	29.68	0.00	80.20	17.41	53.13	20.58	33.55	86.65	23.10	28.56
Pentanal	aldehyde	677	roasted/caramelized	almond butter, malt, oil	0.00	7.32	0.00	0.00	0.00	0.00	12.20	0.00	17.75	0.00	0.00	0.00	77.81	12.49	37.71	31.98	41.57	34.03	0.00
2-Butenal, 2-methyl	aldehyde	741	-	-	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	4.33	0.00	0.00	0.00	0.00	0.00	0.00	0.00
2-Pentenal	aldehyde	744	-	-	3.56	1.60	0.00	0.00	0.00	5.25	0.00	2.67	1.59	6.14	10.49	4.96	8.75	3.55	3.44	4.64	6.30	5.27	3.35
Hexanal	aldehyde	792	green/fat	green, herbal	125.09	52.97	279.56	21.73	455.34	120.34	200.15	146.05	111.85	130.62	300.49	293.85	175.17	0.00	0.00	156.41	2602.53	253.73	703.73
2-Hexenal	aldehyde	845	floral	herbal tea, spearmint, wheat	0.00	0.00	0.00	0.00	0.00	0.00	7.47	8.50	5.58	2.79	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
2-Hexenal	aldehyde	850	floral	herbal tea, spearmint, wheat	323.29	190.82	116.58	10.40	282.20	90.51	291.50	604.27	313.59	147.15	151.55	1212.70	141.50	76.45	44.44	270.08	139.35	592.83	79.43
Styrene	aldehyde	893	green/fat	gasoline	0.00	0.00	0.00	0.00	0.00	0.00	0.00	2.41	0.00	2.97	0.00	0.00	0.00	0.00	0.00	0.00	2.77	2.08	3.50
Heptanal	aldehyde	902	green/fat	chemical, green, fresh	0.00	0.00	0.00	0.00	1.53	0.00	0.00	0.00	0.94	0.16	0.00	2.32	3.66	3.38	2.01	1.38	0.00	1.36	0.17
2,4-Hexadienal, (E,E)	aldehyde	909	green/fat	Olive, peanuts, caviar	10.46	9.56	2.11	2953.87	9.64	0.00	12.06	16.67	4.48	5.92	5.30	44.98	6.45	0.00	0.00	4.83	1.94	16.47	1.93
3-Hepten-1-ol	aldehyde	912	green/fat	green, herbal	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	3.42	0.00	0.00
Benzaldehyde	aldehyde	965	roasted/caramelized	almond, caramel	9.44	14.32	17.94	7.91	13.46	11.12	11.81	8.90	8.81	7.37	17.01	21.89	10.11	11.76	5.64	12.61	30.46	16.76	15.38
Octanal	aldehyde	1003	green/fat	fat, soap, green berry, geranium, honey, nut dandelion, fat	6.71	4.28	53.44	3.28	5.27	6.30	9.30	3.32	3.47	4.21	13.65	9.53	5.17	4.01	4.30	5.37	6.27	29.55	5.26
Phenylacetaldehyde	aldehyde	1050	green/fat	fruit, spice	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	4.83	0.00	0.00	0.00	5.19	0.00	4.36	0.00	0.00	0.00
2-Octenal	aldehyde	1059	green/fat	fat, citrus, green melon	2.12	0.00	0.00	2.17	4.60	5.54	1.90	0.00	2.35	4.53	0.76	3.40	1.77	4.22	0.00	1.15	2.51	2.13	1.45
Nonanal	aldehyde	1104	green/fat	fat, fish, orange	11.86	10.10	5.12	6.24	9.28	9.22	27.81	6.47	4.53	6.38	17.09	20.16	0.00	0.00	5.35	6.67	7.87	18.24	4.34
3-Nonen-1-ol	aldehyde	1156	green/fat	mushroom	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	1.39	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.25	0.00
Octanoic acid	aldehyde	1161	green/fat	cheese, grass	54.47	348.39	121.94	344.63	0.00	114.12	576.75	217.20	118.39	197.68	704.14	863.14	76.87	178.80	97.87	0.00	322.49	558.61	0.00
2-Nonenal	aldehyde	1165	green/fat	paper, soap, orange peel	0.70	1.97	0.67	0.00	0.00	1.02	0.00	0.00	0.91	0.00	0.00	0.00	1.08	0.94	0.00	0.64	0.00	0.42	0.00
Decanal	aldehyde	1206	green/fat	peel	7.37	12.00	5.16	42.14	5.09	5.23	16.77	2.31	10.83	4.88	10.48	14.80	3.25	4.01	2.15	5.37	0.00	13.18	0.00
2,4-Decadienal	aldehyde	1215	green/fat	coriander, fat	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	4.43	0.00	25.27	0.00	0.00	0.00	0.00	1.59	15.36
2-Decenal	aldehyde	1265	green/fat	fat, fish, orange basil, corn, soap, orange	1.91	0.00	0.79	1.31	2.05	3.42	8.17	1.52	1.91	2.11	5.43	7.04	1.86	1.43	1.98	2.01	2.54	5.29	2.53
Undecanal	aldehyde	1308	vegetal	celery	1.12	3.28	0.69	0.00	0.69	1.29	2.16	0.00	0.93	1.03	3.72	1.45	1.29	0.00	0.56	0.80	0.00	3.54	1.25
2-Undecenal	aldehyde	1367	fruity	orange, tea, cognac, herbal	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	6.06	0.00	0.00	0.00	0.00	0.00	0.00	0.00
trans-2-Hexenyl hexanoate	aldehyde	1384	green/fat	wax	0.00	0.00	0.00	0.00	0.00	0.00	1.25	0.00	0.00	0.00	0.00	6.28	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Dodecanal	aldehyde	1411	green/fat	citrus, fat, lily	1.19	0.00	0.00	0.56	0.00	0.67	0.77	0.00	0.00	0.00	1.64	0.88	0.00	0.00	0.00	0.00	1.11	1.51	0.30
2-Heptenal	aldehyde		-	-	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	2.89	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00

\*Relative peak area percent compounds were identified by comparison of mass spectra with NIST14 (National Institute of Standards and Technology, Gaithersburg, MD, USA), Flavors and Fragrances of Natural and Synthetic Compounds (FFNSC3, John Wiley & Sons, Inc., Hoboken, NJ, USA), and Adams Essential Oils (Adams 2007) mass spectral libraries and comparison of calculated Kovats retention indices (Kovats 1958) with previously reported values

Table 5. continued

Compound	Compound class	Measured retention index	Aroma category	Aroma description	A-2526T	A-2528T	A-2547T	A-2587T	A-2610T	A-2620T	A-2625T	A-2658T	A-2701T	APF-409T	Natchez	Osage	Onachita	Prime-Ark® 45	Prime-Ark® Horizon	Prime-Ark® Traveler	Sweet-Ark® Caddo	Sweet-Ark® Ponca	Thuy
Toluene	aromatic hydrocarbon	759	fruity	apple, spearmint, dill parsely, cherry, corn, bell pepper	11.03	61.14	94.00	56.66	89.03	58.06	6.05	73.80	41.85	22.99	40.10	6.12	35.97	87.32	36.58	174.55	24.52	165.25	128.12
Xylene alpha, Para-dimethylstyrene	aromatic hydrocarbon	886	vegetal	spicy, balsamic, musty	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	1.52	0.00	0.42
Terpinolene	aromatic hydrocarbon	1095	vegetal		6.02	52.09	17.78	70.63	60.64	91.49	103.74	43.94	19.80	8.81	25.95	14.05	6.50	3.43	0.91	2.72	21.85	34.73	9.91
Ethyl Acetate	ester	593	fruity	fruity, pineapple, anise	1446.52	303.14	4489.56	313.45	7174.16	9062.21	734.03	209.18	1473.81	1413.14	82.85	3733.83	220.60	2039.94	6015.05	846.38	23134.61	1122.45	12989.67
Ethyl propanoate	ester	699	fruity	apple, pineapple, strawberry	2.80	5.52	9.01	2.52	16.38	2.97	2.07	0.43	0.75	1.79	0.00	22.26	0.00	6.77	13.73	3.90	64.18	5.22	18.12
Methyl butanoate	ester	711	fruity	apple, banana, cheese, floral	0.62	11.35	6.44	6.75	9.19	0.00	0.00	0.48	0.00	0.00	0.00	11.30	0.00	4.66	13.46	4.59	47.75	4.87	9.17
Ethyl isobutanoate	ester	758	fruity	apple, pineapple	0.00	0.00	2.09	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	9.42	0.00	0.63	2.57	0.00	14.84	0.00	8.61
Methyl 2-methylbutanoate	ester	770	-	-	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	2.29	0.00	0.00	0.00	0.00	10.99	0.00	0.00	
2-Butenoic acid, ethyl ester	ester	784	-	-	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	7.98	0.00	0.00	0.00	0.00	0.00	0.00	0.00	
Butyl acetate	ester	815	fruity	apple, banana, glue	21.90	93.68	18.64	46.27	0.00	0.00	0.00	0.00	0.00	0.00	0.00	1331.40	0.00	0.00	518.27	28.97	0.00	85.36	0.00
2-Butenoic acid, ethyl ester	ester	841	-	-	3.28	0.00	25.75	0.00	39.04	0.00	1.08	0.00	0.00	0.00	173.60	0.00	5.53	10.98	17.97	124.93	0.53	23.70	
Ethyl 2-methylbutanoate	ester	851	fruity	apple, kiwi	0.00	0.00	8.54	0.00	0.00	0.00	0.00	0.00	0.00	0.00	84.64	0.00	3.36	13.88	4.80	111.24	7.83	22.35	
Methyl hexanoate	ester	921	fruity	fruit, fresh, paint thinner	1.12	5.52	0.72	1.40	6.72	1.22	2.17	1.80	0.35	0.00	1.12	17.15	0.00	1.42	1.87	1.13	7.59	2.18	4.26
Ethyl 3-hydroxybutyrate	ester	939	fruity	grape, coconut, marshmallow	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	16.51	0.00	0.00	1.20	0.00	10.13	0.82	3.04
Ethyl 2-methyl-2-butenate	ester	939	-	-	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	173.95	0.00	0.00	3.35	0.00	125.00	0.00	0.00	
Ethyl hexanoate	ester	998	fruity	apple peel, strawberry, anise	9.60	17.37	17.49	12.25	48.64	9.67	30.19	5.11	5.74	3.62	5.55	770.98	2.99	17.50	100.41	3.89	278.91	15.41	205.81
Hexyl acetate	ester	1010	fruity	apple, banana	0.72	0.32	0.19	0.00	0.00	0.00	0.00	0.58	0.25	0.15	0.51	0.56	0.14	0.09	0.06	0.18	0.00	0.28	0.00
2-Hexenyl acetate	ester	1013	fruity	apple, peach	0.51	0.00	0.30	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.86	0.00	0.00	0.11	0.35	0.00	0.00	
Methyl octanoate	ester	1122	fruity	fruit, orange, wax, wine	0.00	1.84	0.00	1.41	0.00	0.00	0.81	0.00	0.00	0.00	1.60	8.00	0.00	0.00	0.00	0.00	0.00	0.42	0.52
Ethyl 3-hydroxyhexanoate (4E,6Z)-allo-Ocimene	ester	1128	fruity	pear, red wine	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	2.63	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Neo-allo-ocimene	ester	1146	fruity	celery	10.22	0.00	0.00	0.00	0.00	0.00	1.67	25.86	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	35.16	0.00
Ethyl benzoate	ester	1179	fruity	chamomile, celery, flower	0.88	5.22	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	4.41	0.00	0.00	0.00	0.00	3.16	1.22	4.12
Hexyl butanoate	ester	1189	fruity	apple, citrus	4.88	0.00	0.00	0.00	0.00	0.00	0.00	30.20	0.00	0.00	0.00	11.12	0.00	0.00	0.63	0.00	0.00	0.00	0.00
2-Hexenyl butanoate	ester	1192	fruity	blueberry, blackberry	4.33	9.03	5.43	0.00	0.00	0.00	5.87	13.15	0.00	0.00	9.10	0.00	0.00	1.49	0.52	2.30	0.00	7.43	0.00
Ethyl octoate	ester	1194	fruity	apricot, brandy	2.91	18.24	6.47	0.00	9.25	0.00	25.54	0.00	0.00	2.56	3.80	354.54	2.15	2.29	5.54	1.36	14.56	10.48	38.15
Methyl salicylate	ester	1206	floral	wintergreen	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	1.67	0.00	1.03
Ethyl 2-octenoate	ester	1245	fruity	fruity, tropical	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	7.61	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Methyl decanoate	ester	1322	fruity	pear, blackberry	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	2.36	0.00	0.00	0.00	0.00	0.00	1.96	0.00
Hexyl hexanoate	ester	1385	fruity	apple, peach, plum	3.09	11.14	0.00	0.00	0.00	0.00	2.07	6.42	2.19	0.00	0.00	1.07	0.00	0.00	0.00	0.00	0.00	7.65	0.00
Ethyl decanoate	ester	1392	fruity	grape	2.06	0.00	3.49	0.00	3.65	1.44	4.00	0.65	0.34	0.00	0.95	46.25	0.00	0.54	1.33	0.00	4.93	8.95	5.50
Methyl dodecanoate	ester	1519	-	-	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.70	0.00	0.00	0.00	0.00	0.00	0.00	0.00	
Ethyl dodecanoate	ester	1591	fruity	mango, leaf	0.51	0.00	0.00	0.00	1.73	0.63	2.38	0.00	0.00	0.00	10.64	0.46	0.00	0.00	0.00	0.00	1.24	3.56	1.64
Ethyl butanoate	ester	-	-	-	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.45	1.29	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	
Ethyl 2-hexenoate	ester	-	-	-	0.00	0.00	1.94	0.00	1.61	0.00	0.00	0.00	0.00	0.00	19.85	0.00	1.79	1.37	0.65	10.78	1.11	2.36	

\*Realitive peak area percent Compounds were identified by comparison of mass spectra with NIST 14 (National Institute of Standards and Technology, Gaithersburg, MD, USA), Flavors and Fragrances of Natural and Synthetic Compounds (FFNSC3, John Wiley & Sons, Inc., Hoboken, NJ, USA), and Adams Essential Oils (Adams 2007) mass spectral libraries and comparison of calculated Kovats retention indices (Kovats 1958) with previously reported values

Table 5. continued

Compound	Compound class	Measured retention index	Aroma category	Aroma description	A-2536T	A-2538T	A-2547T	A-2587T	A-2610T	A-2620T	A-2625T	A-2688T	A-2701T	AlF-40T	Nutbez	Oxge	Oxthia	Prms-Ald@45	Prms-Ald@Hrian	Prms-Ald@Taveda	Sweet-Ald@Ckbb	Sweet-Ald@Pura	lupy
1-Penten-3-one	ketone	-	green/fat	fish, mustard	0.00	0.00	0.00	0.00	0.00	0.00	8.17	0.00	0.00	0.00	167.27	10.78	0.00	0.00	0.00	0.00	9.45	0.00	
2-Heptanone	ketone	890	vegetal	blue cheese, fruit, green, nut	11.83	2.85	2.93	0.49	0.00	4.36	6.80	34.46	12.92	3.12	17.36	3.03	2.81	2.25	0.00	0.00	10.02	2.61	5.06
5-Hepten-2-one, 6-methyl	ketone	987	vegetal	mushroom, earthy	2.79	5.23	0.89	0.00	0.00	0.00	0.00	0.00	1.63	1.80	3.04	5.47	1.36	1.90	0.00	1.93	0.00	1.15	0.00
Camphenone, 6	ketone	1130	-	-	6.07	0.00	0.00	2.81	0.00	0.00	9.77	0.00	0.00	5.34	5.41	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Camphor	ketone	1160	floral	mint	8.25	0.00	0.00	0.00	0.00	0.00	0.00	11.96	0.00	4.23	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	133.71
2-Undecanone	ketone	1294	fruity	orange, rose	1.18	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	1.34	0.72	0.00	0.00	0.00	0.00	0.00	2.30	0.42
gamma.-Undecanolactone	lactone	1480	fruity	apricot	0.00	3.68	0.00	0.00	0.00	0.00	13.26	0.00	0.00	0.00	86.93	28.75	0.00	0.00	0.00	0.00	0.00	0.00	14.47
3-Carene	monoterpene	928	green/fat	nutmeg	0.00	0.00	0.00	0.00	0.00	1.15	0.65	0.65	0.42	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.21	0.00
alpha.-Thujene	monoterpene	934	vegetal	herb, woody, green	0.00	12.15	38.33	33.34	29.26	47.42	53.00	29.23	12.42	0.00	0.00	0.00	0.00	8.20	0.00	0.00	0.00	4.18	10.23
alpha.-Pinene	monoterpene	939	floral	cedar, pine	43.69	285.61	18.06	1135.95	1883.42	3801.28	1300.90	859.10	455.13	127.99	53.01	0.00	90.06	105.28	0.00	70.34	0.00	490.99	760.71
Dehydrosabinene	monoterpene	960	-	-	0.00	0.00	0.00	29.56	0.00	0.00	0.00	14.58	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	
Camphene	monoterpene	955	green/fat	camphor, mothball, oil	1.90	10.90	17.74	41.38	32.37	61.17	38.23	35.81	19.05	11.77	0.00	0.00	8.68	9.15	0.00	5.02	12.85	13.21	17.24
beta.-Pinene	monoterpene	985	floral	pine, wood	0.00	0.00	0.00	143.47	138.18	358.96	151.14	89.53	33.53	0.00	0.00	15.11	0.00	0.00	0.00	0.00	0.00	44.62	101.15
beta.-Myrcene	monoterpene	992	fruity	balsamic, fruit, herb	34.39	35.64	9.99	59.06	64.59	143.69	199.17	94.39	20.69	24.12	28.92	133.56	15.32	14.38	10.94	0.00	52.93	138.32	36.66
alpha.-Phellandrene	monoterpene	1009	fruity	citrus, fresh, mint, pepper, spice	0.00	0.00	0.00	0.00	28.69	38.81	68.47	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	18.16	18.33	0.00
alpha.-Terpinene	monoterpene	1022	fruity	lemon	2.94	58.51	11.43	60.01	28.92	25.71	43.20	29.03	14.61	20.88	15.41	0.00	7.31	18.24	0.00	5.31	18.78	12.19	6.74
Cymene	monoterpene	1031	fruity	citrus, fresh, solvent	1.04	5.81	3.17	18.91	10.55	13.67	15.61	9.98	3.51	2.61	3.76	2.29	0.67	1.84	0.35	0.56	2.67	5.29	1.91
D-Limonene	monoterpene	1036	fruity	citrus, mint	57.89	165.23	160.30	846.62	742.64	1504.03	1473.53	758.81	254.73	94.11	99.54	223.14	67.85	109.19	9.85	30.94	266.74	371.78	235.72
m-Cymene	monoterpene	1045	fruity	sweet basil, blackcurrant, fruit	0.00	0.00	0.00	0.12	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Eucalyptol	monoterpene	1040	fruity	camphor.	0.00	0.00	545.82	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	1155.54
beta.-cis-Ocimene	monoterpene	1049	fruity	tea, celery	41.90	0.00	8.13	0.00	4.51	0.00	0.00	94.03	0.00	0.00	0.00	13.99	0.88	0.00	1.09	0.00	14.95	109.15	4.78
gamma.-Terpinene	monoterpene	1063	fruity	bitter, citrus	8.49	23.80	5.71	44.10	17.37	19.65	31.61	16.95	10.97	7.88	11.41	7.92	17.12	3.55	0.00	9.00	6.02	7.36	16.96
Linalool oxide	monoterpene	1078	-	-	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	3.97
Linalool	monoterpene	1100	floral	floral, lavender, Earl Grey tea	46.29	16.34	95.03	6.57	8.53	110.58	153.54	53.08	8.55	9.62	11.56	28.50	6.17	10.20	9.67	4.20	80.69	244.96	36.47
(4E,6E)-Allocimene	monoterpene	1131	-	-	12.70	0.00	0.00	4.94	0.00	0.00	5.00	14.31	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	19.61	0.00
L-Pinocarveol	monoterpene	1153	-	-	0.00	0.00	0.00	0.00	0.00	0.00	20.87	23.25	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Verbenol	monoterpene	1158	green/fat	green	0.00	0.00	0.00	0.00	5.94	0.00	0.00	0.00	1.35	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
alpha.-Phellandrene-8-ol	monoterpene	1175	-	-	0.00	0.00	0.00	43.96	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Verbenol	monoterpene	1158	green/fat	green	0.00	0.00	0.00	0.00	0.00	0.00	9.01	9.66	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
endo-Borneol	monoterpene	1187	green/fat	camphor, fragrant, green, polish	1.54	10.53	5.22	10.68	6.42	17.99	10.87	12.50	9.24	10.48	4.53	1.97	6.84	4.81	0.00	3.52	6.41	3.49	6.98
Menthol	monoterpene	1182	floral	mint	0.79	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	1.30	0.00	0.00	0.00	0.00	0.00	1.23	0.00
Terpinen-4-ol	monoterpene	1188	green/fat	earth, must, nutmeg, wood	0.00	23.11	17.55	91.03	23.93	38.47	50.68	0.00	13.51	9.26	18.60	0.00	4.16	7.33	0.00	3.43	12.08	19.98	12.07
p-Cymene-8-ol	monoterpene	1191	-	-	0.00	0.00	0.00	16.81	0.00	18.41	0.00	0.00	7.23	0.00	0.00	0.00	2.38	0.00	0.00	0.00	0.00	0.00	

\*Relative peak area percent Compounds were identified by comparison of mass spectra with NIST14 (National Institute of Standards and Technology, Gaithersburg, MD, USA), Flavors and Fragrances of Natural and Synthetic Compounds (FFNSC3, John Wiley & Sons, Inc., Hoboken, NJ, USA), and Adams Essential Oils (Adams 2007) mass spectral libraries and comparison of calculated Kovats retention indices (Kovats 1958) with previously reported values

Table 5. continued

Compound	Compound class	Measured retention index	Aroma category	Aroma description	A-2536I	A-2538I	A-2547I	A-2587I	A-2610I	A-2620I	A-2625I	A-2688I	A-2701I	Alf-409I	Natchez	Osage	Quachia	Prime-Alk6:4	Prime-Alk6:Hydro	Prime-Alk6:Flavodor	Sweet-Alk6:Clackb	Sweet-Alk6:Purca	Italy
alpha.-Terpineol	monoterpene	1200	green/fat	anise, fresh, mint, oil	15.03	21.85	26.05	32.82	17.73	67.45	45.34	24.78	15.12	13.23	18.65	12.52	15.05	7.45	4.71	9.28	39.85	56.16	16.74
Myrtenol	monoterpene	1208	green/fat	mint, cool	1.49	4.72	0.00	7.61	0.00	0.00	5.26	11.96	2.83	4.03	5.10	8.29	2.02	4.73	0.00	0.00	7.33	3.27	0.00
Nerol	monoterpene	1224	fruity	floral, fruit	1.28	0.00	0.00	0.00	0.00	0.00	0.00	10.64	0.00	0.00	14.69	4.61	0.00	2.28	0.00	0.00	22.18	8.09	0.00
Pinocamphone	monoterpene	1224	floral	spearmint	0.00	0.00	0.00	79.88	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Verbenone	monoterpene	1228	-	-	0.00	0.00	0.00	0.00	8.20	8.73	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Citronellol	monoterpene	1233	floral	rose, citrus, clove	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.78	0.79	0.00	0.00	0.00	0.00
methyl carvacrol	monoterpene	1250	floral	camphor, spice, wood	0.00	10.38	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	4.66	0.00
D-Carvone	monoterpene	1256	-	-	0.00	13.02	10.78	36.53	11.67	21.28	39.72	20.16	18.58	26.50	0.00	0.00	12.66	21.25	0.00	6.77	0.00	0.00	19.22
Geraniol	monoterpene	1258	floral	geranium, lemon peel, passion fruit, peach, rose	11.04	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	19.85	16.19	0.00	0.00	7.26	0.00	0.00	35.26	0.00
Geranial	monoterpene	1274	-	-	1.02	1.92	0.00	3.08	0.00	0.00	3.31	2.53	1.75	0.00	0.00	2.88	0.00	0.88	0.00	0.00	5.57	3.42	0.00
Carvacrol	monoterpene	1285	vegetal	caraway, spice, thyme	0.00	0.00	0.00	5.61	0.00	0.00	0.00	4.80	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Perillic alcohol	monoterpene	1299	green/fat	fat, green, pungent	0.00	11.65	0.00	0.00	0.00	0.00	8.16	13.43	0.00	0.00	0.00	0.00	0.00	6.15	0.00	0.00	0.00	0.00	0.00
Thymol or Carvacrol	monoterpene	1304	-	-	0.00	0.00	0.00	9.80	0.00	0.00	6.30	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Perillic alcohol	monoterpene	1299	green/fat	fat, green, pungent	0.00	0.00	0.00	28.71	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Geranylacetone	monoterpene	1458	fruity	fruit	1.52	4.71	1.45	2.06	1.10	2.87	1.49	1.00	0.53	1.14	2.16	2.18	0.88	1.35	0.49	1.08	0.00	2.71	0.95
Dehydrosabinene	monoterpene	960	-	-	0.00	144.87	268.54	421.68	100.59	24.36	102.31	148.96	63.32	51.82	0.00	0.00	12.31	28.17	0.00	9.96	40.90	92.19	0.00
beta.-Ocimene	monoterpene	1050	-	-	14.48	3.71	0.00	23.94	0.00	16.12	23.83	0.00	0.00	0.00	5.51	0.00	0.00	0.00	1.34	0.00	0.00	79.96	0.00
Theaspirane B	norisoprenoids	1335	floral	honey	4.62	3.85	2.24	1.17	2.63	3.78	0.00	0.00	0.00	1.35	4.21	8.73	3.27	11.58	12.58	5.83	0.00	0.00	8.82
beta.-Ionone	norisoprenoids	1502	floral	floral, violet	1.15	0.54	0.00	0.00	0.00	0.78	0.00	0.71	0.83	0.00	1.73	0.59	0.00	0.00	0.64	0.00	0.00	0.71	0.89
Theaspirane B	norisoprenoids	1335	-	-	3.91	5.64	1.34	1.61	1.84	4.82	0.00	2.18	0.00	0.72	2.61	7.85	2.19	8.53	11.05	5.85	0.92	0.00	7.31
delta.-Elemene	sesquiterpenes	1351	-	-	0.43	0.00	0.00	0.00	0.00	0.00	0.44	0.00	0.00	0.00	0.41	0.00	0.00	0.00	0.00	0.00	0.00	2.56	0.41
alpha.-Cubebene	sesquiterpenes	1367	green/fat	herbal, wax	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	4.18	0.00
delta.-Elemene	sesquiterpenes	1381	-	-	0.00	0.00	0.00	0.00	0.00	0.00	0.60	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	2.84	0.00
Ylangene	sesquiterpenes	1393	-	-	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	1.08	0.00	0.00	0.00	0.00	0.00	0.00
Copaene	sesquiterpenes	1398	floral	spice, wood	1.44	0.46	0.59	0.95	1.02	1.74	1.88	0.82	0.00	0.00	1.34	0.63	1.78	0.00	0.55	0.00	1.27	4.78	1.01
beta.-Panasinsene	sesquiterpenes	1428	floral	tea	1.21	0.00	0.59	0.74	0.95	1.17	1.82	0.82	0.30	0.00	0.92	0.76	1.21	0.00	0.50	0.00	1.11	6.11	1.24
Cadinene	sesquiterpenes	1448	-	-	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.96	0.00
alpha.-Himachalene	sesquiterpenes	1482	fruity	apple, oregano, anise	0.00	0.00	0.00	0.00	0.00	0.00	0.15	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	1.43	0.00
alpha.-Caryophyllene	sesquiterpenes	1491	floral	wood	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.25	0.52	0.00	0.00	0.00	0.00	0.00	0.00	0.00
germacrene D	sesquiterpenes	1499	floral	spice, wood	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.41	0.00
delta.-Cadinene	sesquiterpenes	1542	floral	thyme, wood	0.59	0.00	0.35	0.64	1.18	4.11	1.80	1.22	0.20	0.00	0.53	0.80	0.57	0.00	0.35	0.00	0.93	1.76	0.36
trans-Calamenene	sesquiterpenes	1545	-	-	0.88	0.00	0.26	2.14	0.44	0.00	1.16	0.50	0.18	0.00	0.63	0.68	3.09	0.00	0.29	0.00	5.90	2.50	0.49
Cubenene	sesquiterpenes	1558	fruity	lemon, orange, mint	0.29	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.17	0.00
alpha.-Calacorene	sesquiterpenes	1572	-	-	0.49	0.00	0.00	0.40	0.00	0.65	0.34	0.32	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.53	0.80	0.13
alpha.-Cubebene	sesquiterpenes	1660	green/fat	herbal, wax	1.67	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.64
Di-epi-1,10-cubanol	sesquiterpenes	1661	-	-	0.00	0.00	0.00	0.00	0.00	0.00	18.43	6.37	0.00	0.00	10.61	8.70	0.00	0.00	0.00	0.00	0.00	25.05	0.00
alpha.-Cadinol	sesquiterpenes	1669	-	-	5.60	0.00	0.00	3.29	0.00	0.00	8.99	1.71	0.00	0.00	6.85	0.00	0.00	0.00	0.00	0.00	0.00	29.56	0.00
Cadalene	sesquiterpenes	1707	-	-	0.69	0.00	0.84	0.69	0.00	0.00	0.25	0.29	0.17	0.00	0.95	0.84	1.57	0.00	0.18	0.00	2.16	1.68	0.38
Di-epi-1,10-cubanol	sesquiterpenes	1661	-	-	0.00	0.00	0.00	0.00	0.00	0.00	18.43	6.37	0.00	0.00	10.61	8.70	0.00	0.00	0.00	0.00	0.00	25.05	0.00
alpha.-Cadinol	sesquiterpenes	1669	-	-	5.60	0.00	0.00	3.29	0.00	0.00	8.99	1.71	0.00	0.00	6.85	0.00	0.00	0.00	0.00	0.00	0.00	29.56	0.00
Cadalene	sesquiterpenes	1707	-	-	0.69	0.00	0.84	0.69	0.00	0.00	0.25	0.29	0.17	0.00	0.95	0.84	1.57	0.00	0.18	0.00	2.16	1.68	0.38

\*Relative peak area percent Compounds were identified by comparison of mass spectra with NIST14 (National Institute of Standards and Technology, Gaithersburg, MD, USA), Flavors and Fragrances of Natural and Synthetic Compounds (FNESC3, John Wiley & Sons, Inc., Hoboken, NJ, USA), and Adams Essential Oils (Adams 2007) mass spectral libraries and comparison of calculated Kovats retention indices (Kovats 1958) with previously reported values



**Table 6.** Volatile aroma compounds identified in fresh-market blackberries grown at the University of Arkansas System Division of Agriculture Fruit Research Station, Clarksville, AR (2021).

Compound	Compound Class	Measured retention index	Aroma category	Aroma discription	A-2547T	A-2610T	A-2620T	A-2658T	A-2701T	Ouachita	Sweet-Ark® Caddo	Sweet-Ark® Ponca
1-Butanol	alcohol	666	fruity	banana, alcohol, sweet	3.00	0.00	0.00	6.27	8.40	2.73	0.00	2.05
1-Penten-3-ol	alcohol	683	green/fat	butter, fish, green	0.00	0.00	0.00	0.00	2.17	1.49	0.00	2.93
3-Buten-1-ol, 3-methyl-	alcohol	731	fruity	cherry, herbal, spices	0.00	0.00	3.87	0.00	2.72	0.00	0.00	5.84
1-Butanol, 3-methyl	alcohol		roasted/carmelized	burnt, cocoa, floral, malt	0.00	0.00	0.00	5.39	0.00	7.73	0.00	1.25
1-Pentanol	alcohol	763	-	-	3.00	3.14	2.79	2.37	3.58	5.39	1.47	2.66
2-Penten-1-ol	alcohol	767	-	-	1.18	0.40	0.97	0.96	1.03	1.46	0.00	1.63
2-Hexenol	alcohol		-	-	15.54	8.03	0.00	0.00	0.00	4.08	5.63	0.00
1-Hexanol	alcohol	865	green/fat	grass, herbal, banana	30.69	22.46	29.65	45.02	41.54	23.62	20.39	73.99
2-Heptanol	alcohol	896	vegetal	mushroom, herbal	39.93	5.26	47.59	277.32	135.64	94.90	116.80	50.19
1-Heptanol	alcohol	966	green/fat	chemical, green, fresh	0.00	2.47	1.11	1.16	1.28	2.58	0.39	4.99
1-Octen-3-ol	alcohol	977	green/fat	fat, floral, mushroom	2.44	1.82	4.47	1.80	3.74	5.15	1.90	2.48
3-Ethyl-4-methyl-1-pentanol	alcohol	1021	-	-	1.60	0.00	0.00	0.00	6.62	0.00	4.06	0.49
1-Hexanol, 2-ethyl-	alcohol	1026	green/fat	green, rose	8.70	10.97	0.00	8.56	7.92	6.47	5.35	6.38
1-Octanol	alcohol	1066	chemical	chemical, metal	5.24	7.49	0.00	29.57	36.91	14.29	14.08	26.61
3-Nonen-1-ol	alcohol	1149	green/fat	mushroom	0.00	0.00	0.00	0.00	3.48	0.00	0.00	3.88
1-Nonanol	alcohol	1167	green/fat	fat, green	3.92	3.72	4.88	4.47	9.06	5.87	4.26	6.44
Geraniol	alcohol	1254	floral	spice, wood	17.68	10.74	66.15	8.81	22.03	15.43	0.00	28.86
1-Decanol	alcohol	1268	green/fat	fat	1.08	1.35	3.00	7.05	17.00	2.13	11.17	3.12
1-Dodecanol	alcohol	1471	green/fat	fat	1.28	1.01	2.50	0.00	2.78	3.79	1.27	3.15

\*Relative peak area percent Compounds were identified by comparison of mass spectra with NIST14 (National Institute of Standards and Technology, Gaithersburg, MD, USA), Flavors and Fragrances of Natural and Synthetic Compounds (FFNSC3, John Wiley & Sons, Inc., Hoboken, NJ, USA), and Adams Essential Oils (Adams 2007) mass spectral libraries and comparison of calculated Kovats retention indices ( Kováts 1958) with previously reported values

**Table 6. continued**

Compound	Compound Class	Measured retention index	Aroma category	Aroma discription	A-2547T	A-2610T	A-2620T	A-2658T	A-2701T	Ouachita	Sweet-Ark® Caddo	Sweet-Ark® Ponca
2-Butenal	aldehyde	627	roasted/carmelized	malt, chocolate	0.00	0.00	1.17	0.00	0.00	0.00	0.00	12.22
Butanal, 3-methyl-	aldehyde	660	-	-	7.22	5.88	7.77	6.59	17.31	4.18	4.70	14.09
Butanal, 2-methyl	aldehyde	669	roasted/carmelized	almond, cocoa	11.99	0.00	5.90	7.40	0.00	5.26	11.97	4.49
3-Buten-2-one, 3-methyl-	aldehyde	677	-	-	0.00	0.00	0.00	10.11	0.00	0.00	7.98	58.10
Pentanal	aldehyde	698	roasted/carmelized	almond butter, malt, oil	24.56	19.60	31.29	6.27	12.81	18.64	24.83	10.12
2-Pentenal	aldehyde	754	-	-	0.00	0.00	3.86	3.65	2.35	4.11	0.00	1.75
Hexanal	aldehyde	799	green/fat	green, herbal	64.74	63.96	99.68	0.00	67.81	148.51	0.00	124.04
2-Hexenal,	aldehyde	845	floral	herbal tea, spearmint, wheat	0.77	0.00	2.26	2.71	2.61	2.09	0.00	4.16
2-Hexenal	aldehyde	853	floral	herbal tea, spearmint, wheat	132.90	39.73	124.95	76.13	206.31	93.44	130.56	183.18
Heptanal	aldehyde	900	green/fat	chemical, green, fresh	1.57	1.99	2.36	0.99	1.38	2.91	1.58	1.39
2,4-Hexadienal	aldehyde	911	green/fat	Olive, peanuts, caviar	4.04	4.42	2.09	0.00	2.41	1.91	0.00	2.01
2-Heptenal	aldehyde	957	-	-	0.00	0.23	1.35	0.48	1.21	0.00	0.42	0.52
Benzaldehyde	aldehyde	968	-	-	13.30	9.42	10.96	10.59	9.26	12.97	11.83	7.89
Octanal	aldehyde	1002	green/fat	fat, soap, green	5.77	6.60	9.90	4.03	5.83	10.66	8.09	6.80
Phenylacetaldehyde	aldehyde	1051	green/fat	berry, geranium, honey, nut	0.00	0.00	0.00	0.00	10.20	0.00	0.00	0.00
2-Octenal,	aldehyde	1059	green/fat	dandelion, fat, fruit, spice	3.44	1.39	3.58	4.34	2.91	5.14	3.17	3.19
Nonanal	aldehyde	1103	green/fat	green	15.07	13.79	23.19	9.17	15.96	21.54	15.76	15.91
Octanoic acid	aldehyde	1155	green/fat	cheese, grass	0.00	217.72	181.39	205.13	360.55	170.26	356.60	250.12
2-Nonenal,	aldehyde	1161	green/fat	paper	2.00	1.11	2.77	2.49	3.62	2.21	1.39	2.93
Decanal	aldehyde	1205	green/fat	soap, orange peel	10.27	9.79	13.14	5.57	16.42	16.41	8.24	13.74
2,4-Decadienal	aldehyde	1217	green/fat	coriander, fat	1.46	2.41	0.00	0.00	3.34	3.90	0.00	1.26
2-Decenal,	aldehyde	1263	green/fat	orange.	3.13	3.01	4.71	3.57	2.51	5.69	5.48	3.29
Undecanal	aldehyde	1306	vegetal	basil, corn, celery	0.81	1.07	2.25	0.00	2.68	2.64	1.92	1.90
Dodecanal	aldehyde	1408	green/fat	-	0.94	1.47	1.55	1.18	1.54	2.04	0.91	1.24

\*Realitive peak area percent Compounds were identified by comparison of mass spectra with NIST14 (National Institute of Standards and Technology, Gaithersburg, MD, USA), Flavors and Fragrances of Natural and Synthetic Compounds (FNNSC3, John Wiley & Sons, Inc., Hoboken, NJ, USA), and Adams Essential Oils (Adams 2007) mass spectral libraries and comparison of calculated Kovats retention indices ( Kováts 1958) with previously reported values

Table 6. continued

Compound	Compound Class	Measured retention index	Aroma category	Aroma discription	A-2547T	A-2610T	A-2620T	A-2688T	A-2701T	Ordinia	Sweet-Ark®C4hb	Sweet-Ark®Purna
Toluene	aromatic hydrocarbon	771	fruity	apple, spearmint, dill	3.99	4.10	3.26	3.94	2.74	2.87	3.35	2.79
.alpha.,Para-dimethylstyrene	aromatic hydrocarbon	1097	vegetal	spicy, balsamic, musty	17.99	17.29	294.33	51.94	39.64	14.03	37.49	38.18
Ethyl Acetate	ester	615	fruity	fruity, pineapple, anise	93.26	497.62	466.90	55.07	237.95	258.37	257.19	62.23
Ethyl propionate	ester	711	fruity	apple, pineapple, strawberry	0.00	0.00	1.30	1.04	0.00	0.00	3.37	0.73
Methyl butanoate	ester	721	fruity	apple, banana, cheese, floral	0.00	0.00	0.00	2.21	0.00	0.00	4.13	0.87
Ethyl butanoate	ester	-	-	-	0.00	0.00	0.00	27.82	0.00	0.00	173.48	0.00
Butyl acetate	ester	-	fruity	apple, banana, glue	0.00	0.00	0.00	1.75	0.00	4.19	0.00	0.20
Ethyl (2E)-2-butenolate	ester	842	-	-	0.00	2.44	0.00	0.00	0.00	0.81	12.84	1.28
Ethyl 2-methylbutanoate	ester	850	fruity	apple, kiwi	0.00	0.26	0.00	0.00	1.22	0.00	12.74	1.39
Methyl hexanoate	ester	922	fruity	fruit, fresh, paint thinner	0.73	0.46	0.63	2.54	0.76	0.24	2.30	0.78
Pentanoic acid, 4-methyl	ester	931	-	-	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.47
Ethyl 3-hydroxybutyrate	ester	933	fruity	grape, coconut, marshmellow	0.00	1.26	0.00	0.00	0.00	0.00	4.05	0.00
Ethyl (2E)-2-methyl-2-butenolate	ester	-	-	-	0.00	1.70	0.00	0.00	0.00	0.00	17.02	0.00
Hexanoic acid	ester	-	-	-	82.09	202.58	132.37	0.00	0.00	91.20	252.96	21.88
Ethyl hexanoate	ester	995	fruity	apple peel, strawberry, anise	3.35	4.44	4.78	7.69	3.72	2.11	70.29	7.71
Hexyl acetate / Hexyl ethanoate	ester	1008	-	-	0.04	0.02	0.20	0.32	0.22	0.08	0.11	0.08
Methyl octanoate	ester	1121	fruity	-	0.00	1.06	0.00	0.00	3.52	1.70	0.00	4.48
Ethyl benzoate	ester	1177	fruity	chamomile, celery, flower	0.00	0.00	0.00	0.00	0.00	0.00	4.00	0.00
Hexyl butanoate	ester	1189	fruity	blueberry, blackberry	0.00	0.00	0.00	22.77	18.94	0.00	0.00	17.69
Ethyl octanoate	ester	-	-	brandy	0.00	1.17	0.00	0.00	0.00	2.93	37.36	0.00
Methyl salicylate	ester	1208	floral	wintergreen	0.55	0.00	1.12	0.00	0.00	0.97	4.08	1.11
Hexyl hexoate	ester	1382	-	-	3.44	0.00	0.00	5.23	3.74	0.00	2.64	0.00
2-Hexenyl hexanoate	ester	-	-	apple, peach, plum	0.00	0.00	0.00	5.01	0.59	0.00	2.52	0.00
Ethyl decanoate	ester	1389	fruity	grape.	0.00	2.27	0.00	0.00	0.55	1.68	11.58	4.83

\*Realitive peak area percent Compounds were identified by comparison of mass spectra with NIST14 (National Institute of Standards and Technology, Gaithersburg, MD, USA), Flavors and Fragrances of Natural and Synthetic Compounds (FNNSC3, John Wiley & Sons, Inc., Hoboken, NJ, USA), and Adams Essential Oils (Adams 2007) mass spectral libraries and comparison of calculated Kovats retention indices ( Kováts 1958) with previously reported values

**Table 6. continued**

Compound	Compound Class	Measured retention index	Aroma category	Aroma discription	A-2547T	A-2610T	A-2620T	A-2658T	A-2701T	Ouachita	Sweet-Ark® Caddo	Sweet-Ark® Ponca
2-Heptanone	ketone	889	vegetal	blue cheese, fruit, green, nut	2.68	1.82	3.76	21.78	33.60	5.28	3.61	1.82
5-Hepten-2-one, 6-methyl-	ketone	985	vegetal	mushroom, earthy	2.80	2.80	3.41	2.92	5.82	2.57	2.35	4.25
2-Undecanone	ketone	1291	fruity	orange, rose	0.00	0.41	0.00	0.00	0.93	0.65	0.90	0.42
.alpha.-Thujene	monoterpene	933	vegetal	herb, woody, green	0.00	0.00	37.46	28.59	10.05	5.67	0.00	7.11
alpha.-Pinene	monoterpene	943	floral	cedar, pine	14.93	172.91	920.27	383.92	172.12	43.46	136.83	43.89
Camphene	monoterpene	962	green/fat	camphor, mothball, oil	2.82	2.56	13.82	7.69	5.28	2.33	3.00	6.82
Dehydrosabinene	monoterpene		-	-	18.65	0.00	0.00	163.37	68.90	0.00	0.00	0.00
beta.-Myrcene	monoterpene	991	fruity	balsamic, fruit, herb citrus, fresh, mint,	173.57	11.32	306.76	82.61	60.63	287.25	161.14	70.26
alpha.-Phellandrene	monoterpene	1012	fruity	pepper, spice	0.00	0.00	98.72	0.00	21.17	0.00	0.00	22.83
.alpha.-Terpinene	monoterpene	1024	fruity	lemon	7.14	2.75	71.11	13.15	14.62	6.40	8.37	14.36
Cymene	monoterpene	1032	fruity	blackcurrant, fruit	1.86	1.57	16.32	6.73	3.35	1.13	2.56	2.85
<b>D-Limonene</b>	<b>monoterpene</b>	<b>1037</b>	<b>fruity</b>	<b>citrus, mint</b>	<b>74.93</b>	<b>165.60</b>	<b>2166.32</b>	<b>748.86</b>	<b>283.46</b>	<b>70.81</b>	<b>297.47</b>	<b>118.50</b>
(Z)-beta-Ocimene	monoterpene	1039	fruity	tea, celery	0.00	0.00	39.30	0.00	0.00	0.00	111.85	6.35
trans-.beta.-Ocimene	monoterpene	1048	fruity	- lavender, Earl Grey	14.64	0.00	0.00	81.08	4.20	5.43	0.00	50.63
Linalool	monoterpene	1100	floral	tea	212.38	2.93	237.78	51.70	11.31	18.78	142.13	191.95
4,6-Allocimene	monoterpene	1130	-	-	2.22	1.72	7.11	6.22	1.94	1.28	9.32	6.25
4,6-Allocimene	monoterpene	1145	-	-	2.43	0.00	6.96	0.00	0.54	3.29	10.98	5.16
Verbenol	monoterpene	1177	-	-	8.49	3.55	0.00	8.38	6.13	4.48	0.00	4.40
Menthol	monoterpene	1182	floral	mint	0.00	0.00	0.00	0.00	0.00	0.00	2.45	0.00

\*Realitive peak area percent Compounds were identified by comparison of mass spectra with NIST14 (National Institute of Standards and Technology, Gaithersburg, MD, USA), Flavors and Fragrances of Natural and Synthetic Compounds (FFNSC3, John Wiley & Sons, Inc., Hoboken, NJ, USA), and Adams Essential Oils (Adams 2007) mass spectral libraries and comparison of calculated Kovats retention indices ( Kováts 1958) with previously reported values

**Table 6. continued**

Compound	Compound Class	Measured retention index	Aroma category	Aroma discription	A-2547T	A-2610T	A-2620T	A-2658T	A-2701T	Ouachita	Sweet-Ark® Caddo	Sweet-Ark® Ponca
endo-Borneol	monoterpene	1183	green/fat	camphor, fragrant, green, polish	3.18	3.47	5.52	6.66	4.60	3.42	0.00	1.74
Terpinen-4-ol	monoterpene		green/fat	-	23.27	12.68	85.65	42.93	6.96	12.93	9.96	1.96
p-Cymene-8-ol	monoterpene	1193	-	-	7.16	0.00	43.04	3.50	0.00	0.00	0.00	13.18
L-.alpha.-Terpineol	monoterpene	1201	-	anise, fresh, mint, oil	29.61	4.88	136.99	16.59	11.59	10.84	32.00	31.03
Myrtenol	monoterpene	1213	green/fat	mint, cool	0.00	1.95	0.00	10.91	11.54	0.00	0.00	0.00
Nerol	monoterpene	1231	fruity	fruity, floral	0.00	0.00	0.00	19.92	0.00	0.00	8.03	19.03
methyl carvacrol	monoterpene	1249	floral	lemon peel	0.00	0.00	22.01	0.00	0.00	0.00	0.00	9.28
Carvone	monoterpene	1257	floral	-	14.92	8.59	20.22	4.42	15.53	9.30	0.00	4.46
Geranial	monoterpene	1273	-	-	1.07	0.89	5.34	2.07	2.71	1.11	2.25	2.09
Perilla alcohol	monoterpene	1300	green/fat	fat, grenn, pungent	0.00	0.00	15.07	34.07	6.22	5.05	4.25	3.47
Geranylacetone	monoterpene	1455	fruity	-	3.11	1.42	6.00	0.00	11.19	2.34	2.84	8.83
Theaspirane	norisoprenoid	1322	floral	honey	3.06	1.56	2.97	0.50	0.00	2.80	1.16	0.00
Theaspirane	norisoprenoid	1337	floral	honey	2.76	1.36	1.77	0.88	0.00	1.37	0.60	0.00
alpha.-Cubebene	sesquiterpene	1368	green/fat	herbal, wax	0.00	0.00	0.00	0.00	0.00	1.71	0.00	2.37
Ylangene	sesquiterpene	1395	-	-	0.00	0.43	1.09	0.92	1.18	1.10	0.00	1.39
Copaene	sesquiterpene	1399	floral	-	0.00	0.80	1.87	2.70	0.72	1.67	0.00	2.32
.beta.-Panasinsene	sesquiterpene	1430	floral	-	0.30	1.12	1.38	2.67	0.63	0.72	1.08	3.26

\*Realitive peak area percent Compounds were identified by comparison of mass spectra with NIST14 (National Institute of Standards and Technology, Gaithersburg, MD, USA), Flavors and Fragrances of Natural and Synthetic Compounds (FFNSC3, John Wiley & Sons, Inc., Hoboken, NJ, USA), and Adams Essential Oils (Adams 2007) mass spectral libraries and comparison of calculated Kovats retention indices ( Kováts 1958) with previously reported values

**Table 7.** Lexicon used by a trained descriptive sensory panel (n=7-9) to evaluate attributes of purees of different Arkansas-grown fresh-market blackberry genotypes grown in Clarksville, AR (2020 and 2021).

Term	Definition	Technique	Reference
<b>Aroma</b>			
Jam	Sweet aroma reminiscent of fruit/berry jam	Blackberry jam	Universal Aromatic Scale <sup>z</sup>
Berry	Aroma associated with any type of berry	Fresh blackberries	Universal Aromatic Scale
Fruity	Aroma associated with a mixture of non-specific fruits (not including berries): apples/ pears, tropical, melons, banana	Peach tea, hibiscus tea, fruit punch	Universal Aromatic Scale
Vegetative	Aroma associated with fresh vegetables and herbs	Fresh cut grass	Universal Aromatic Scale
<b>Basic tastes</b>			
Sweet	Basic taste, perceived on the tongue, stimulated by sugars and high potency sweeteners	Solutions of sucrose in spring water	2% = 2.0, 5% = 5.0, 10% = 10.0, 16% = 15.0
Sour	Basic taste, perceived on the tongue, stimulated by acids, such as citric acid	Solutions of citric acid in spring water	0.05% = 2.0, 0.08% = 5.0, 0.15% = 10.0, 0.20% = 15.0
Bitter	Basic taste, perceived on the tongue, stimulated by substances such as caffeine	Solutions of caffeine in spring water	0.05% = 2.0, 0.08% = 5.0, 0.15% = 10.0, 0.20% = 15.0
<b>Aromatics</b>			
Overall intensity	Overall impact of all aromatics in the berry	Combination of all aromatics	Universal Aromatic Scale
Blackberry	Aromatic associated with blackberries	Fresh blackberries, blackberry jam	Universal Aromatic Scale
Earthy/dirty	Aromatic associated with damp soil or wet foliage	Damp potting soil, allspice	Universal Aromatic Scale
Green/unripe	Aromatic associated with freshly cut green vegetation; unripe banana	Unripe banana	Universal Aromatic Scale
Overripe/fermented	Aromatic associated with overripe fruit	Over ripened fruit	Universal Aromatic Scale
Chemical	Off-flavors associated with petroleum, sulfur, wet paper, paint, alcohol, soap, resin, solvents, wax, etc., having a distinctly “chemical” nature	Combination of a variety of chemical off-flavors, magic marker, turpentine	Universal Aromatic Scale
Mold/mildew	Aromatic associated with moldy or mildew aromas	Old, mildewed clothes	Universal Aromatic Scale
Metallic	Aromatic associated with metals, tin, or iron	Canned pineapple (sniff can only)	Universal Aromatic Scale
<b>Feeling factors</b>			
Astringent	Feeling factor on the tongue or other skin surfaces of the mouth described as puckering or drying	Chew sample to point of swallow, expectorate and feel surfaces of the mouth. Swish references in mouth, swallow or expectorate and wait 5 seconds.	0.53 g alum/500mL water=6.0
Metallic	Flat chemical feeling factor stimulated on the tongue and teeth by metal (coins, tin foil).	Tin foil to bite	Universal Aromatic Scale

<sup>z</sup> Intensities based on Universal Scale (Saltine = 3.0; Applesauce = 7.0; Orange juice = 10.0; Grape juice = 14.0; Big Red Gum<sup>®</sup> = 15.0)

**Table 8.** Descriptive sensory attributes of University of Arkansas System Division of Agriculture grown fresh-market blackberries, Clarksville, AR (2020).

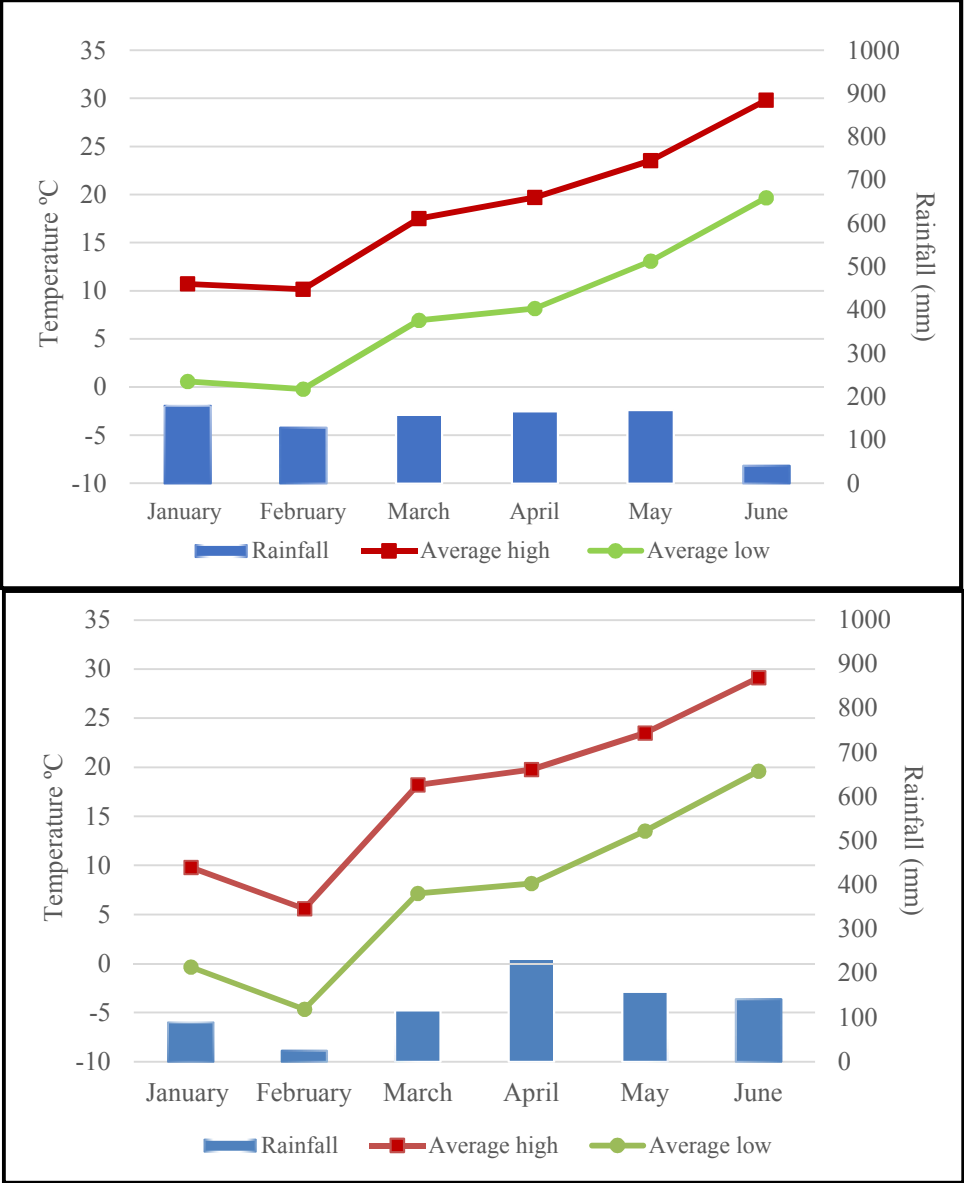
<b>Genotype<sup>z</sup></b>	<b>A-2547T</b>	<b>A-2625T</b>	<b>A-2701T</b>	<b>Ouachita</b>	<b>Prime- Ark<sup>®</sup> Horizon</b>	<b>Sweet-Ark<sup>®</sup> Caddo</b>	<b>P-value</b>
<b>Aroma</b>							
Jam	3.79 a	3.92 a	4.68 a	3.43 a	4.14 a	4.53 a	0.3046
Berry	5.38 a	5.71 a	5.68 a	5.14 a	5.33 a	6.07 a	0.7259
Fruity	3.38 ab	3.19 ab	3.19 ab	2.76 b	2.59 b	4.33 a	0.0251
Vegetative	1.74 a	2.57 a	2.01 a	2.28 a	1.84 a	1.74 a	0.2941
<b>Aromatics</b>							
Overall intensity	5.79 a	5.13 a	5.34 a	6.54 a	6.03 a	6.08 a	0.0752
Blackberries	5.47 a	5.05 a	5.28 a	5.75 a	5.40 a	5.57 a	0.6650
Earthy/dirty	1.64 a	1.78 a	1.61 a	1.47 a	2.14 a	1.91 a	0.7049
Green/unripe	2.56 ab	1.90 ab	1.48 b	3.18 a	2.66 ab	2.37 ab	0.0507
Overripe/fermented	0.58 a	0.67 a	1.38 a	0.57 a	1.16 a	1.17 a	0.5373
Chemical	0.58 a	0.67 a	0.25 a	0.89 a	0.65 a	0.82 a	0.4649
Mold mildew	0.18 a	0.47 a	0.00 a	0.00 a	0.01 a	0.34 a	0.0167
Metallic	1.37 a	1.04 a	1.17 a	1.62 a	1.94 a	1.42 a	0.2174
<b>Basic tastes</b>							
Sweet	3.99 a	4.21 a	5.18 a	3.97 a	4.39 a	4.39 a	0.2252
Sour	5.94 ab	5.57 ab	4.05 b	7.49 a	5.82 ab	6.04 ab	0.0145
Bitter	2.96 a	3.06 a	2.78 a	4.25 a	3.14 a	3.37 a	0.3460
<b>Feeling factors</b>							
Astringency	5.12 a	5.16 a	4.90 a	5.83 a	5.81 a	5.99 a	0.3851
Metallic feeling factor	1.80 a	1.33 a	1.22 a	1.56 a	1.63 a	1.64 a	0.5751

**Table 9.** Descriptive sensory attributes of University of Arkansas System Division of Agriculture grown fresh-market blackberries, Clarksville, AR (2021).

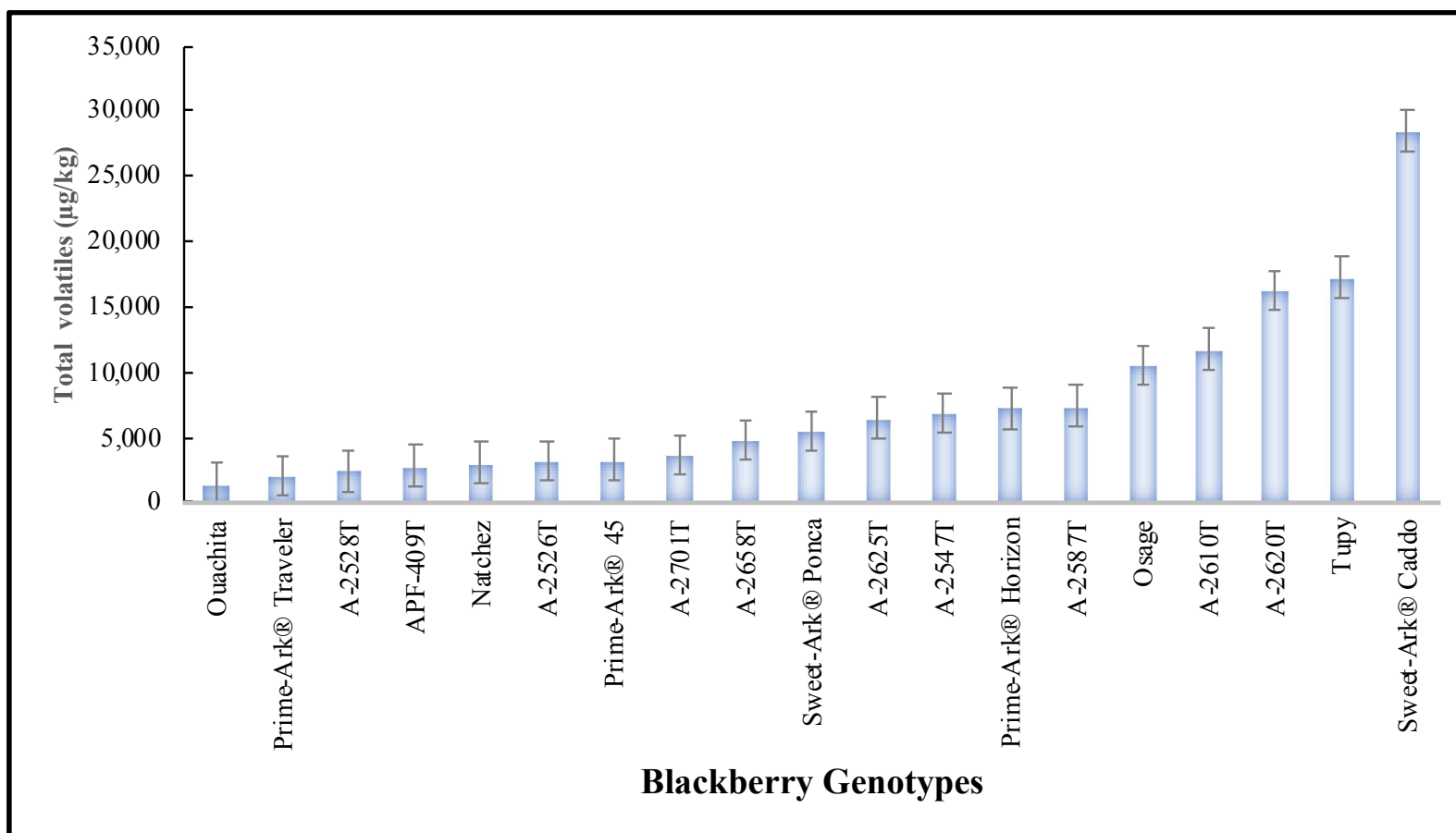
<b>Genotype<sup>z</sup></b>	<b>A-2547T</b>	<b>A-2610T</b>	<b>A-2701T</b>	<b>Ouachita</b>	<b>Sweet-Ark<sup>®</sup> Caddo</b>	<b>Sweet-Ark<sup>®</sup> Ponca</b>	<b><i>P-value</i></b>
<b>Aroma</b>							
Jam	5.04 a	4.50 a	5.30 a	4.72 a	6.18 a	5.54 a	0.2004
Berry	5.62 a	5.34 a	5.98 a	5.14 a	6.03 a	6.00 a	0.2681
Fruity	2.98 a	2.86 a	3.32 a	3.31 a	3.74 a	3.32 a	0.4693
Vegetative	1.88 a	2.04 a	2.00 a	2.29 a	1.64 a	2.23 a	0.4374
<b>Aromatics</b>							
<b>Overall intensity</b>	<b>6.71 ab</b>	<b>7.28 a</b>	<b>5.79 b</b>	<b>7.55 a</b>	<b>7.32 a</b>	<b>6.46 ab</b>	<b>0.0009</b>
Blackberries	5.79 a	6.42 a	5.54 a	6.36 a	6.29 a	5.75 a	0.4323
Earthy/dirty	1.84 a	1.67 a	2.14 a	1.64 a	2.89 a	1.46	0.2897
Green/unripe	2.68 a	2.71 a	2.20 a	3.20 a	2.54 a	1.96 a	0.1224
Overripe/fermented	0.96 a	0.57 a	0.97 a	0.68 a	1.11 a	1.43 a	0.5332
Chemical	0.89 a	0.71 a	0.45 a	1.09 a	0.46 a	0.18 a	0.1498
Mold mildew	0.11 a	0.00 a	0.00 a	0.07 a	0.14 a	0.00 a	0.4775
Metallic	2.61 a	2.71 a	2.56 a	2.72 a	2.29 a	2.06 a	0.3672
<b>Basic tastes</b>							
<b>Sweet</b>	<b>1.89 b</b>	<b>2.41 b</b>	<b>2.54 ab</b>	<b>2.09 b</b>	<b>2.24 b</b>	<b>3.54 a</b>	<b>0.0002</b>
<b>Sour</b>	<b>3.40 ab</b>	<b>3.67 a</b>	<b>3.03 ab</b>	<b>3.97 a</b>	<b>3.49 ab</b>	<b>2.42 b</b>	<b>0.0028</b>
<b>Bitter</b>	<b>2.20 ab</b>	<b>2.22 ab</b>	<b>2.07 ab</b>	<b>2.53 a</b>	<b>2.32 a</b>	<b>1.56 b</b>	<b>0.0081</b>
<b>Feeling factors</b>							
Astringency	5.04 a	4.61 a	4.95 a	4.34 a	4.68 a	4.09 a	0.6587
Metallic feeling factor	2.77 a	2.68 a	2.51 a	2.40 a	2.48 a	2.16 a	0.4721

<sup>z</sup>Genotypes were evaluated in triplicate. Means with different letters for each attribute are significantly different ( $p < 0.05$ ) using Tukey's Honestly Significant Difference test. Highlighted attributes are significant.

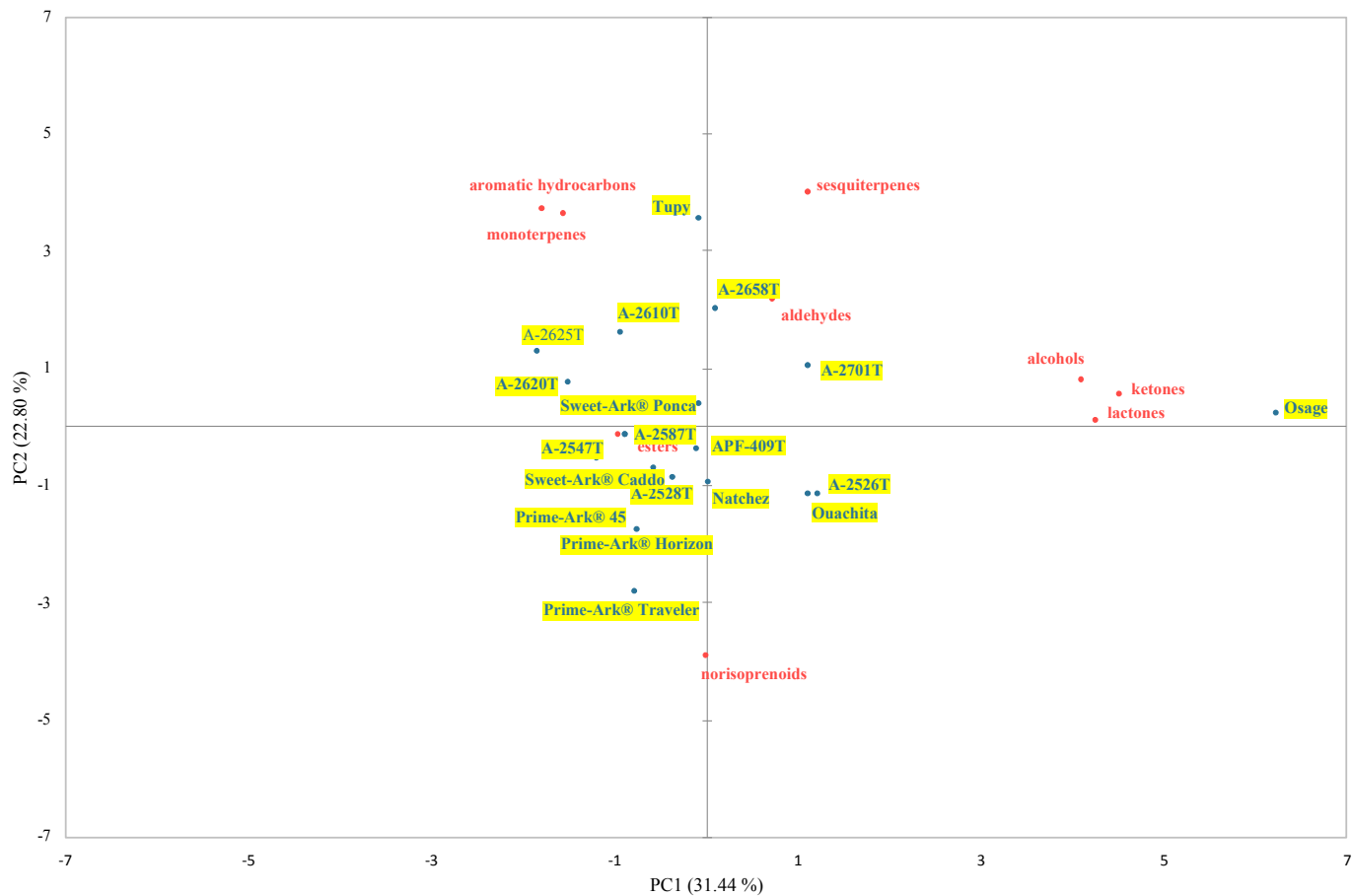




**Fig. 1.** Temperature and rain conditions at the University of Arkansas System Division of Agriculture Fruit Research Station, Clarksville, AR (2020 top and 2021 bottom).



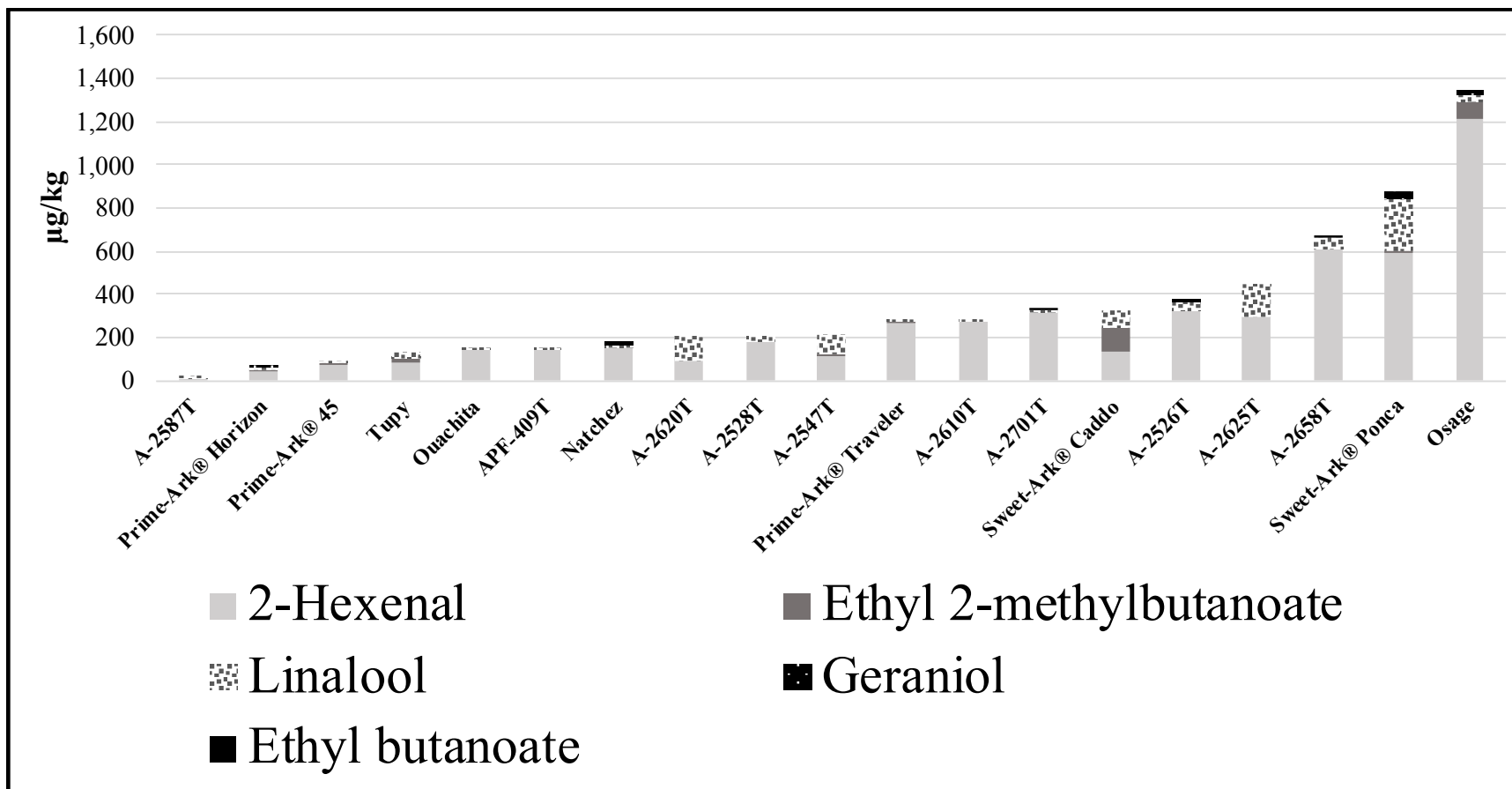
**Fig. 2.** Total volatile aroma compounds of fresh-market blackberry genotypes, Clarksville, AR (2020). Total volatile aroma compounds are the cumulative concentration of each class of volatile aroma compounds for each genotype.



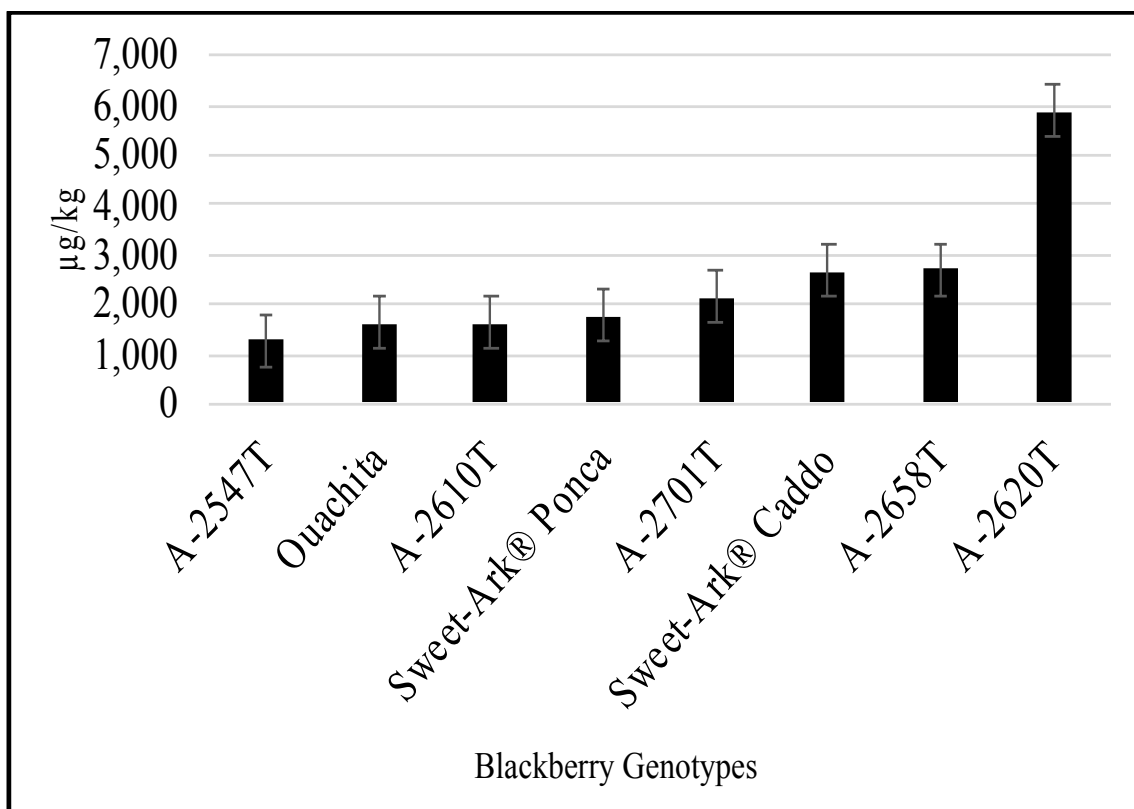
**Fig. 3.** Principal components (PC) analysis on volatile aroma compounds in fresh-market blackberries grown at the University of Arkansas System Division of Agriculture Fruit Research Station, Clarksville, AR (2020).

*Percent of variation in data explained by each component.*

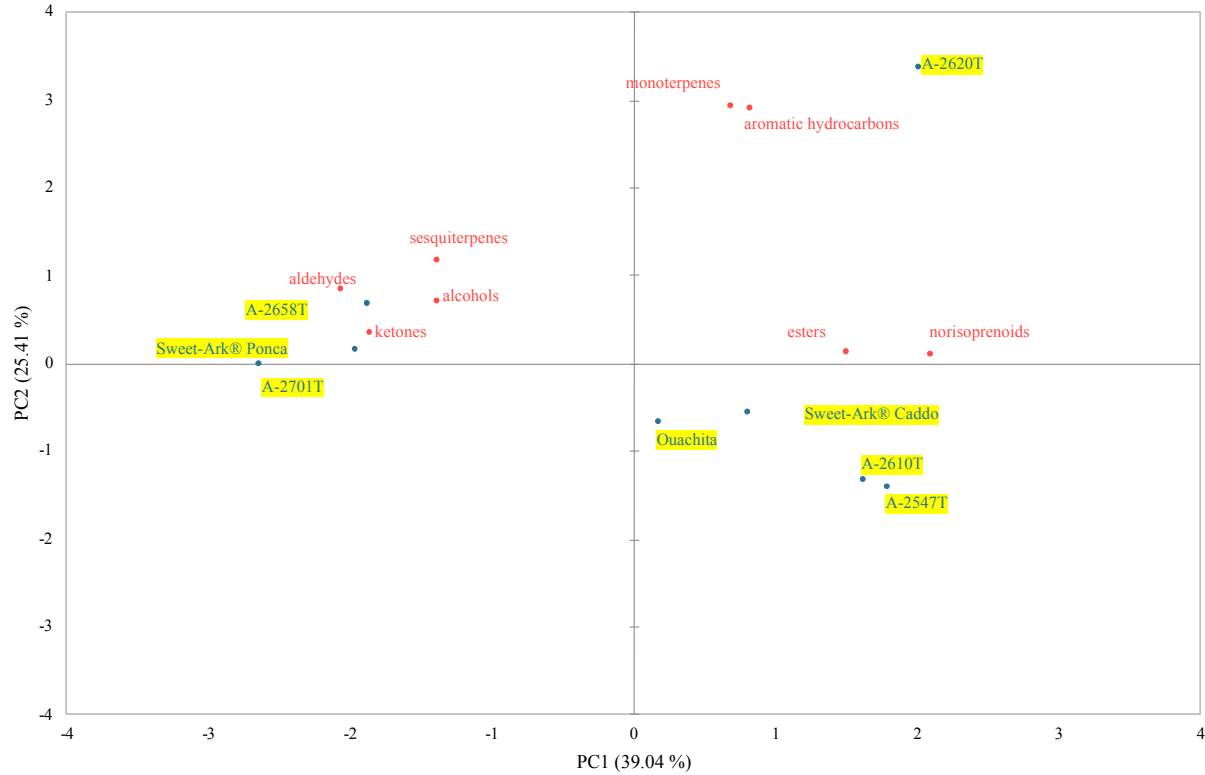
*Compound class variables represent the sum of the total ion chromatogram (TIC) relative peak areas (%) of positively identified compounds within each compound class (Table 5).*



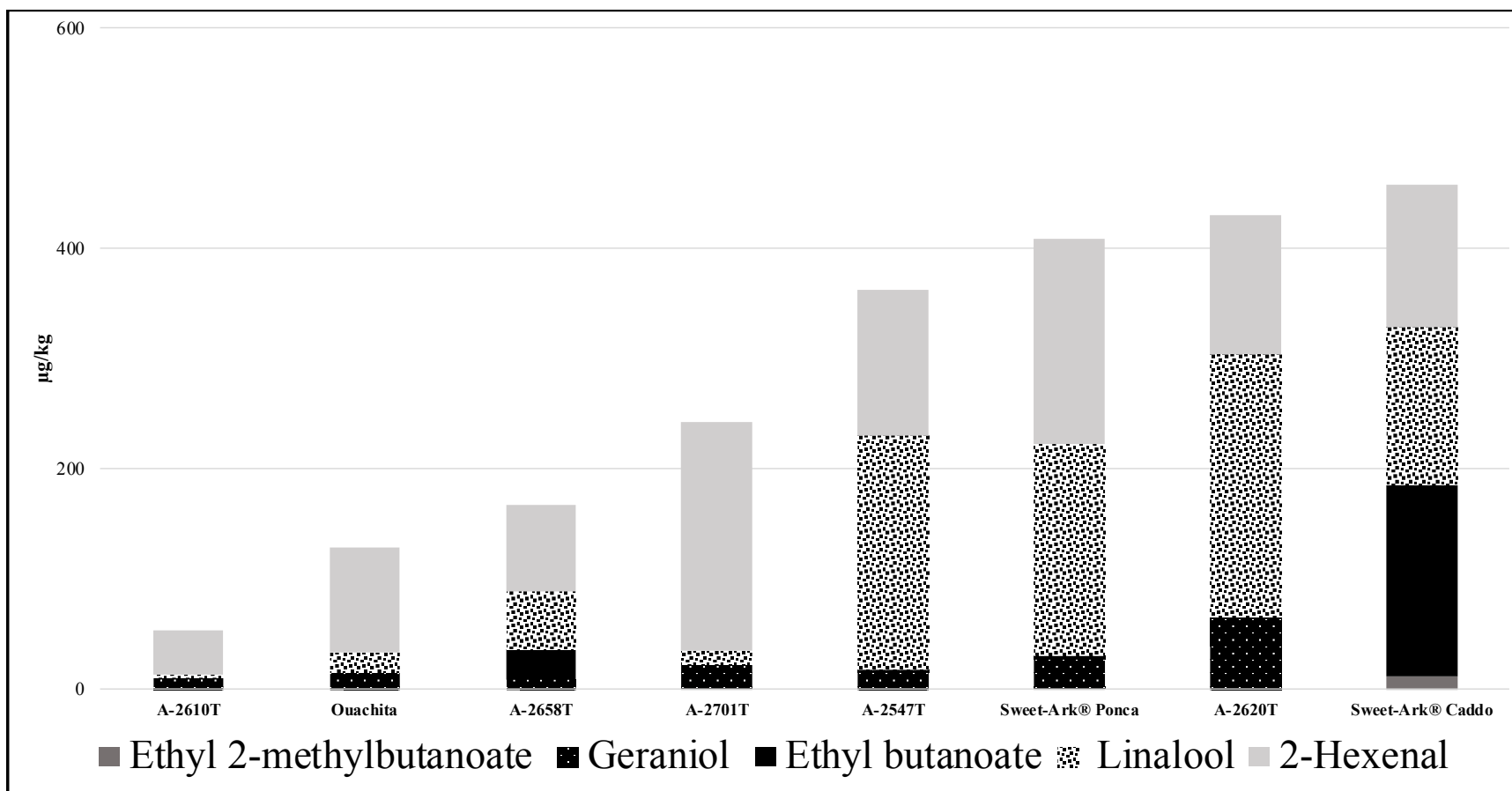
**Fig. 4.** Total concentrations of major volatiles aroma compounds identified (µg/kg) in fresh-market blackberry genotypes, Clarksville, AR (2020).



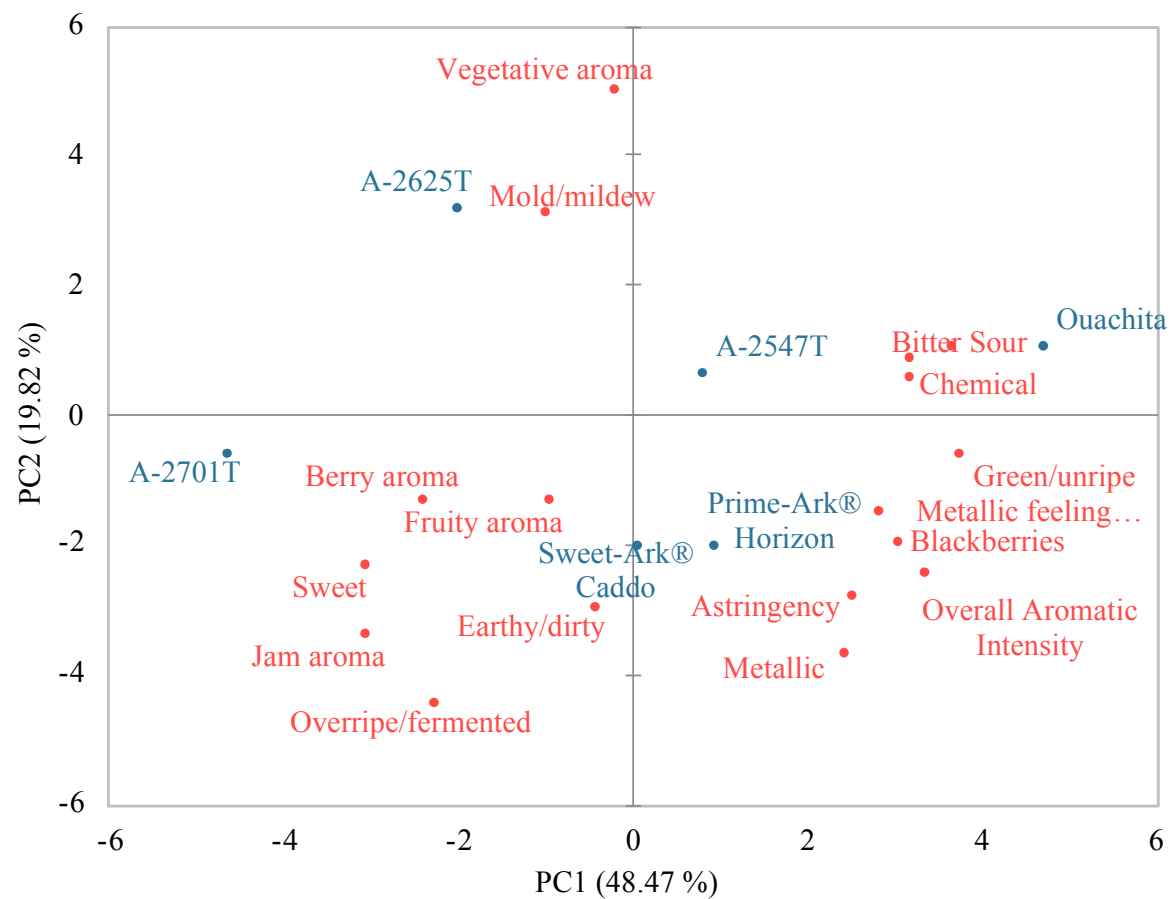
**Fig. 5.** Total volatile aroma compounds of fresh-market blackberry genotypes, Clarksville, AR (2021). Total volatile aroma compounds are the cumulative concentration of each class of volatile aroma compounds for each genotype



**Fig. 6.** Principal components (PC) analysis on volatile aroma compounds in fresh-market blackberries grown at the University of Arkansas System Division of Agriculture Fruit Research Station, Clarksville, AR (2021).  
*Percent of variation in data explained by each component.*  
*Compound class variables represent the sum of the total ion chromatogram (TIC) relative peak areas (%) of positively identified compounds within each compound class (Table 6)*

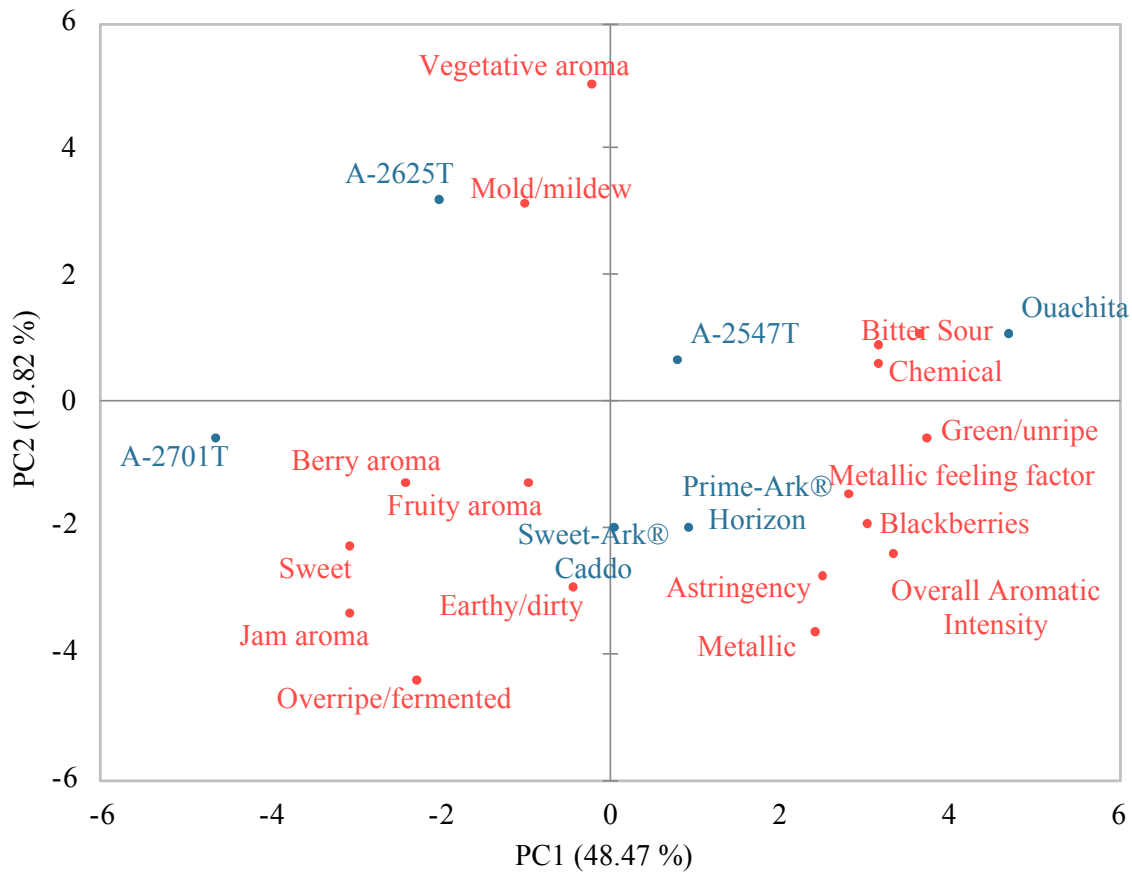


**Fig. 7.** Total concentrations of major volatiles aroma compounds identified ( $\mu\text{g}/\text{kg}$ ) in fresh-market blackberry genotypes, Clarksville, AR (2021).



**Fig 8.** Principal components (PC) analysis on sensory attributes of fresh-market blackberries grown at the University of Arkansas System Division of Agriculture Fruit Research Station, Clarksville, AR (2020). *Percent of variation in data explained by each component.*





**Fig 9.** Principal components (PC) analysis of descriptive sensory attributes in fresh-market blackberries grown at the University of Arkansas System Division of Agriculture Fruit Research Station, Clarksville, AR (2021).

*Percent of variation in data explained by each component*

## Chapter II

### **Determining Best Handling Practices for Fresh-market Blackberries to Increase Postharvest Quality**

#### **Abstract**

Fresh-market blackberries (*Rubus* subgenus *Rubus*) are a delicate fruit that is sold commercially worldwide. Handling the fruit during and after harvest and storage impact marketability and profitability. In 2020 and 2021, physical and composition attributes were evaluated at harvest and marketability attributes were evaluated after postharvest storage for 21 days at 2 °C. Different cultivars ('Natchez', 'Osage', 'Prime-Ark<sup>®</sup> Traveler', and 'Sweet-Ark<sup>®</sup> Caddo') of blackberries were grown at commercial operations in northwest Arkansas and harvested at optimal ripeness using two harvest methods (gentle and rough) and two acclimation treatments (berries without acclimation placed at 2 °C after harvest and berries with acclimation placed at 10 °C for 4 h then transferred to 2 °C). Cultivar, harvest method, and acclimation temperature were examined to determine best handling practices for fresh-market blackberries to increase postharvest quality. For both years berries were 3-9 g, 18-31 mm long, 18-23 mm wide, 6-10 N firm, 8-12% soluble solids, 3.0-3.3 pH, and 1.0-1.4% titratable acidity. At harvest in both years, leakage was less than 9% with no to little decay or red drupelet reversion. Harvest method x acclimation x cultivar had a significant impact on weight loss for both years. In 2020, weight loss was less than 21% and in 2021, less than 11%. Decay was less than 2% and 43% in 2020 and 2021, respectively. Harvest method x cultivar impacted red drupelet reversion in 2020, and acclimation x cultivar impacted leakage and red drupelet reversion in 2021. Red drupelet reversion was less than 15% in 2020, and 13% in 2021. Leakage was less than 5% in 2020, and less than 56%, at 21 days postharvest in 2021. Cultivar had the biggest impact on marketability

attributes which varied by year. Overall, there were no clear trends on marketability degradation of the blackberries, and for both years the damage was considered low for fresh-market blackberries stored at 2 °C for 21 days.

### **Introduction**

Blackberry (*Rubus* subgenus *Rubus*) plants are native to several continents, including Asia, Europe, and North and South America, and are generally referred to as caneberries. Blackberry plant architecture can be erect, semi-erect, or trailing. There are many different cultivars of blackberries that are grown commercially. Most cultivars are floricanes fruiting, producing berries on the second-year canes (floricanes). However, there are cultivars that produce fruit on first-year canes (primocane). Blackberries are grown domestically and internationally for both fresh and processing industries.

Fresh-market blackberries are harvested for direct sale to consumers, while processing blackberries are harvested for other uses, such as freezing, canning, and beverages. Blackberries can be harvested by hand or machine, and the method of harvesting depends on the architecture of the plant, the use of the fruit, and other factors. Fresh-market blackberries are hand-picked to ensure the fruit maintains quality from harvest to consumption, typically 10-21 days. Depending on cultivar, blackberries can be harvested from either floricanes for 2-3 weeks or around 8 weeks for double-cropped (primocane and floricanes fruiting plants) as fruit on the different cane types ripens at different times (Clark and Perkins-Veazie, 2011). However, hand harvesting has downfalls, as berries must be harvested at peak ripeness; while firm and shiny black, but not overripe. Overabundance of fruit, limited labor availability, or weather disruptions can lead to yield loss at harvest or a surplus of underutilized produce. These events are devastating for fresh-

market blackberry growers since primary quality fruit sells for much more than secondary quality fruit.

Fresh-market blackberries in the United States have become more commercially available in the last decade. Consumers that once only had fresh blackberries seasonally, either harvested from a wild plant or pick-your-own operations, now expect to have year-round availability of fresh-market blackberries at retail markets. Blackberries have established a more prominent place in the market due to enhanced shipping capability, prolonged postharvest shelf-life, off-season availability, and double blossom/rosette disease resistance (Clark, 2005; Strik et al., 2007). To meet demand, both public and private blackberry breeding programs have focused on enhancing blackberry plant and fruit attributes. Fresh-market blackberries have unique attributes that impact, harvest-handling, postharvest storage, marketability, and consumer perception.

Blackberries are an aggregate fruit comprised of drupelets surrounding a soft tissue receptacle (torus). Each drupelet has a thin exocarp, a fleshy mesocarp, and a hard-lignified endocarp, or pyrene, that encloses a single seed (Tomlik-Wyremblewska et al., 2010). Thus, the blackberry fruit are very delicate at full ripeness. The size and shape of a fully-ripened blackberry varies among cultivars. On average, the weight of each blackberry will range from 5-15 g with length of 15-30 mm (Carvalho and Betancur, 2015). The berries can have different shapes, such as a round shape, or the berries can be long and oval shaped. In addition, firmness, force, measured in Newtons to compress an individual blackberry, can vary. Firmness is influenced by protopectin in the inter-cellular structures of blackberry drupelets, which act like cement to give blackberries a firm texture, but hydrolysis, large respiration rates, and warmer conditions during ripening decrease protopectin activity (Jennings, 2003). Evaluation of genotypes (cultivars and breeding selections) of blackberries grown in Arkansas showed that the

average firmness was 3-8 N (Threlfall et al., 2016b; Segantini et al., 2018; Salgado and Clark, 2016).

One of the major factors impacting commercial availability of fresh-market blackberries is the postharvest potential or shelf-life. Proper harvest and handling of the berries can minimize damage, which can decrease yield loss and increase consumer satisfaction. Fresh-market blackberries can have problems associated with leakage, decay, and red drupelet reversion, a postharvest disorder that causes individual drupelets on the berry that are black at harvest to turn, or revert, to a reddish color.

The U.S. Department of Agriculture (USDA) has strict guidelines on the grades and standards for the sale of fresh-market blackberries (USDA, 2018). Fresh-market blackberries are typically sold commercially in clear, plastic clamshells, so appearance of the berry is important. These postharvest quality attributes include decay (visible rot or mold on berries), leakage (juice leaking from berry drupelets), shrivel (drying of berries), and red drupelet reversion (drupelets on berries turn from black to red). Quality of fresh-market blackberries must be maintained from harvest, during storage, and consumer purchase. Threlfall et al. (2021) showed that consumers preferred to purchase blackberries in clamshells that did not have red drupelet reversion. The USDA defines grades and standards for produce that are sold commercially to help maintain quality and guidance. The fruit is graded on appearance, texture, composition, and marketability attributes.

Fresh-market blackberry appearance is graded a Number 1 if they are firm, well-colored, well-developed, and if 99% of the packaged blackberries are free from any mold or decay (USDA, 2018). A blackberry is graded Number 2 if more than 90% of the berry is free from damage or 2% of the packaged berries are free from mold and decay (USDA, 2018).

Discoloration affecting individual drupelets that are reddish black or reddish blue and blend in color are not considered a defect; however, discoloration that is noticeably bluish red to bright red and do not blend in color will be scored as damage or serious damage (USDA, 2018). Red drupelet damage is noted when it detracts from the appearance, and serious damage when it seriously detracts from the appearance. In terms of white drupelet disorder (tan or white color) damage is noted when impacting two or more drupelets, and serious damage if more than five drupelets (USDA, 2018). White drupelets occur prior to harvest and may be caused by weather or ultra-violet radiation when there is hot, dry air with little humidity (Stafne et al., 2017). Research continues to identify cultivars that are best for fresh-market blackberries, and public and private blackberry breeding programs continue to develop new cultivars to meet the needs of growers, packers, processors, and consumers (Clark and Finn, 2014).

The method of harvesting blackberries impacts the potential for shipping and storage. Mechanical harvesting of fresh-market blackberries has been attempted but typically causes too much damage to the fruit. Edgley et al. (2020) reported that mechanical harvest injury caused anthocyanin degradation, which is thought to be the mechanism that causes red drupelet reversion. Edgley (2019) investigated how harvest and handling of fresh blackberries impacted fruit quality showing that in 85% of blackberries that were handled roughly (picked from cane and dropped into a container with no regard to how or where the berry lands in relation to other berries already in the container) during harvest had red drupelet reversion (one drupelet per berry). In comparison, only 6% of blackberries that were handled gently (picked from cane and placed into container with delicate regard, ensuring it did not damage berries already in the container) displayed red drupelet reversion. However, Flores-Sosa et al. (2021) found that red drupelet reversion produced by mechanical damage was not linked to anthocyanin degradation,

but rather a loss of anthocyanin accumulation and therefore a disruption in the interaction between anthocyanins and other cellular compounds.

Previous theories suggested time of day had an impact on postharvest qualities of fresh-market blackberries. Edgley et al. (2019) evaluated the temperatures at time of harvest and showed that greater temperatures positively correlated with development and severity of red-drupelet reversion. Most of the color change happened within 24 h of the blackberries entering cool storage, but the number of reverted drupelets can increase for up to two weeks postharvest (Edgley et al., 2019; Yin, 2017). Edgley et al. (2019) found that ‘Ouachita’ berries exposed to impact damage at a warmer initial temperature (25 °C) had increased rates of red drupelet reversion compared to berries that were harvested at a cooler temperature (15 °C). Other researchers also found that the time of day impacts red drupelet reversion, with most reversion occurring within 24 h after entering cool storage, although the number of reverted drupelets can continue to increase during postharvest storage (Armour et al., 2021; Edgley et al., 2019; McCoy et al., 2016; Yin, 2017). However, Felts et al. (2020) showed there was minimal impact of harvest time (7:00 am versus 12:00 pm), but genotype and length of time in storage had the greatest impact.

Another factor that contributes to the unique attributes of fresh-market blackberries is the composition of sugars and acids. Sugars and organic acids are the main soluble constituents of berries that impact the sweetness and sourness (Mikulic-Petkovsek et al., 2012). Sugars are the major soluble solids in blackberries, but other soluble materials include organic acids, amino acids, and soluble pectin. The soluble solids of commercially-acceptable fresh-market blackberries ranges from 8-11% (Threlfall et al., 2016a). Cultivated blackberries, in contrast to wild blackberries, have a greater size, but lower soluble solids, titratable acidity, and pH (Yilmaz

et al., 2009). The pH of a commercially-acceptable fresh-market blackberry ranges from 3.0-3.6, and titratable acidity ranges from 0.7-1.4% (Threlfall et al., 2016a). Segantini et al. (2018) determined important attributes for quality, demonstrating fresh-market blackberries had a good balance of acidity and sugar content, as noted by descriptive sensory panelists. The balance of sugars and acids along with maintaining fruit quality during storage are important attributes for fresh-market blackberries.

Postharvest storage can impact the quality of berries that arrive to the market, remain on the shelf at the market, and are stored in the consumer's home. Roughly one-third of fresh produce is lost at various points in the distribution system (Kader, 2002). Blackberries can be held in postharvest cold storage for a week or more, but storability depends on many factors, including temperature and humidity. Temperature is one of the most important factors that influences the deterioration of harvested commodities. Most perishable horticultural commodities last longer at optimal temperatures and can deteriorate two to three-fold for each 10 °C rise in temperature (Kader, 2002). Blackberries are susceptible to water loss during storage and an effective method for reducing water loss is to increase the relative humidity in the storage environment. However, high humidity in storage can lead to bacterial and fungal growth. A relative humidity of 85 - 90 % proved satisfactory for storage of blackberries (Willis et al., 1989).

Blackberries are one of the most perishable types of fruit because of their thin and fragile skin and large respiration and transpiration rates. Hence, rapid changes in blackberry composition and sensory properties, along with decay can occur during postharvest storage. Temperature management, including rapid cooling after harvest and maintenance of low temperatures, is the most important factor in minimizing blackberry deterioration and



maximizing quality and postharvest storage (Bolda et al., 2012; Kader, 2002). For blackberry storage, temperatures from 0-5 °C and modified atmosphere (5–10% oxygen/15–20% carbon dioxide) are recommended during shipping (Cia et al., 2007; Kader, 2002).

Storage time, storage temperature, and handling of blackberries can damage berries and make berries less appealing to consumers. Mold growth can occur on blackberries during postharvest storage, and the most predominant species of mold growth is *Botrytis cinerea* Pers. and *B. caroliniana*, also known as gray mold (Li et al., 2012). *B. cinerea* has an affinity for a high-pectin content host and destroys plant cell structure then colonizes on dead tissue on the fruit. Although optimum growth of *B. cinerea* is 20 °C, its ability to grow at colder temperatures (as low as 0 °C) leads to slow decay during storage of fresh-market fruit (Bautista-Baños, 2014). Other research has shown storage temperature of blackberries was directly related to degree of deterioration (Palharini et al., 2015; Perkins-Veazie et al., 1999; Perkins-Veazie and Clark, 2005; Segantini et al., 2018). Kim et al. (2015) showed that blackberries stored at 1 °C had better postharvest quality compared to 20 °C, and room temperature storage reduced quality in all cultivars. Types of storage container, packing procedures, storage temperature, and humidity affect marketability of the fruit (Joo et al., 2011). Kim et al. (2015) reported that fruit stored at 1 °C retained consistent marketability, however, when removed from cold storage and placed in room temperature, fruit deterioration rapidly increased. Kader (2002) also showed stage of picking (dull black versus shiny black) affected leakage, decay, and red-drupelet reversion.

Firmer berries have longer shelf life (Segantini et al., 2017; Kim et al., 2015) and have enhanced potential for shipping and postharvest storage. The University of Arkansas System (UA System) Division of Agriculture has one of the most prominent public fresh-market breeding programs and has evaluated postharvest storage potential of “crispy” genotypes with a

firmer texture, which might be better suited for fresh-market sales in contrast to softer berries that can be used for processing or local market sales. According to Salgado and Clark (2016), the crispy genotypes maintain cell-wall and cell-to-cell adhesion and have better storage potential compared to less-crispy genotypes. Softness was positively correlated to decay/leakage of blackberries (Kim et al., 2015).

Cultivar could also affect postharvest disorders with some cultivars are better suited for early morning harvesting. Different cultivars can produce berries that have large yield and longer storage potential (Clark and Moore, 1999), therefore those berries will be more profitable for the grower, and the consumer will have a berry with a longer post-purchase life. Lawrence and Melgar (2018) reported leakiness was greater in ‘Chester’ and ‘Triple Crown’ when harvested later in the day compared to other cultivars in the study.

Fresh-market blackberries are a valuable fruit with unique attributes. The way blackberries are grown, harvested, and stored impacts the quality of the fruit which is an important driver for consumer purchasing. Identifying unique cultivar attributes, harvesting strategies, and postharvest storage practices of the fresh-market blackberries will help advance future breeding efforts for the expansion of the blackberry industry. Determining the best handling practices for fresh-market blackberries is important to the future of the fresh-market blackberry industry. Therefore, the purpose of this project was to determine the impact of harvest methods and acclimation temperatures of fresh-market blackberry cultivars on the physical and composition attributes at harvest and marketability attributes after postharvest storage.

## **Materials and Methods**

### **Blackberry cultivars and harvest**

Four blackberry cultivars, ‘Natchez’, ‘Osage’, ‘Prime-Ark<sup>®</sup> Traveler’, and ‘Sweet-Ark<sup>®</sup> Caddo’ were harvested from the floricanes in late June to early July of 2020 and 2021 from a commercial grower in Fayetteville, AR (Cold Hardiness Zone 6b). Berries were harvested at the shiny-black stage of ripeness from 7:00-10:00 am. About 2 kg of each cultivar was harvested and placed into 170 g vented clamshells in triplicate. In each year for each cultivar, berries were harvested using two harvest methods (gentle and rough). After harvest, the blackberries were transported to UA System Food Science Department, Fayetteville and placed in cold storage with two acclimation treatments (no acclimation and acclimation).

**Harvest method.** Gentle harvesting was performed by picking a berry and gently placing the berry in the clamshell making sure the berry did not come into contact with other blackberries and that the clamshell lid was not touching the blackberries when closed. Rough harvest was performed by picking the berry and dropping the berry into the clamshell without regard to the berries touching each other.

**Acclimation.** After harvest, the blackberries without acclimation were placed into cold storage at 2 °C until analysis. The blackberries with acclimation were placed in at 10 °C for 4 h then transferred to 2 °C storage until analysis.

### **Attribute analysis**

In 2020 and 2021, physical and composition attributes were evaluated at harvest and marketability attributes were evaluated after postharvest storage for 21 d at 2 °C.

**Physical attribute analysis.** Five berries per cultivar, harvest method, acclimation, and replication were used for berry attributes. Each berry was weighed (g) using a precision digital scale (PA224 Analytic Balance, Ohaus Corporation, Parsippany, NJ), then the length (mm) and width (mm) were measured using digital calipers. Firmness of each berry was measured by a

Stable Micro Systems TA.TX. XT plus Texture Analyzer (Texture Technologies Corporation, Hamilton, MA). Firmness was measured using a 7.6-cm diameter cylindrical probe to compress with a trigger force of 0.02 N, an individual berry placed horizontally on a flat surface. The force needed to compress the berry was measured in Newtons (N). The berries were then frozen (-10 °C) for compositional analysis.

**Composition attribute analysis.** Composition of the juice from five berries per cultivar, harvest method, acclimation, and replication were measured for soluble solids, pH, and titratable acidity. The five berries were thawed at room temperature (21 °C) and squeezed through cheesecloth to extract juice for analysis. Soluble solids of the juice were measured and expressed as percent (%) using an Abbe Mark II refractometer (Bausch and Lomb, Scientific Instrument, Keene, NH). The pH and titratable acidity were measured using a Metrohm 862 Compact Titrosampler (Metrohm AG, Herisau, Switzerland) fitted with a pH meter. The titratable acidity (expressed as % w/v (g/100 mL) citric acid) of juice was measured using a 3-g sample added to 50 mL degassed, deionized water and titrated with 0.1 N sodium hydroxide to an endpoint of pH 8.2.

**Marketability attributes analysis.** Marketability attributes (decay, leakiness, and red drupelet reversion) were evaluated after postharvest storage for 21 d at 2 °C. The decay of each blackberry was evaluated by determining if the blackberry had visible mold or rot on the blackberry surface. Decay of the blackberries in a clamshell was calculated as (number of decayed berries / numbers of total berries) × 100 and expressed as a percent. The leakiness was evaluated by gently rolling each blackberry on a white paper towel to determine if the berry drupelets leaked. The leakage of the blackberries in a clamshell was calculated as (number of leaky berries / numbers of total berries) × 100 and expressed as a percent. The red drupelet reversion (black drupelets changed to red) of the blackberries was evaluated by determining if

each blackberry in the clamshell had more than one red drupelet. Red drupelet reversion was calculated as (number of berries that developed red drupelets / number of total berries) x 100 and expressed as a percent.

### **Statistical design and analysis**

In both years, two harvest methods (gentle and rough), two acclimation temperatures (2 °C and 10 °C), and four cultivars ('Natchez', 'Osage', 'Prime-Ark<sup>®</sup> Traveler', and 'Sweet-Ark<sup>®</sup> Caddo') were evaluated in triplicate for physical and composition attributes at harvest and marketability attributes after postharvest storage for 21 d at 2 °C. Data was analyzed by year. Statistical analysis was conducted using JMP<sup>®</sup> Pro Statistical Software (version 16.0; SAS Institute Inc., Cary, NC). A univariate analysis of variance (ANOVA) was used to determine the significance of the main factors and their interactions. Tukey's Honest Significant Difference test or Student's T test were used to detect differences among means ( $p < 0.05$ ). Figures were created with each standard error bar constructed using 1 standard error from the mean.

## **Results and Discussion**

The 2020 and 2021 blackberry harvest seasons were relatively typical in Fayetteville, AR with an average high temperature of 24 °C and a low of 19 °C during June for both years. The 2021 harvest season had additional challenges resulting from a record freeze (-25 °C) in February followed by a frost in April impacting plant survival and fruit production of some cultivars. Harvest temperatures averaged 21 °C in 2020 and 2021.

### **Physical, composition, and marketability attributes at harvest**

The cultivars were evaluated for physical attributes (berry weight, length, width, and firmness) and composition attributes (soluble solids, pH, and titratable acidity) at harvest. The leakage, decay, and red drupelet reversion were also evaluated at harvest. In either year, cultivar

impacted the physical and composition attributes more than harvest method or acclimation. For both years, berries were 3-9 g, 18-31 mm long, 18-23 mm wide, 6-10 N firm, 8-12% soluble solids, 3.0-3.3 pH, and 1.0-1.4% titratable acidity. The physical and compositional attributes were within ranges established by previous research by Felts et al. (2020) and Segantini et al. (2017) on Arkansas fresh-market blackberries. Segantini et al. (2017) harvested 11 genotypes in 2015, with firmness 4.9-9.0 N, soluble solids 4.7-19.5%, pH 3.0-3.4, and titratable acidity 0.5-1.5%.

In both years ‘Natchez’ was the biggest blackberry in terms of weight, length, and width. ‘Natchez’ also had the highest titratable acidity in both years. Fruit firmness was similar in both years 7.07-9.02 N for 2020 and 6.23- 9.40 N in 2021. ‘Sweet-Ark<sup>®</sup> Caddo’ (9.02 N) had the firmest berry in 2020, and ‘Natchez’ had the firmest in 2021 (9.40 N). In both years ‘Prime-Ark<sup>®</sup> Traveler’ had the highest soluble solids and lowest titratable acidity. For pH ‘Osage’ (3.32) had the highest and ‘Natchez’ (3.03) had the lowest.

At harvest, ‘Natchez’ had the highest leakage in 2020 (9.17%), and ‘Prime-Ark<sup>®</sup> Traveler’ had the highest leakage in 2021 (3.29%). There was no decay of any cultivars at harvest in either year. Red drupelet reversion was negligible in both years with ‘Natchez’ and ‘Prime-Ark<sup>®</sup> Traveler’ just having one red drupelet each at harvest in 2020.

### **Marketability attributes during postharvest storage**

The main and interaction effects of harvest method, acclimation, temperature, and cultivar stored for 21 d at 2 °C on marketability attributes of fresh-market blackberries grown were evaluated (Tables 2 and 3). Cultivar seemed to have the biggest impact on the marketability attributes which varied by cultivar and year. In general, the marketability attributes were higher in 2020 as compared to 2021, especially for leakage.

**2020.** Harvest method x acclimation x cultivar had a significant effect on weight loss at 21 d postharvest in 2020 (Figure 1). Weight loss for these blackberries in 2020 was less than 21%. Weight loss for ‘Prime-Ark<sup>®</sup> Traveler’ harvested gently and not acclimated was significantly higher (18.32%) than rough harvested, not acclimated (15.03%) and gently harvested and acclimated (12.70%). Non-acclimated ‘Osage’ harvested gently (17.62%) had lower weight loss than rough harvested fruit (20.39%) but acclimated ‘Osage’ harvested gently (20.72%) had higher weight loss than fruit harvested roughly (18.60%). ‘Sweet-Ark<sup>®</sup> Caddo’ roughly harvested and acclimated (12.88%) had higher weight loss than gently harvested (11.17%) and rough harvest and not acclimated (10.69%). Felts et al. (2020) evaluated storage at 14 d and 10 °C, weight loss was 13%, and in contrast to our study the weight loss highest observed was 19% for ‘Osage’ in 2020.

The main effects of interactions were not significant for leakage or decay. After 21 d of storage, the blackberries had less than 5% leakage and less than 2% decay. Harvest method x cultivar significantly impacted red drupelet reversion at 21 d postharvest (Fig. 2) with ‘Sweet-Ark<sup>®</sup> Caddo’ roughly harvested (15%) significantly higher than all harvest methods and cultivars. Harvest method did not impact the other cultivars in terms of red drupelet reversion. Felts et al. (2020) determined blackberries stored at lower temperatures (2 °C) retained marketability attributes longer than blackberries stored at higher temperatures (10 °C). Blackberries in our study were acclimated for 4 hours at 10 °C, but stored for most of the analysis at 2 °C. In our study, the cultivar effect on red drupelet reversion after 21 days of storage was significant in 2020, but not in 2021. Armour et al (2021) determined blackberries harvested earlier in the day had less red drupelet reversion than blackberries harvested later in the day. In addition, smaller clamshells (170 g) were used for our harvest compared to 240 g

clamshells used in Amour et al. (2021) and Segantini et al. (2017), which could have impacted marketability attributes. Edgley et al. (2019) harvested berries into shallow buckets and then transferred the berries to 125 g clamshells lined with a soaker pad. The smaller clamshells could limit berry contact.

**2021.** Harvest method x acclimation x cultivar had a significant effect on weight loss and decay at 21 d postharvest in 2021 (Figure 3). Weight loss was less than 11%, and decay was less than 43%. ‘Prime-Ark<sup>®</sup> Traveler’ harvested roughly and acclimated (10.42%) had significantly higher weight loss than ‘Prime-Ark<sup>®</sup> Traveler’ gently harvested and acclimated (6.25%) and all other cultivars and treatments. ‘Prime-Ark<sup>®</sup> Traveler’ harvested gently and not acclimated (34.04%) was significantly higher in decay than gentle harvested and acclimated (11.15%). However, ‘Prime-Ark<sup>®</sup> Traveler’ harvested roughly and not acclimated (5.56%) was significantly lower in decay than rough harvested and acclimated (36.56%). ‘Sweet-Ark<sup>®</sup> Caddo’ harvested roughly, not acclimated (42.68%) had significantly higher decay than gently harvested, not acclimated (7.97%) and rough harvested, acclimated (10.66%) and gently harvested and acclimated (6.23%). Decay was 70% in Felts et al. (2020). Lawence and Melgar (2018) found the response to time of harvest, delay until cold storage, and storage length on postharvest quality of blackberries was cultivar specific.

Acclimation x cultivar significantly impacted leakage and red drupelet reversion (Fig. 4). Red drupelet reversion was less than 13% and leakage was less than 56%. ‘Natchez’ acclimated had significantly higher red drupelet reversion (12.17%) than all other cultivars. Harvesting all the blackberries early in the morning (before 10 am) could be the reason overall red drupelet reversion was low in both 2020 and 2021. McCoy et al. (2016) reported that fruit harvested at 7 am had lower red drupelet reversion compared to blackberries harvested at 10 am, 1 pm, and 4



pm because the skin temperature of the blackberry increased as the ambient temperature increased. ‘Natchez’ not acclimated (36.34%) had more leakage than the acclimated fruit (15.07%), however the inverse occurred for the other cultivars but only significantly for ‘Sweet-Ark<sup>®</sup> Caddo’. ‘Prime-Ark<sup>®</sup> Traveler’ acclimated had the highest leakage (55.54%). Lawrence and Melgar (2018) found 46% leakage after 14 d storage at 4 °C in ‘Prime-Ark<sup>®</sup> Traveler’ and as high as 86% leakage after 14 d in other cultivars. Kim et al. (2015) showed that blackberries had up to 77% leakage after 15 d at 1 °C for 13 d and 20 °C for 2d (retail temperature). Felts reported 40% leakage at 14 days, our study had 36% for ‘Natchez’ in 2021. Storage temperature preserved marketability qualities. Our study held the blackberries a week longer in storage with less, or similar postharvest degradation.

Another factor to consider is the harvest methods employed. Gentle harvest was performed by picking a berry and gently placing the berry in the clamshell making sure the berry had limited contact with other blackberries and that the clamshell lid was not touching the blackberries when closed. Rough harvest was performed by picking the berry and dropping the berry into the clamshell without regard to the berries touching each other. In both methods, the berries were touched and removed from the plant by either grabbing the berry behind the receptacle. Edgley et al. (2019) defined gentle harvest by carefully cutting the pedicle with pruning shears, approximately 1 cm above the receptacle and placing each berry into individual cotton wool lined cells of 30 mm square seedling trays. Perhaps, there was not enough of a difference between the two harvest methods in our study, not gentle enough and not rough enough. Rough harvest in Edgley et al. (2019) study was industry standard; hand harvested into shallow buckets and then transferred into 125 g clamshells lined with an absorbent pad. In our study, rough harvest was performed by picking the berry and dropping the berry into the

clamshell, not from a bucket to a clamshell as in Edgley et al. (2019). The difference of not transferring the berries reduces the amount of times berries are handled, and less handling could impact the amount of damage. Harvest technique was the most significant factor in Edgley et al. (2019).

Edgley et al. (2019) found that the berry surface temperatures were the same at 6 am and 8 am and as air temperature increased the skin temperature increased increasing red drupelet reversion. If berry surface temperature was a major cause for red drupelet reversion, our harvest from 7:00-10:00 am did not give the berries time to heat to incite red drupelet reversion damage. Lawrence and Melgar (2018) found that postharvest degradation increased at harvest times past 10 am.

### **Conclusion**

Harvest method, acclimation temperature, and cultivar were examined to determine best handling practices for fresh-market blackberries to increase postharvest quality. For both years berries were 3-9 g, 18-31 mm long, 18-23 mm wide, 6-10 N firm, 8-12% soluble solids, 3.0-3.3 pH, and 1.0-1.4% titratable acidity. At harvest in both years, leakage was less than 9% with no to little decay or red drupelet reversion. Harvest method x acclimation x cultivar had a significant impact on weight loss for both years. In 2020 weight loss was less than 21% and in 2021 was less than 11%. Decay in 2020 was less than 2% and less than 43% in 2021. Harvest method x cultivar impacted red drupelet reversion in 2020, and acclimation x cultivar impacted leakage and red drupelet reversion in 2021. Red drupelet reversion was less than 15% in 2020, and 13% in 2021. Leakage was less than 5% in 2020, and less than 56% in 2021. Cultivar had the biggest impact on marketability attributes which varied by year. Overall, there were no clear trends on

marketability degradation of the blackberries, and for both years the damage was considered low for fresh-market blackberries stored at 2 °C for 21 days.

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**Table 1.** Mean and standard error<sup>z</sup> of physical, composition, and marketability attributes at harvest for fresh-market blackberries grown in Arkansas (2020 and 2021).

Year and cultivar	Berry weight (g)	Berry length (mm)	Berry width (mm)	Firmness (N)	Soluble solids (%)	pH	Titrateable acidity (%)	Leakage (%)	Decay (%)	Red drupelet reversion
<b>2020</b>										
Natchez	8.50±0.75	31.18±0.75	22.75±0.32	7.23±1.03	12.06±0.18	3.09±0.09	1.43±0.03	9.17±2.67	0±0	0.42±0.42
Osage	3.88±0.12	20.18±0.29	19.90±0.22	7.07±0.29	12.16±0.12	3.32±0.02	1.18±0.02	8.75±2.69	0±0	0±0
Prime-Ark® Traveler	4.23±0.25	20.92±0.78	20.53±0.30	7.12±0.41	12.24±0.10	3.29±0.03	1.01±0.02	4.17±1.49	0±0	0.42±0.42
Sweet-Ark® Caddo	7.73±0.28	30.09±0.53	22.64±0.19	9.02±0.23	11.58±0.23	3.23±0.03	1.18±0.04	7.92±2.98	0±0	0±0
<b>2021</b>										
Natchez	8.97±0.33	31.12±0.67	21.94±0.28	9.40±0.43	9.86±0.29	3.03±0.08	1.33±0.11	2.43±0.95	0±0	0±0
Osage	5.14±0.22	21.28±0.39	19.85±0.33	6.23±0.19	9.88±0.26	3.28±0.04	1.12±0.06	2.47±0.70	0±0	0±0
Prime-Ark® Traveler	3.91±0.15	18.84±0.50	18.68±0.22	7.77±0.49	11.37±0.49	3.21±0.06	0.93±0.06	3.29±1.13	0±0	0±0
Sweet-Ark® Caddo	6.51±0.30	26.06±0.49	20.33±0.29	6.54±0.19	8.39±0.18	3.10±0.04	1.13±0.09	2.06±1.02	0±0	0±0

<sup>z</sup> Values represent means ± standard error; values highlighted have the largest mean and values underlined have the lowest mean.



**Table 2.** Main and interaction effects of harvest method (gentle and rough), acclimation temperature (2 °C and 10 °C), and cultivar stored for 21 d at 2 °C on marketability attributes for fresh-market blackberries grown in Arkansas (2020).

<b>Effects<sup>z</sup></b>	<b>Weight loss (%)</b>	<b>Leakage (%)</b>	<b>Decay (%)</b>	<b>Red drupelet reversion (%)</b>
<b>Harvest method (HM)<sup>y</sup></b>				
Gentle	15.01 a	1.25 a	0.42 a	3.33 a
Rough	14.31 a	3.33 a	0.42 a	6.25 a
<i>P-value</i>	<i>0.0898</i>	<i>0.3475</i>	<i>0.9999</i>	<i>0.1585</i>
<b>Acclimation (A)<sup>x</sup></b>				
2 °C	14.66 a	2.29 a	0.42 a	4.79 a
10 °C	13.92 b	4.38 a	0.21 a	3.33 a
<i>P-value</i>	<i>0.0124</i>	<i>0.1870</i>	<i>0.5677</i>	<i>0.3149</i>
<b>Cultivar (C)</b>				
Natchez	11.84 c	0.83 a	0.00 a	5.00 b
Osage	19.01 a	1.67 a	1.67 a	0.00 b
Prime-Ark <sup>®</sup> Traveler	16.67 b	5.00 a	0.00 a	1.67 b
Sweet-Ark <sup>®</sup> Caddo	11.12 c	1.67 a	0.00 a	12.50 a
<i>P-value</i>	<i>&lt;0.0001</i>	<i>0.5502</i>	<i>0.0644</i>	<i>0.0006</i>
<i>HM x A</i>	<i>0.0304</i>	<i>0.5934</i>	<i>0.5677</i>	<i>0.6646</i>
<i>HM x C</i>	<i>&lt;0.0001</i>	<i>0.9395</i>	<i>0.9999</i>	<i>0.0009</i>
<i>A x C</i>	<i>&lt;0.0001</i>	<i>0.8143</i>	<i>0.8013</i>	<i>0.3637</i>
<i>HM x A x C</i>	<i>&lt;0.0001</i>	<i>0.4417</i>	<i>0.8013</i>	<i>0.0592</i>

<sup>z</sup>Cultivars were evaluated in triplicate. Means with different letter(s) for each attribute within effects are significantly different ( $p < 0.05$ ) using Students t-test.

<sup>y</sup>Gentle hand harvest versus rough hand harvest

<sup>x</sup>Berries without acclimation placed at 2 °C after harvest; berries with acclimation placed at 10 °C for 4 h then transferred to 2 °C.

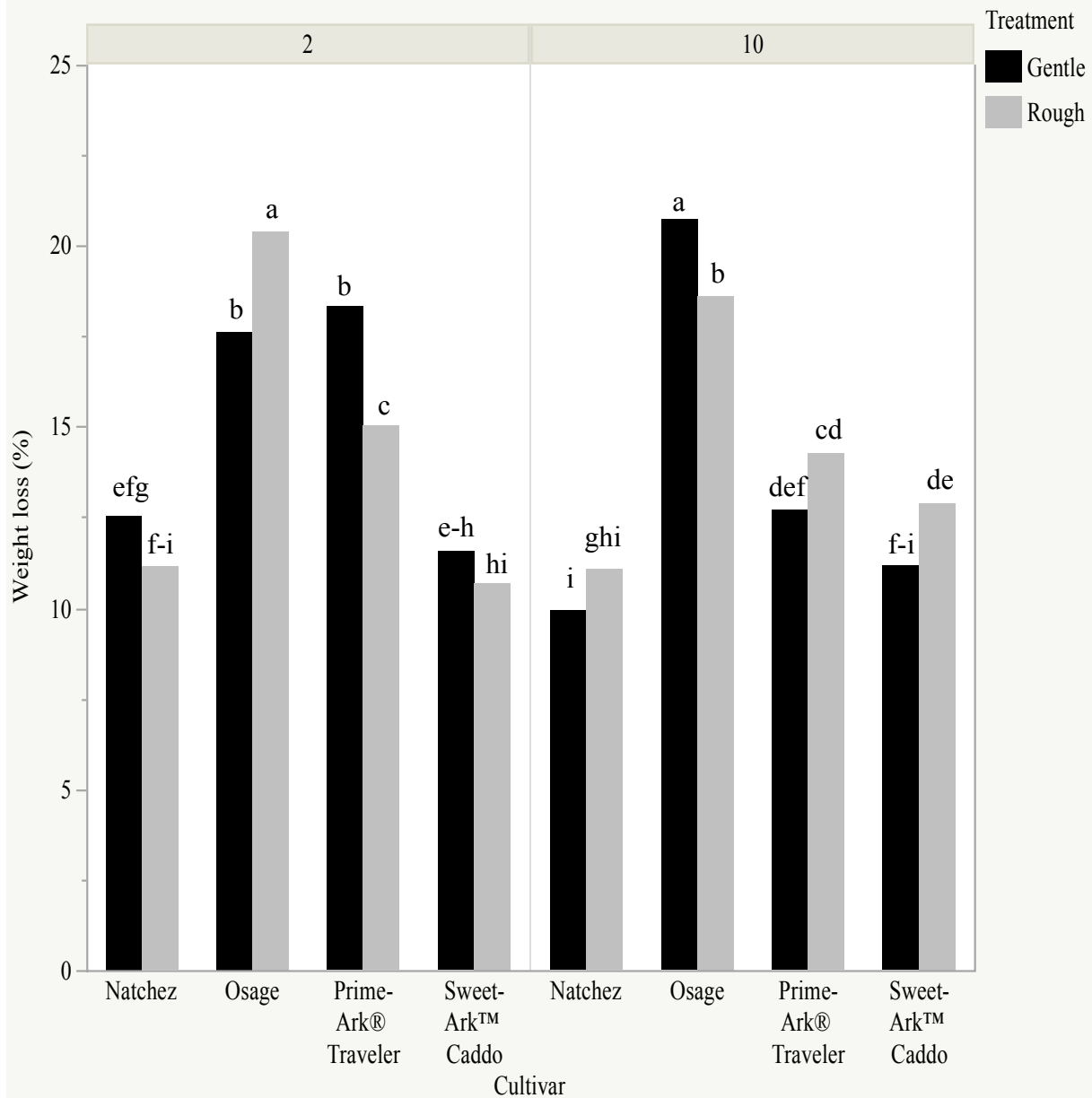
**Table 3.** Main and interaction effects of harvest method (gentle and rough), acclimation temperature (2 °C and 10 °C), and cultivar stored for 21 d at 2 °C on marketability attributes for fresh-market blackberries grown in Arkansas (2021).

<b>Effects<sup>z</sup></b>	<b>Weight loss (%)</b>	<b>Leakage (%)</b>	<b>Decay (%)</b>	<b>Red drupelet reversion (%)</b>
<b>Harvest method (HM)<sup>y</sup></b>				
Gentle	5.98 a	23.58 a	16.67 a	1.40 a
Rough	6.34 a	20.39 a	17.93 a	0.55 a
<i>P-value</i>	<i>0.2455</i>	<i>0.4859</i>	<i>0.9240</i>	<i>0.6490</i>
<b>Acclimation (A)<sup>x</sup></b>				
2°C	6.16 a	21.99 b	16.93 a	0.97 b
10°C	6.38 a	29.41 a	16.18 a	4.13 a
<i>P-value</i>	<i>0.3190</i>	<i>0.0269</i>	<i>0.8478</i>	<i>0.0224</i>
<b>Cultivar (C)</b>				
Natchez	5.31 c	36.34 a	4.24 b	1.85 a
Osage	6.34 ab	4.80 c	18.36 ab	0.00 a
Prime-Ark <sup>®</sup> Traveler	6.98 a	28.15 ab	19.80 ab	0.95 a
Sweet-Ark <sup>®</sup> Caddo	6.01 bc	18.65 b	25.33 a	1.09 a
<i>P-value</i>	<i>0.0051</i>	<i>0.0002</i>	<i>0.0625</i>	<i>0.9181</i>
<i>HM x A</i>	<i>0.1864</i>	<i>0.8554</i>	<i>0.4367</i>	<i>0.1234</i>
<i>HM x C</i>	<i>0.8219</i>	<i>0.1552</i>	<i>0.0029</i>	<i>0.7113</i>
<i>A x C</i>	<i>0.0280</i>	<i>&lt;0.0001</i>	<i>0.0925</i>	<i>0.0315</i>
<i>HM x A x C</i>	<i>0.0011</i>	<i>0.3147</i>	<i>0.0052</i>	<i>0.4588</i>

<sup>z</sup>Cultivars were evaluated in triplicate. Means with different letter(s) for each attribute within effects are significantly different ( $p < 0.05$ ) using Student's t-test.

<sup>y</sup>Gentle hand harvest versus rough hand harvest

<sup>x</sup>Berries without acclimation placed at 2 °C after harvest; berries with acclimation placed at 10 °C for 4 h then transferred to 2 °C.

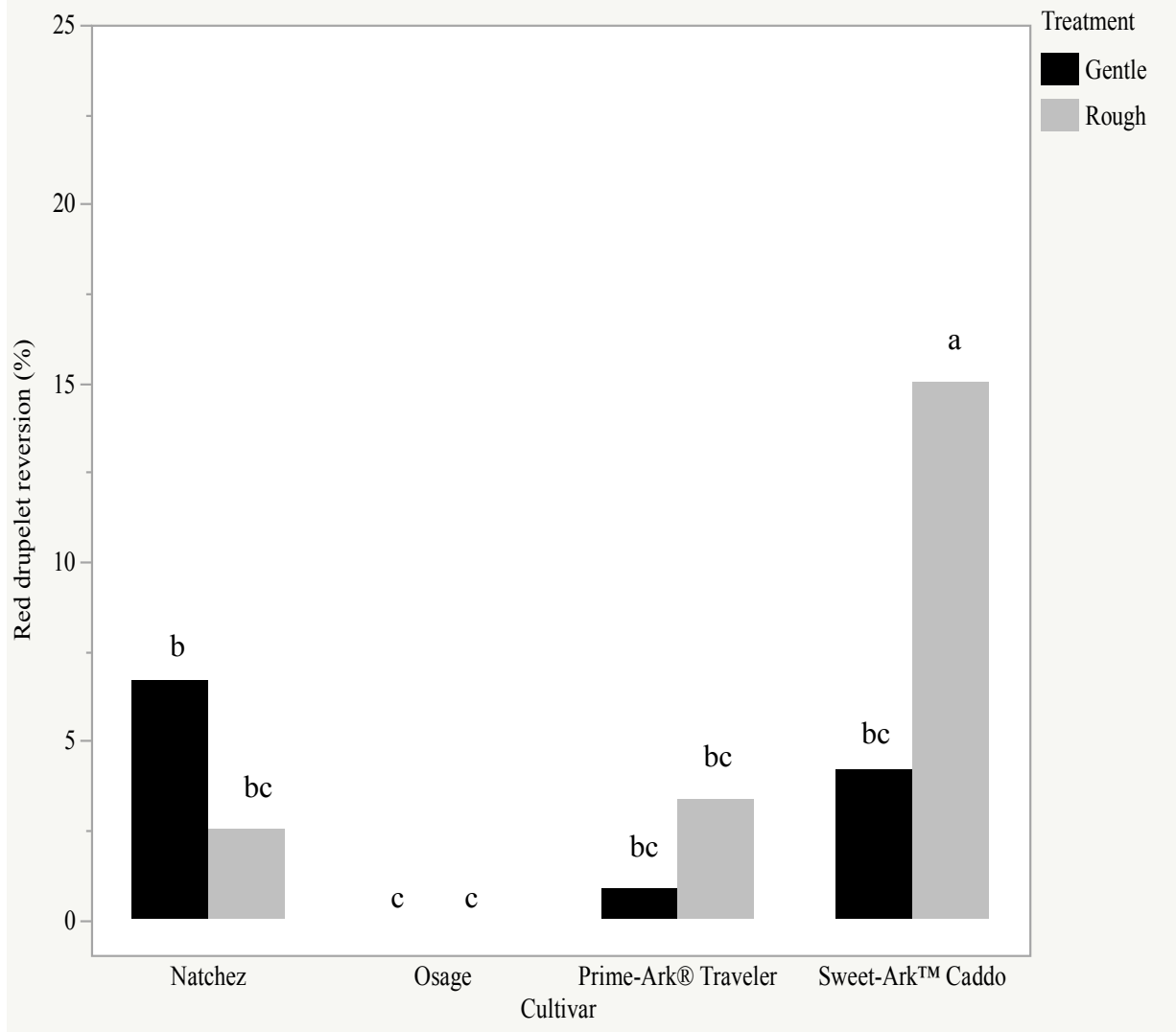


**Fig. 1.** Effect of harvest method (gentle and rough), acclimation temperature (2 °C and 10 °C), and cultivar stored for 21 d at 2 °C on weight loss of fresh-market blackberries grown in Arkansas (2020).

*Cultivars were evaluated in triplicate. Means with different letter(s) for each attribute within effects are significantly different ( $p < 0.05$ ) using Student's *t*-test.*

*Gentle hand harvest versus rough hand harvest*

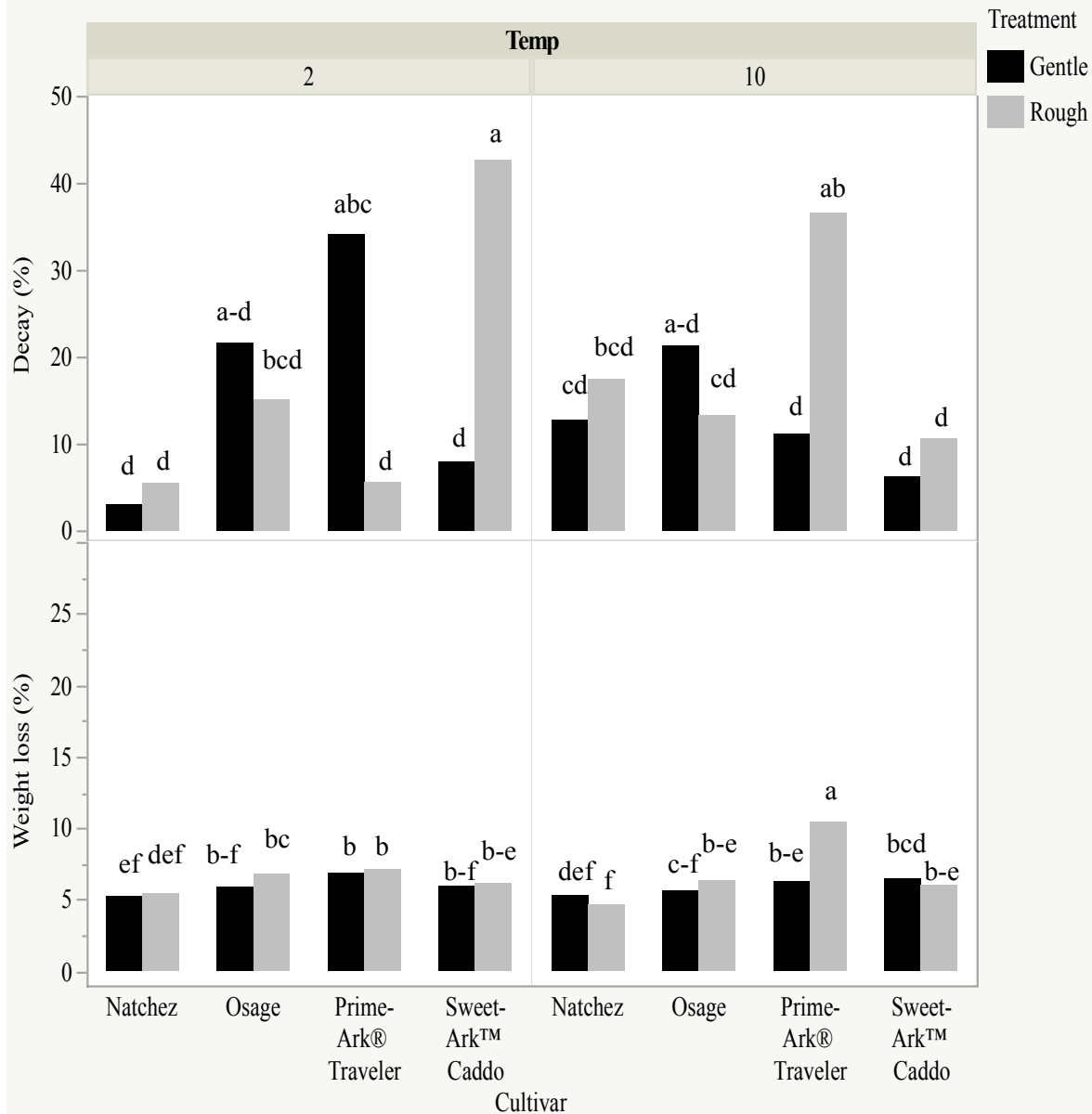
*Berries without acclimation placed at 2 °C, after harvest; berries with acclimation placed at 10 °C for 4 h then transferred to 2 °C.*



**Fig. 2.** Effect of harvest method (gentle and rough) and cultivar stored for 21 d at 2 °C, non-acclimated and acclimated, on red drupelet reversion of fresh-market blackberries grown in Arkansas (2020).

*Cultivars were evaluated in triplicate. Means with different letter(s) for each attribute within effects are significantly different ( $p < 0.05$ ) using Student's *t*-test.*

*Gentle hand harvest versus rough hand harvest*

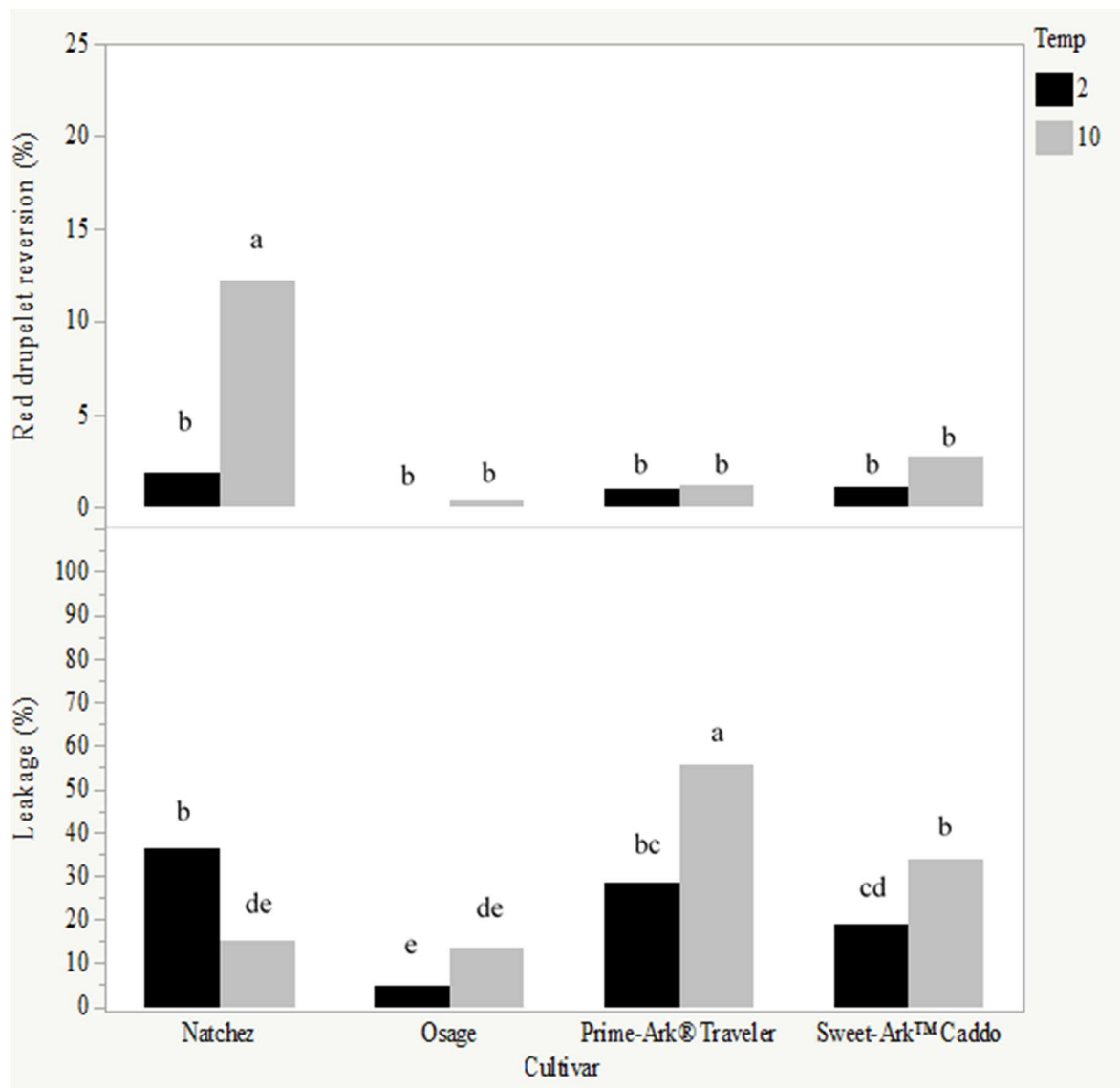


**Fig. 3.** Effect of harvest method (gentle and rough), acclimation temperature (2 °C and 10 °C), and cultivar stored for 21 d at 2 °C on weight loss of fresh-market blackberries grown in Arkansas (2021).

*Cultivars were evaluated in triplicate. Means with different letter(s) for each attribute within effects are significantly different ( $p < 0.05$ ) using Student's *t*-test.*

*Gentle hand harvest versus rough hand harvest*

*Berries without acclimation placed at 2 °C, after harvest; berries with acclimation placed at 10 °C for 4 h then transferred to 2 °C.*



**Fig. 4.** Effect of acclimation temperature (2 °C and 10 °C) on leakage and red drupelet reversion and cultivar stored for 21 d at 2 °C of fresh-market blackberries grown in Arkansas (2021).

*Cultivars were evaluated in triplicate. Means with different letter(s) for each attribute within effects are significantly different ( $p < 0.05$ ) using Student's *t*-test.*

*Gentle hand harvest versus rough hand harvest*

*Berries without acclimation placed at 2 °C, after harvest; berries with acclimation placed at 10 °C for 4 h then transferred to 2 °C.*

## Chapter III

### **Evaluate Potential for a Soft Robotic Gripper for Harvesting Fresh-market Blackberries**

#### **Abstract**

Fresh-market blackberries (*Rubus* L. subgenus *Rubus* Watson) are grown worldwide and are typically hand harvested because the fruit is delicate at full ripeness. Advances in automated and robotic harvesting can play a role in the future of fresh-market fruit industries. At the University of Arkansas, a custom-made force sensing apparatus (sensors) attached to the thumb and fingers of a person was developed to determine the amount of force (N) used to harvest blackberries and the thumb and fingers essential for harvesting in 2020. Then, this data was used to create a soft robotic gripper prototype (gripper) in 2021. Blackberries were also harvested gently by hand to compare to the fruit harvested with the sensors and gripper. In both years, physical and compositional attributes were evaluated at harvest, and marketability attributes were evaluated after postharvest storage for 21 days at 2 °C of different Arkansas-grown cultivars (Natchez, Osage, Prime-Ark<sup>®</sup> Traveler, and Sweet-Ark<sup>®</sup> Caddo). In each year and for each cultivar, 240 berries were harvested then in placed into 170-g clamshells (20 berries/clamshell) in triplicate. The force used by the thumb and middle finger (0.77 N and 0.37 N, respectively) were greatest for harvesting blackberries, whereas the index and ring fingers used lower force (0.16 N and 0.06 N, respectively) primarily to stabilize the berry. A prototype of a 3-prong (fingered) soft robotic gripper was designed using results from the force sensing apparatus. In addition, the forces applied to grab, stabilize, and harvest the blackberries with the sensors, or the gripper did not cause excessive marketability damage (20% weight loss, 40% leakage, 32% decay, and 12% red drupelet reversion) to the blackberries after 21 days postharvest storage. This

project determined harvest and postharvest parameters that were used to further develop a prototype of a soft-robotic gripper for the harvest of fresh-market blackberries.

### **Introduction**

Blackberry (*Rubus* subgenus *Rubus*) plants are native to several continents, including Asia, Europe, and North and South America, and are generally referred to as caneberries. Blackberry plant architecture can be erect, semi-erect, or trailing. Blackberries are an aggregate fruit that have a torus surrounded by drupelets containing pyrenes (seeds). Thus, the blackberry fruit are very delicate at full ripeness. There are many different cultivars of blackberries that are grown commercially. Most cultivars are floricanes fruiting, producing berries on the second-year growth (floricane). However, there are cultivars that produce fruit on first-year growth (primocane). Blackberries are grown domestically and internationally for both fresh and processing industries.

Fresh-market blackberries are harvested for direct sale to consumers, while processing blackberries are harvested for other uses, such as freezing, canning, and beverages. Blackberries can be harvested by hand or machine, and the method of harvesting depends on the architecture of the plant, the use of the fruit, and other factors. Fresh-market blackberries are hand-picked to ensure the fruit maintains quality from harvest to consumption, typically 10-14 days. Depending on cultivar, blackberries can be harvested from either floricane for two to three weeks or around eight weeks for double-cropped (primocane and floricanes fruiting) as fruit on the different cane types ripens at different times (Clark and Perkins-Veazie, 2011). However, hand harvesting has downfalls, as berries must be harvested at peak ripeness; while firm and shiny black but not overripe. If there is an overabundance of fruit, limits in labor availability, or weather disruptions can lead to yield loss at harvest or a surplus of underutilized produce. These events are



devastating for fresh-market blackberry growers, since primary quality fruit sells for much more than secondary quality fruit. The U.S. Department of Agriculture (USDA) has strict guidelines on the grades and standards for the sale of fresh-market blackberries (USDA, 2018). These postharvest quality attributes include decay (visible rot or mold on berries), leakage (juice leaking from berries), shrivel (drying of berries), and red drupelet reversion (drupelets on the berry turn from black to red). Quality of fresh-market blackberries must be maintained from harvest, during storage, and consumer purchase.

The method of harvesting blackberries impacts the potential for shipping and storage. Since the blackberries are delicate, damage at harvest can drastically reduce the postharvest storage potential. Edgley et al. (2020) reported that mechanical injury caused anthocyanin (color) degradation and increased red drupelet reversion. Edgley et al. (2019) found that ‘Ouachita’ berries exposed to impact damage at a warmer initial temperature (25 °C) had increased rates of red drupelet reversion compared to berries that were harvested at cooler temperatures (15 °C). Other researchers also found that the time of day impacts red drupelet reversion, with most reversion occurring within 24 hours after entering cool storage, although the number of reverted drupelets can continue to increase during postharvest storage (Armour et al., 2021; Edgley et al., 2019; McCoy et al., 2016; Yin, 2017). However, Felts et al. (2020) showed there was minimal impact of harvest time (7:00 am versus 12:00 pm), but blackberry genotype (cultivar or breeding selections) and length of time in storage had the greatest impact. Other studies found that red drupelet reversion may not be caused by changes in anthocyanin content, but rather a loss of cellular anthocyanin accumulation and therefore a break between anthocyanin and other cellular compounds (Flores-Sosa et al., 2021).

There have been recent advancements using automation in the food industry as well as the agriculture industry. Automated machine harvesters have become common in agriculture to offset the decreasing harvest workforce and increasing costs. Harvest labor costs can account for over one-fourth of total labor costs (Feng et al., 2018). Concern for the welfare of the workers, most migrants, spurred the development of mechanical harvesters to alleviate labor costs (Morris, 1999). In 1964, a prototype was developed at the University of Arkansas for one of the first mechanical harvesters for caneberries by mechanically shaking the canes (Morris et al., 1978). Most mechanical harvesters have beater bars that shake the plant and cause the berries to fall from the plant into a container or onto a conveyer. The harvester units can be designed to run down the row (beside the plants) or over the rows (over the plants). Takeda and Peterson (1999) showed existing bramble mechanical harvesters can detach blackberries from the plant more cheaply than hand harvesting, however the fruit did not maintain fresh-market quality standards. Cavender et al. (2014) evaluated an over-the-row rotary harvester but was not ideal because of the damage caused to the blackberries. Pérez-Pérez et al. (2018) reported that vibration increased incidences of red drupelet reversion. In addition, mechanical harvesters were unable to discern stage of ripeness and berries were harvested regardless of ripeness. Robotic harvesters with infrared sensors have been used to harvest strawberries based on fruit ripeness by cutting the stem without touching the berry (Xiong et al., 2019). The caneberries, such as blackberries and raspberries, pose challenges for robotic harvesting since the fruit is delicate, hidden in a dense canopy of leaves and canes, and fruit ripens indeterminately for 3-4 weeks.

Advancements have been made in robotics that can be applied in terms of harvesting specialty crops. Soft robotic grippers, made from rubber, silicone, or other flexible and durable materials, driven by an actuation mechanism have been introduced into the fruit harvest sector.

Soft robotics were initially used to perform minimally invasive surgeries, while minimizing tissue damage (Runciman et al., 2019). Much like surgery, the goal of fresh-market machine harvesting is to minimize damage to delicate fruit tissue to prolong postharvest storage. Soft robotics are ideal for grasping and manipulating delicate objects. The action that causes the mechanical device operate (actuation) and control of pressure distribution (force to grasp or pull) can be programmed at the design stage reducing the need for high-resolution sensory feedback (Venter and Dirven, 2017). Different thicknesses of cables inside the soft touch coating can mimic the force exerted in relation to harvesting the fruit by hand. The cable actuation provides motion and gives dexterity to the robotic device. These soft robotic grippers can mimic the motion of a human hand.

Continuum robots, robots that do not have rigid links or joints but can bend continuously, are a good candidate for fruit harvesting. These robots can have multiple “arms” that can demonstrate dexterous control, bending, and stiffening while working simultaneously. The downfall is that they need some sort of base support, and it is more difficult to exert force over a longer length span (Runciman et al., 2019). Soft robotics must have a balance of sensitivity and durability, especially for agricultural use. Touch of objects provides feedback to the controls relaying information such as the position of the object, the service curvature, friction, and force exerted by robots (Bartolozzi et al., 2016). Current advancements in soft robotics can emulate the movement of a human hand. Development of a soft robotic gripper has the capacity to reduce labor-intensive harvesting (Venter and Dirven, 2017).

The development of a harvester that can optimize the quality and quantity of fruit harvested is important to the future of the fresh-market blackberry industry. Therefore, the purpose of this project was to develop and evaluate the potential for a soft robotic gripper for

harvesting fresh-market blackberries. A custom-made force sensing apparatus was developed to quantify the forces associated with hand harvesting blackberries and identify appendages essential for harvesting for the development of a soft robotic gripper prototype. Physical, composition, and marketability attributes of different cultivars of blackberries were evaluated to provide guidance for developing a soft robotic gripper prototype and evaluate the impact on quality of blackberries harvested using the soft robotic gripper prototype.

## **Materials and Methods**

### **Blackberry cultivars and harvest**

Four blackberry cultivars, ‘Natchez’, ‘Osage’, ‘Prime-Ark<sup>®</sup> Traveler’, and ‘Sweet-Ark<sup>®</sup> Caddo’ were harvested from the floricanes in late June to early July of 2020 and 2021 from a commercial grower in Fayetteville, Arkansas (Cold Hardiness Zone 6b). Berries were harvested at the shiny-black stage of ripeness from 7:00-10:00 am. About 2 kg of each cultivar was harvested and placed into 170 g (6 oz) vented clamshells in triplicate. In each year for each cultivar, 240 berries were harvested (20 berries per clamshell). In both years, blackberries were gently hand harvested and used as a comparison to the berries harvested with a custom-made force sensing apparatus (2020) and a soft robotic gripper prototype (2021).

**Harvest 2020.** The blackberries were harvested by a person wearing a custom-made force sensing apparatus to measure forces applied by the thumb and each finger on berries during harvest in 2020 (Fig. 1). The apparatus was designed with resistive force sensors (FlexiForce A301, Tekscan, South Boston, MA) placed on silicone finger covers positioned on the thumb and three fingers (index, middle, and ring) of the right hand. Sensors were oriented on silicone finger covers to maximize contact with the berry surface during harvesting. Voltage data was measured by pairing each force sensor with a single power source non-inverting op-amp circuit.

Voltage measurements were sent through Bluetooth to MATLAB (MathWorks, Inc. Natick, MA) then converted to force values measured in Newtons (N). Data recording and processing were conducted in a portable water-resistant case housed in a backpack.

**Harvest 2021.** The blackberries were harvested by the soft robot gripper prototype in 2021 (Fig. 2). The gripper was designed with a three prong “finger” system made of silicone and an internal structure of a “tendon”. The tendon was a guitar string (36-gauge, Ernie Ball, CA) that was terminated in the upper plastic component with a 3 mm lateral offset from the nitinol (a metal alloy of nickel and titanium) strip. This offset eccentrically loads the finger, resulting in inward bending during tendon retraction. The silicone fingers were mounted on a custom-designed lead screw mechanism that was mounted beneath the fingers, providing a method for retracting the tendons. The berries were harvested at a fingertip contact force value of 0.69 N. For more information on the design of the soft robotic gripper prototype refer to Gunderman et al. (2022). The prototype was manually placed in the position to initiate grasping, harvest, and release of the berry into the clamshell.

### **Attribute analysis**

After harvest, the clamshells of blackberries were closed, placed in lugs, and transported to the University of Arkansas System Division of Agriculture Food Science Department, Fayetteville. In 2020 and 2021, physical attributes and composition attributes were evaluated at harvest and marketability attributes were evaluated after postharvest storage for 21 days at 2 °C.

**Physical attribute analysis.** Five berries per cultivar and replication were used for berry attributes. Each berry was weighed (g) using a precision digital scale (PA224 Analytic Balance, Ohaus Corporation, Parsippany, NJ), then the length (mm) and width (mm) were measured using digital calipers. Firmness of each berry was measured by a Stable Micro Systems TA.TX. XT

plus Texture Analyzer (Texture Technologies Corporation, Hamilton, MA). Firmness was measured using a 7.6-cm diameter cylindrical probe to compress with a trigger force of 0.02 N, an individual berry placed horizontally on a flat surface. The force needed to compress the berry was measured in Newtons (N). The berries were then frozen (-10 °C) for compositional analysis.

**Composition attribute analysis.** Composition of the juice from five berries per cultivar and replication were measured for soluble solids, pH, and titratable acidity. The five berries were thawed at room temperature (21 °C) and squeezed through cheesecloth to extract juice for analysis. Soluble solids of the juice were measured and expressed as percent (%) using an Abbe Mark II refractometer (Bausch and Lomb, Scientific Instrument, Keene, NH). The pH and titratable acidity were measured using a Metrohm 862 Compact Titrosampler (Metrohm AG, Herisau, Switzerland) fitted with a pH meter. The titratable acidity (expressed as % w/v (g/100 mL) citric acid) of juice was measured using a 3-g sample added to 50 mL degassed, deionized water and titrated with 0.1 N sodium hydroxide to an endpoint of pH 8.2.

**Marketability.** Marketability attributes (decay, leakiness, and red drupelet reversion) were evaluated after postharvest storage for 21 days at 2 °C. The decay of each blackberry was evaluated by determining if the blackberry had visible mold or rot on the blackberry surface. Decay of the blackberries in a clamshell was calculated as (number of decayed berries / number of total berries) × 100 and expressed as a percent. The leakiness was evaluated by gently rolling each blackberry on a white paper towel to determine if the berry leaked. The leakage of the blackberries in a clamshell was calculated as (number of leaky berries / number of total berries) × 100 and expressed as a percent. The red drupelet reversion (black drupelets changed to red) of the blackberries was evaluated by determining if each blackberry in the clamshell had more than one red drupelet. Red drupelet reversion was calculated as (number of berries that developed red

drupelets / number of total berries) x 100 and expressed as a percent.

### **Statistical design and analysis**

In both years, two harvest methods and four cultivars ('Natchez', 'Osage', 'Prime-Ark<sup>®</sup> Traveler', and 'Sweet-Ark<sup>®</sup> Caddo') were evaluated in triplicate for physical and composition attributes at harvest and marketability attributes after postharvest storage for 21 day at 2 °C. In 2020, the harvest methods were hand and a custom-made force sensing apparatus (sensor). In 2021, the harvest methods were hand and a soft robotic gripper prototype (gripper). Data was analyzed by year. Statistical analysis was conducted using JMP<sup>®</sup> Pro Statistical Software (version 16.0; SAS Institute Inc., Cary, NC). A univariate analysis of variance (ANOVA) was used to determine the significance of the main factors and their interactions. Tukey's Honest Significant Difference test or Student's T test were used to detect differences among means ( $p < 0.05$ ). Figures were created with each standard error bar constructed using 1 standard error from the mean.

### **Results and Discussion**

The 2020 and 2021 blackberry harvest seasons was relatively typical in Fayetteville, AR with an average high temperature of 24 °C and a low of 19 °C during June for both years. The 2021 harvest season had additional challenges resulting from a record freeze (-25 °C) in February followed by a frost in April impacting plant survival and fruit production of some cultivars. Harvest temperatures averaged 21 °C in 2020 and 2021.

#### **Custom-made force sensing apparatus**

The force measured using a custom-made force sensing apparatus on the thumb and fingers were significantly different for harvesting each cultivar of blackberry (Table 1). 'Sweet-Ark<sup>®</sup> Caddo' (1.18 N) had the highest force on the thumb, and 'Prime-Ark<sup>®</sup> Traveler' (0.51 N)

had the lowest. For the index and middle fingers, ‘Natchez’ had the highest force (0.27 N and 0.49, respectively) and ‘Osage’ had the lowest (0.09 N and 0.31 N, respectively). In addition, ‘Sweet-Ark<sup>®</sup> Caddo’ also had the lowest force on the middle finger (0.31 N). For the ring finger, ‘Sweet-Ark<sup>®</sup> Caddo’ (0.15 N) had the highest force, and ‘Prime-Ark<sup>®</sup> Traveler’ (0.01 N) had the lowest. Generally, the size of the berry was related to the force needed to harvest. ‘Sweet-Ark<sup>®</sup> Caddo’ and ‘Natchez’ were the largest berries (7-8 g), while ‘Osage’ and ‘Prime-Ark<sup>®</sup> Traveler’ were smaller (3-4 g) (Table 2). ‘Sweet-Ark<sup>®</sup> Caddo’, the largest berry, had the greatest force on the thumb needed to harvest.

Regardless of cultivar, the thumb applied the highest force (0.77 N), followed by middle finger (0.37 N), index finger (0.16 N), and ring finger (0.06 N). For harvesting these blackberries, the thumb and middle finger were the primary force applicators while the index and ring fingers stabilized the berry. In terms of providing guidance to develop a robotic soft gripper prototype, the underutilization of the ring finger in the harvest of blackberries showed the potential for designing a gripper with three robotic fingers (Gunderman et al., 2021; Gunderman et al., 2022). The force sensing apparatus for harvesting blackberries in 2020 provided data on force parameters and appendages used in the design of a soft robotic gripper prototype in 2021. Thus, a soft robotic gripper with three silicon fingers was designed to harvest blackberries using tendons to expand, grasp, and then release the berry.

### **Physical and composition attributes at harvest**

The cultivars harvested in 2020 and 2021 were evaluated for physical attributes (berry weight, length, width, and firmness) and composition attributes (soluble solids, pH, and titratable acidity) at harvest. In either year, cultivar impacted the physical and composition attributes more than harvest method. For both years berries were 4-8 g, 19-31 mm long, 19-23 mm wide, 6-10 N



firm, 8-12% soluble solids, 3.1-3.4 pH, and 1.0-1.4% titratable acidity. The physical and compositional attributes were within ranges established by previous research on Arkansas fresh-market blackberries. Segantini et al. (2017) harvested 11 genotypes in 2015, with firmness 4.9-9.0 N, soluble solids 4.7-19.5%, pH 3.0-3.4, and titratable acidity 0.5-1.5%. The main and interaction effects of harvest method and cultivar are shown in Table 1 (2020) and Table 2 (2021).

**2020.** Only soluble solids had a significant harvest method x cultivar interaction (12.05-12.42%) but none of the cultivars or harvest methods were significantly different from each other (data not shown). The only harvest method that was significant was firmness, where berries that were hand harvested were firmer than blackberries harvested with the custom-made force sensing apparatus (8.25 N and 6.96 N, respectively). There were significant differences among cultivars for weight, width, firmness, pH and titratable acidity. Berry length of these cultivars was 21-30 mm. ‘Sweet-Ark<sup>®</sup> Caddo’ had the highest berry weight (8.31 g) and firmness (9.22 N). ‘Natchez’ had the widest berry (22.65 mm), lowest pH (3.09), and highest titratable acidity (1.36%). ‘Osage’ had the lowest berry weight (3.88 g) and narrowest berry (19.57 mm), and ‘Osage’ and ‘Prime-Ark<sup>®</sup> Traveler’ had the highest pH (3.33).

**2021.** Only berry width had a significant harvest method x cultivar interaction with ‘Osage’ and ‘Sweet-Ark<sup>®</sup> Caddo’ harvested by hand was significantly different from the other two cultivars (data not shown). Harvest method did not impact weight, firmness, soluble solids, or pH (Table 3). There were significant differences among cultivars for all physical and composition attributes. ‘Natchez’ had the largest berry weight (8.39 g), length (31.33 mm), and firmness (9.80 N). ‘Prime-Ark<sup>®</sup> Traveler’ had the smallest berry in terms of weight (4.43 g) and length (19.16 mm), but had the highest soluble solids (11.03%) and lowest titratable acidity (0.99%).

### **Marketability attributes during postharvest storage**

The blackberry cultivars harvested in both years were harvested by two methods (hand and sensor in 2020 and hand and gripper in 2021) stored at 2 °C and evaluated at 21 d for marketability attributes. At harvest in both years, there was minor decay (<0%) and red drupelet reversion (<5%), but leakage was higher in 2021 (<35%) than 2020 (data not shown). Regardless of cultivar in both years after 21 d storage, there was minimal weight loss (<20%), leakage (<41%), decay (<32%), and red drupelet reversion (<12%) (Fig. 4). Armour et al. (2021) also reported berries with a firmer texture had a lower incidence in red drupelet reversion. In both years, red drupelet reversion was low (<12%) at 21 d storage and firmness of the berries was 6-10 N.

In comparison with previous research of marketability of Arkansas fresh-market blackberries, Felts et al. (2020) evaluated storage at 10 °C for 14 d, and weight loss was 13%, leakage was 40%, decay was 70%, and red drupelet reversion was 5%. Although, storage temperature was 8 °C higher in that study, storage days in this study were 7 days shorter and marketability attributes were less negatively impacted using either harvest method.

**2020.** At harvest in 2020, there was no decay (0%) and minor red drupelet reversion (< 5 %), but leakage was < 20% which could have been caused since berries were warmer just after harvest (data not shown). The interaction between harvest method x cultivar was not significant for any marketability attributes (Table 4). The main effects of both harvest method and cultivar were not significant for leakage, decay, or red drupelet reversion. Weight loss was not impacted by harvest method but was impacted by cultivar. The two smaller cultivars, ‘Osage’ and ‘Prime-Ark<sup>®</sup> Traveler’, had higher weight loss (19.32 and 17.73%, respectively) than the larger cultivars, ‘Natchez’ and ‘Sweet-Ark<sup>®</sup> Caddo’ (14.53 and 12.36%, respectively).

**2021.** At harvest in 2021, there was no decay (0%) or red drupelet reversion (0%), and leakage was <8% (data not shown). The interaction between harvest method x cultivar was significant for weight loss, decay, and red drupelet reversion but not leakage (Table 5 and Fig. 3). Fig. 3 shows that ‘Prime-Ark<sup>®</sup> Traveler’ had significant differences between harvest methods (hand versus gripper) for weight loss (6.87 and 11.06%, respectively) and red drupelet reversion (1.91 and 21.35%, respectively), while ‘Osage’ had significant difference in harvest methods for decay (21.62 and 3.25%, respectively). For this prototype, the contact force was the same regardless of the cultivar harvested. Impact on quality parameters could be minimized by adjusting the contact force of the gripper based on the cultivar size, shape, or firmness.

Fig. 4 shows the marketability attributes at 21 d postharvest storage at 2 °C in both years averaged over cultivars. With the sensor and the gripper, contact with the berry caused more weight loss (about 2%), leakage (5-14%), decay (about 1%), and red drupelet reversion (2-7%) as compared to hand harvest. However, for a delicate fruit like blackberries this is considered minimal damage at 21 days postharvest.

### **Conclusion**

A custom-made force sensing apparatus for hand harvesting blackberries was developed to quantify the forces applied by the thumb and fingers during harvesting, identifying essential harvesting appendages. The thumb and middle finger used the greatest force, whereas the index and ring fingers used lower forces to harvest blackberries while the ring finger primarily acted as a stabilizer. A soft robotic gripper prototype with three fingers was developed and evaluated. The forces applied to grab, stabilize, and harvest the blackberries with the custom-made force sensing apparatus and the soft robotic gripper prototype caused minimal marketability damage after postharvest storage. In 2020, there was minimal difference in marketability attributes of hand

harvesting versus harvesting using the custom-made force sensors. However, in 2021, weight loss, leakage, decay, and red drupelet reversion was higher in blackberries harvested with the soft robotic gripper prototype as compared to hand harvest. Leakage had the biggest difference between the two methods, 24% hand, 38% gripper, but these are still considered low for blackberries stored for 21 days. This project determined harvest and postharvest marketability impacts to further develop a soft robotic gripper for robotic harvesting of fresh-market blackberries. Further work will need to be done to refine the gripper design, integrate sensory tools to identify ripe berries, adjust gripper contact force for different cultivars, and determine the impact of placing the berries in clamshells. This project is only the first step to develop a soft robotic gripper to create an autonomous harvester for fresh-market blackberries.

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**Table 1.** Force (N) determined using a custom-made apparatus with sensors on the thumb and three fingers placed on the hand for harvesting fresh-market blackberries (Arkansas, 2020).

<b>Cultivar<sup>z</sup></b>	<b>Thumb</b>	<b>Index</b>	<b>Middle</b>	<b>Ring</b>
Natchez	0.75 b	0.27 a	0.49 a	0.05 b
Osage	0.65 bc	0.09 b	0.31 b	0.03 bc
Prime-Ark <sup>®</sup> Traveler	0.51 c	0.10 b	0.37 ab	0.01 c
Sweet-Ark <sup>®</sup> Caddo	1.18 a	0.17 ab	0.31 b	0.15 a
<i>P-value</i>	<i>&lt;0.0001</i>	<i>&lt;0.0001</i>	<i>0.0136</i>	<i>&lt;0.0001</i>
<b>Average</b>	<b>0.77</b>	<b>0.16</b>	<b>0.37</b>	<b>0.06</b>

<sup>z</sup>Cultivars were evaluated in triplicate. Means with different letter(s) for each attribute are significantly different ( $p < 0.05$ ) using Tukey's Honestly Significant Difference test.

**Table 2.** Main and interaction effects of harvest method (hand versus sensor) and cultivar on physical and composition attributes at harvest for fresh-market blackberries grown in Arkansas (2020).

<b>Effects<sup>y</sup></b>	<b>Berry weight (g)</b>	<b>Berry length (mm)</b>	<b>Berry width (mm)</b>	<b>Firmness (N)</b>	<b>Soluble solids (%)</b>	<b>pH</b>	<b>Titrateable acidity (%)<sup>z</sup></b>
<i>Harvest method<sup>x</sup></i>							
Hand	6.25 a	25.88 a	21.72 a	8.25 a	12.17 a	3.25 a	1.15 a
Sensor	5.75 a	26.87 a	21.01 a	6.96 b	12.16 a	3.25a	1.19 a
<i>P-value</i>	<i>0.3328</i>	<i>0.7188</i>	<i>0.0632</i>	<i>0.0373</i>	<i>0.9645</i>	<i>0.8605</i>	<i>0.2641</i>
<i>Cultivar</i>							
Natchez	7.51 a	29.14 a	22.65 a	7.81 ab	12.05 a	3.09 b	1.36 a
Osage	3.88 b	24.80 a	19.57 c	6.83 b	12.08 a	3.33 a	1.16 b
Prime-Ark <sup>®</sup> Traveler	4.29 b	21.42 a	20.70 b	6.56 b	12.42 a	3.33 a	1.02 c
Sweet-Ark <sup>®</sup> Caddo	8.31 a	30.14 a	22.53 a	9.22 a	12.10 a	3.25 a	1.13 bc
<i>P-value</i>	<i>&lt;0.0001</i>	<i>0.1205</i>	<i>&lt;0.0001</i>	<i>0.0183</i>	<i>0.4827</i>	<i>0.0007</i>	<i>&lt;0.0001</i>
<i>Harvest method x Cultivar</i>							
<i>P-value</i>	<i>0.1146</i>	<i>0.2368</i>	<i>0.1112</i>	<i>0.4987</i>	<i>0.0257</i>	<i>0.3524</i>	<i>0.3306</i>

<sup>z</sup>Titrateable acidity expressed as % citric acid.

<sup>y</sup>Cultivars were evaluated in triplicate. Means with different letter(s) for each attribute within effects are significantly different ( $p < 0.05$ ) using Students t-test.

<sup>x</sup>Gentle hand harvest (hand) versus harvest with a custom-made force sensing apparatus (sensor) on thumb, index, middle, and ring fingers.



**Table 3.** Main and interaction effects of harvest method (hand versus gripper) and cultivar on physical and composition attributes at harvest for fresh-market blackberries grown in Arkansas (2021).

<b>Effects<sup>y</sup></b>	<b>Berry weight (g)</b>	<b>Berry length (mm)</b>	<b>Berry width (mm)</b>	<b>Firmness (N)</b>	<b>Soluble solids (%)</b>	<b>pH</b>	<b>Titrateable acidity (%)<sup>z</sup></b>
<i>Harvest method<sup>x</sup></i>							
Hand	5.76 a	23.56 b	19.66 b	7.70 a	9.67 a	3.23 a	1.27 a
Gripper	6.41 a	25.22 a	20.58 a	7.61 a	10.20 a	3.30 a	1.01 b
<i>P-value</i>	<i>0.0868</i>	<i>0.0329</i>	<i>0.0300</i>	<i>0.8576</i>	<i>0.1361</i>	<i>0.2772</i>	<i>0.0012</i>
<i>Cultivar</i>							
Natchez	8.39 a	31.33 a	21.39 a	9.80 a	9.58 b	3.28 ab	1.17 ab
Osage	5.20 c	21.51 c	20.13 b	6.33 b	10.63 a	3.43 a	1.08 b
Prime-Ark <sup>®</sup> Traveler	4.43 c	19.16 d	18.87 c	6.71 b	11.03 a	3.24 ab	0.99 b
Sweet-Ark <sup>®</sup> Caddo	6.31 b	25.55 b	20.10 b	7.78 b	8.48 c	3.13 b	1.32 a
<i>P-value</i>	<i>&lt;0.0001</i>	<i>&lt;0.0001</i>	<i>0.0032</i>	<i>0.0005</i>	<i>0.0003</i>	<i>0.0298</i>	<i>0.0178</i>
<i>Harvest method x Cultivar</i>							
<i>P-value</i>	<i>0.1235</i>	<i>0.3917</i>	<i>0.0111</i>	<i>0.3173</i>	<i>0.0655</i>	<i>0.1921</i>	<i>0.3564</i>

<sup>z</sup>Titrateable acidity expressed as % citric acid.

<sup>y</sup>Cultivars were evaluated in triplicate. Means with different letter(s) for each attribute within effects are significantly different ( $p < 0.05$ ) using Students t-test.

<sup>x</sup>Gentle hand harvest (hand) versus harvest with a soft robotic gripper prototype (gripper).

**Table 4.** Main and interaction effects of harvest method (hand versus sensor) and cultivar stored for 21 d at 2 °C on marketability attributes for fresh-market blackberries grown in Arkansas (2020).

<b>Effects<sup>z</sup></b>	<b>Weight loss (%)</b>	<b>Leakage (%)</b>	<b>Decay (%)</b>	<b>Red drupelet (%)</b>
<b>Harvest method<sup>x</sup></b>				
Hand	15.01 a	1.25 a	0.42 a	3.33 a
Sensor	16.96 a	6.25 a	0.42 a	5.00 a
<i>P-value</i>	<i>0.0724</i>	<i>0.0760</i>	<i>0.9999</i>	<i>0.3464</i>
<b>Cultivar</b>				
Natchez	14.53 b	5.00 a	0.00 a	6.67 a
Osage	19.32 a	2.50 a	1.67 a	0.83 a
Prime-Ark <sup>®</sup> Traveler	17.73 a	6.67 a	0.00 a	3.33 a
Sweet-Ark <sup>®</sup> Caddo	12.36 b	0.83 a	0.00 a	5.83 a
<i>P value</i>	<i>0.0008</i>	<i>0.4326</i>	<i>0.1546</i>	<i>0.1106</i>
<b>Harvest method x Cultivar</b>				
<i>P-value</i>	<i>0.3081</i>	<i>0.6243</i>	<i>0.9999</i>	<i>0.3787</i>

<sup>z</sup>Cultivars were evaluated in triplicate. Means with different letter(s) for each attribute within effects are significantly different ( $p < 0.05$ ) using Students t-test.

<sup>x</sup>Gentle hand harvest (hand) versus harvest with a custom-made force sensing apparatus (sensor) on thumb, index, middle, and ring fingers.

**Table 5.** Main and interaction effects of harvest method (hand versus gripper prototype) and cultivar stored 21 d at 2 °C on marketability attributes for fresh-market blackberries grown in Arkansas (2021).

<b>Effects<sup>z</sup></b>	<b>Weight loss (%)</b>	<b>Leakage (%)</b>	<b>Decay (%)</b>	<b>Red drupelet (%)</b>
<i>Harvest method<sup>y</sup></i>				
Hand	5.98 b	23.58 b	16.67 a	1.40 b
Gripper	7.67 a	37.96 a	17.93 a	8.74 a
<i>P-value</i>	<i>&lt;0.0001</i>	<i>0.0071</i>	<i>0.7478</i>	<i>0.0012</i>
<i>Cultivar</i>				
Natchez	5.59 c	40.07 a	10.95 b	6.15 ab
Osage	6.32 b	8.49 b	12.44 b	0.00 c
Prime-Ark <sup>®</sup> Traveler	8.97 a	38.19 a	31.82 a	11.63 a
Sweet-Ark <sup>®</sup> Caddo	6.41 b	36.33 a	13.99 b	2.50 bc
<i>P value</i>	<i>&lt;0.0001</i>	<i>0.0005</i>	<i>0.0047</i>	<i>0.0027</i>
<i>Harvest method x Cultivar</i>				
<i>P-value</i>	<i>&lt;0.0001</i>	<i>0.3020</i>	<i>0.0231</i>	<i>0.0119</i>

<sup>z</sup>Cultivars were evaluated in triplicate. Means with different letter(s) for each attribute within effects are significantly different ( $p < 0.05$ ) using Students t-test.

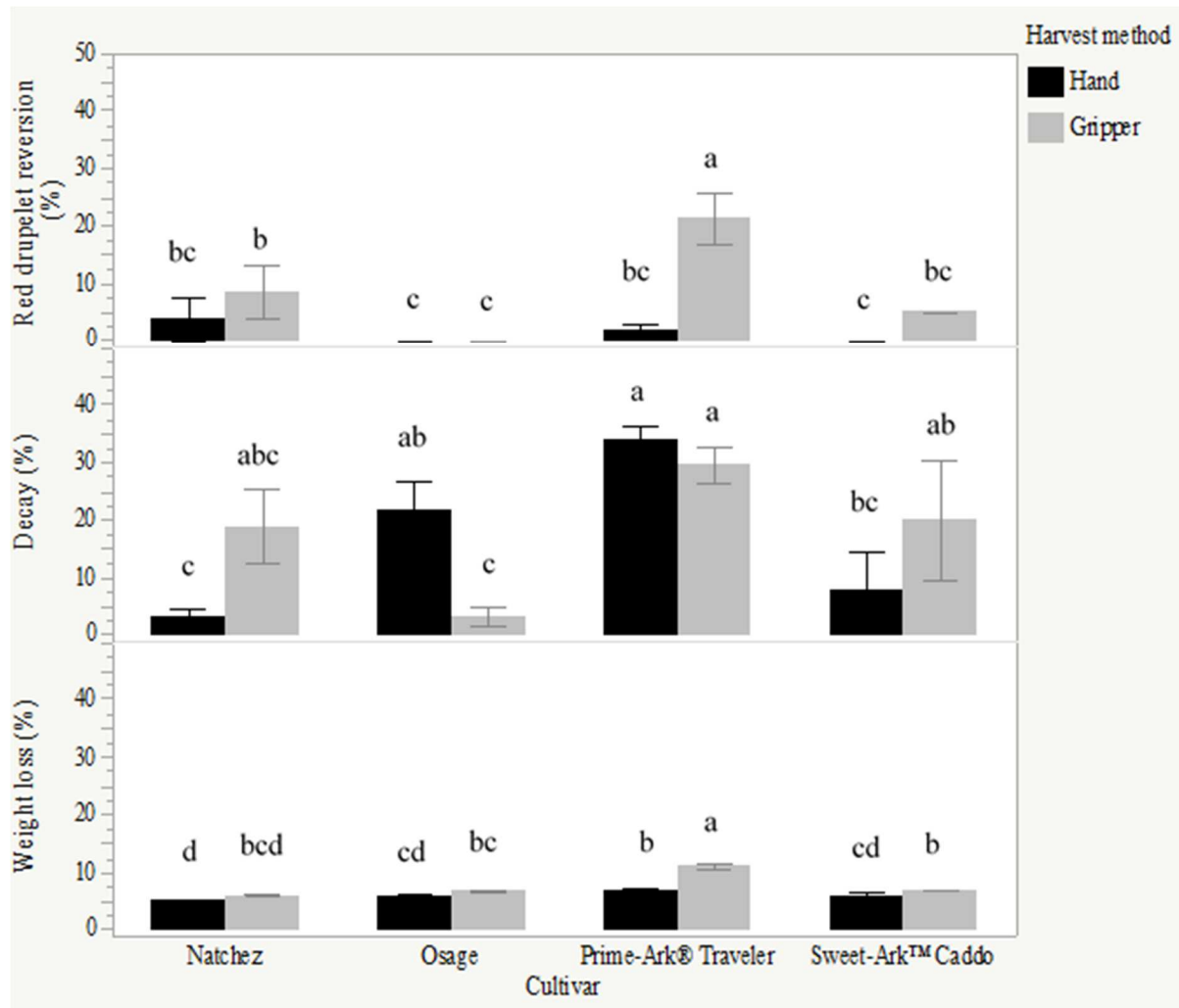
<sup>y</sup>Gentle hand harvest (hand) versus harvest with a soft robotic gripper prototype (gripper).



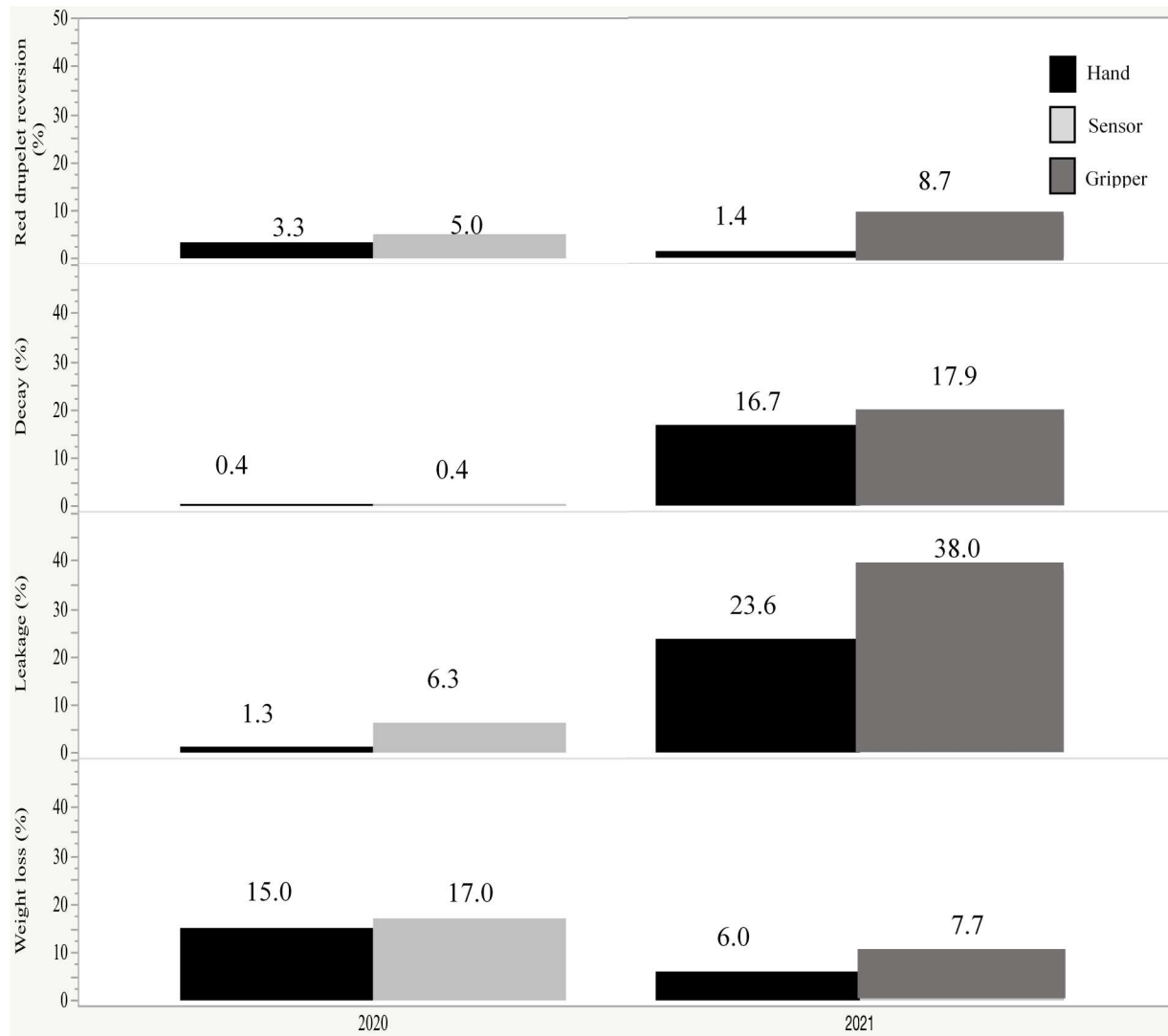
**Fig. 1.** Custom-made force sensing apparatus developed at the University of Arkansas with sensors on the thumb and three fingers (left) placed on the hand for harvesting (right) fresh-market blackberries (Arkansas, 2020).



**Fig. 2.** Soft robotic gripper prototype developed at the University of Arkansas grasping a fresh-market blackberry (Arkansas, 2021)



**Fig. 3.** Marketability attributes after postharvest storage for 21 days at 2 °C for different cultivars of fresh-market blackberries grown in Arkansas harvested by hand versus gripper. Cultivars were evaluated in triplicate. Means with different letter(s) for each attribute within effects are significantly different ( $p < 0.05$ ) using Student's *t*-test. Gentle hand harvest (hand) versus harvest with a soft robotic gripper prototype (gripper). Each standard error bar is constructed using 1 standard error from the mean.



**Fig. 4.** Marketability attributes after postharvest storage for 21 days at 2 °C for fresh-market blackberries grown in Arkansas harvested by hand versus custom-made force sensing apparatus (sensor) in 2020 and hand versus soft robotic gripper prototype in 2021. *Four cultivars were evaluated in triplicate.*

## OVERALL CONCLUSIONS

The purpose of this research on Arkansas fresh-market blackberries was to 1) identify the unique attributes 2) determine the best handling practices to increase postharvest quality, and 3) evaluate the potential for a soft robotic gripper for harvesting. For the first objective, blackberry genotypes were harvested in 2020 and 2021 from the University of Arkansas System Division of Agriculture Fruit Research Station in Clarksville, AR to evaluate physical, composition and volatiles and then some of the genotypes were evaluated for descriptive sensory attributes. The composition attributes of these genotypes differed each year but were within the typical ranges for fresh-market genotypes grown in Arkansas. There were 159 volatile aroma compounds identified in Arkansas blackberry genotypes in 2020 and 103 in 2021, mainly monoterpenes, esters, aldehydes, and alcohols. For descriptive sensory attributes in 2020, the genotypes differed in fruity aroma, green/unripe aromatics, and sour basic tastes but differed in overall intensity of aromatics and basic tastes in 2021. For objective two in 2020 and 2021, cultivar, harvest method, and acclimation temperature were examined to determine best handling practices for fresh-market blackberries to increase postharvest quality. Cultivar had the biggest impact on marketability attributes which varied by year. Overall, there were no clear trends on marketability degradation of the blackberries, and for both years the damage was considered low for fresh-market blackberries stored at 2 °C for 21 days. For objective three in 2020 and 2021, a custom-made force sensing apparatus (sensors) attached to the thumb and fingers of a person was developed in 2021 to determine the force (N) to harvest blackberries and the thumb and fingers essential for harvesting, then in 2021, this data was used to create a soft robotic gripper prototype (gripper) for harvesting blackberries. The force used by the thumb and middle finger (0.77 N and 0.37 N, respectively) were greatest for harvesting blackberries, whereas the index and ring



fingers used lower force (0.16 N and 0.06 N, respectively) primarily to stabilize the berry. A prototype of a 3-prong (fingered) soft robotic gripper was designed using results from the force sensing apparatus. The forces applied to grab, stabilize, and harvest blackberries with the sensors or gripper did not cause excessive marketability damage (20% weight loss, 40% leakage 32% decay, and 12% red drupelet reversion) to the blackberries at harvest or after 21 days at 2 °C postharvest storage. This project identified unique attributes, determined the best handling practices to increase postharvest quality, and evaluated the potential for a soft robotic gripper for harvesting.

## Appendix



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**To: From:**

**Date: Action: Action Date: Protocol #: Study Title:**

Renee Terrell Threlfall FDSC B-3

Douglas James Adams, Chair IRB Committee

04/01/2019

**Exemption Granted**

04/01/2019 1903180959

Identify marketable attributes of commercial and Arkansas fresh-market blackberry genotypes

The above-referenced protocol has been determined to be exempt.

If you wish to make any modifications in the approved protocol that may affect the level of risk to your participants, you must seek approval prior to implementing those changes. All modifications must provide sufficient detail to assess the impact of the change.

If you have any questions or need any assistance from the IRB, please contact the IRB Coordinator at 109 MLKG Building, 5-2208, or [irb@uark.edu](mailto:irb@uark.edu).

cc: John R Clark, Key Personnel  
Margaret Leigh Worthington, Key Personnel