ABSTRACT

KADERABEK, LAURA ELLEN BARTH. Effects of Aging on the Physical, Chemical, and Hydrologic Properties of Pine Bark Substrates. (Under the direction of Dr. Brian E. Jackson and William C. Fonteno).

Pine bark (PB) is one of the most commonly used horticultural substrates, and may be used as a fresh, aged, or composted product. Aged PB is most commonly used; however, interest and demand for fresh PB has increased. While both aged and fresh PB can be used successfully, little research has been done to investigate the differences between fresh and aged PB, as well as PB of specific ages. Additionally, the actual age of PB used for research in the literature is usually not reported, making it hard to extrapolate anything more than general trends for bark that is considered “fresh” or “aged.” To gain a greater understanding of the effects of aging and pile management on PB substrates, a twelve-month study was conducted at a local PB supplier in Eagle Springs, NC. Fresh PB was processed and fines (less than or equal to 1.27 cm) were placed in three pile replications of 191 cubic meters each. Beginning at time 0 and every 4 to 5 weeks for a period of twelve months, stratified subsamples were taken from each pile at three different heights (top, middle, and bottom) at horizontal depths of 0.3 – 1.2 m to account for variations in pile depth and height, and mixed into one composite sample for each pile to test the physical, hydrologic, and chemical properties. Physical properties included pile volume, temperature, particle size distribution, total porosity, container capacity, air space, bulk density, white wood content, and color. Hydrologic properties included wettability, saturated hydraulic conductivity, moisture retention curves, equilibrium capacity variable models for nursery containers, and pore fraction volume analysis. Chemical properties included pH, EC, pH buffering capacity, CEC, plant available nutrients, C: N ratio, lignocellulose content, and seedling germination tests. The
data generated from this study can improve the understanding, product consistency and usability of PB substrates of different ages.
Effects of Aging on the Physical, Chemical, and Hydrological Properties of Pine Bark Substrates

by
Laura Ellen Barth Kaderabek

A thesis submitted to the Graduate Faculty of
North Carolina State University
in partial fulfillment of the
requirements for the degree of
Master of Science

Horticultural Science

Raleigh, North Carolina
2017

APPROVED BY:

Dr. Brian Jackson
Committee Chair

Dr. William Fonteno
Committee Chair

Dr. Dean Hesterberg

Dr. Wei Shi

DEDICATION
“Equipped with his five senses, man explores the universe around him and calls the adventure Science.” - Edwin Powell Hubble

I dedicate this work to my family, who have always gone above and beyond to support me and help me achieve my goals, and for instilling in me a sense of curiosity, and a love of nature and all its wonders. I also dedicate this to my beloved mutts, for their unconditional love, entertainment, and cuteness; as well as their need to go outside periodically which prevented me from being a complete shut-in while writing this thesis.
Laura Ellen Barth Kaderabek was born in Eau Claire, WI to Dr. Robert J. Barth, a professor of archaeology at the University of Wisconsin – Eau Claire, and Janine L. Polk, an avid birder and herpetologist. After graduating with a degree in Music Performance (flute) from the University of Wisconsin – Eau Claire, she moved to North Carolina in 2007 in pursuit of adventure and warmer climes. The tall trees, lush greenery, extended growing season, and wide array of beautiful landscape plants helped Laura to foster a deeper connection to nature and gardening. After working at the Durham Crisis Response Center, a non-profit organization for survivors of domestic violence and sexual assault, for three years she decided to return to school for an undergraduate degree in horticulture at North Carolina State University. During her studies at NCSU, Laura had the opportunity to work in several labs on campus, including the Gould Entomology Biolab, the NCSU Plant Disease and Insect Clinic, and the Horticultural Substrates Lab, all of which afforded her opportunities for practical multi-disciplinary experience to round out her education, as well as participation in undergraduate research projects. Laura graduated with honors in May 2014, and was selected to be an intern in Dr. Tom Ranney’s plant breeding program at the Mountain Crop Improvement Lab in Mills River. After seven months of plant breeding and hiking to mountain peaks and waterfalls, Laura returned to Raleigh to pursue a Master of Science degree in horticulture under Drs. Brian Jackson and Bill Fonteno. When not playing in the dirt, Laura still utilizes her first degree and is a freelance musician in the Raleigh/Durham area. After completion of her Master’s degree, Laura will begin a doctorate program at Virginia Tech under Dr. Jim Owen.
ACKNOWLEDGMENTS

I feel incredibly grateful and humbled to have had the help and support of so many wonderful people during the course of this adventure. To my advisors, Drs. Fonteno and Jackson, you have done so much for me over the past 5 (!) years during both of my horticulture degrees at NCSU. The opportunities you have given me provided me with a greater scientific and practical knowledge of horticulture, awards, travel, connections, and friendships. I will always be deeply grateful for the support, guidance, patience, wisdom, and humor throughout my studies, and for your faith in my abilities. I have grown so much as a scientist, scholar, and professional, and I will use the tools that you have given me for the rest of my life’s journey, wherever it may take me. I hope you both know how much I have truly loved being a part of your program. Bill – thank you for always lending a sympathetic ear if I was struggling in any area of my life (or if I just needed to vent); for your advice; and for your scientific, artistic and musical talent and sensibilities. You are more than an advisor to me, you are a true friend and kindred spirit. Brian – you have taught me so much, both in and out of the classroom. Thank you for teaching me to always strive for more and never be complacent; to work as hard as I can but still have fun; to always maintain my curiosity because that is the core of research; and that taking action and just DOING is often the best solution if I’m thinking too hard and losing sight of the bigger picture. Your drive, dedication, and passion for what you do have continually inspired and motivated me, and after all these years I still want to be you when I grow up!

A HUGE thank you to Layne, Alvin and everyone at TH Blue, who provided the site, materials, and on-site management for this project. Without your expertise, time, and resources this research would not have been possible. I think I have learned more valuable practical information talking with you and seeing your (very successful) operation than I have in any of
my classes. Every time I go out to the research piles I am reminded of the scope, importance, and awesomeness of this project. Working with you has been a pleasure, and this has been such a cool experience for me. I hope we will keep in contact after my degree is completed!

To my committee members Drs. Dean Hesterberg and Wei Shi, thank you for your advice and expertise, and for always taking time out of your busy schedules to discuss any questions I had. I really enjoyed taking both of your classes, which were interesting and challenging, and provided me with a strong foundation in soil science that will carry me through my next degree and beyond.

A big thanks to Abby Wunch, Super Mike, Clay Harris, Corey Ames, Rob Schulz, Logan Haislip, Goose, Tucker Brandt, Adam LeMay, and Logan Stewart for the technical help in the lab. A lot of work was needed to complete all the tests that were required for this project, and I couldn’t have done it on my own. I had a lot of fun working with all of you in the lab!

To the former and current HSL grad students Ted Yap, Jeb Fields, Lesley Judd, Paul Bartley, and Turner Smith for the advice, insight, technical help, and sympathetic ears. Ted – thank you for your insight into the pine bark aging process as I was beginning my research. Lesley – thank you for always making time to assist me with any statistical or procedural needs that I had as an undergrad and throughout the course of this research, even after you graduated. Jeb – thank you for our discussions, and for helping me understand substrate physics and water relations better through your answers to my questions. Paul – I hope you know how much I’ve enjoyed working with you, and I will miss your curiosity, enthusiasm, passion, and wit. I’m looking forward to seeing all the great things that come out of your research, and competing with you at future conferences! Turner – I’m so glad that you joined our program, and it’s been great
working with someone who has so much enthusiasm and curiosity about substrates! I look forward to seeing your project develop, and hearing you present at future conferences.

Thank you to my graduate student peers in the department – I have learned from you, and had a lot of fun with you! There are many of you that I wish I could have spent more time with over the past two semesters. I hope we maintain the connections, no matter where life takes us.

Lastly, thank you to Dr. Wayne Robarge, April Shaeffer, Dr. Joy Smith, Dr. Cheryl Boyer, Guillermo Ramirez, Dr. Consuelo Arellano, Dr. Cavell Brownie for their assistance with various aspects of my research, and a huge thank you to Rachel McLaughlin for the administrative support and making sure my requirements were met and paperwork was completed.
# TABLE OF CONTENTS

LIST OF TABLES .......................................................................................................................... ix

LIST OF FIGURES ....................................................................................................................... xi

**Chapter I: Literature Review**

Literature Review ....................................................................................................................... 1

Literature Cited ............................................................................................................................. 12

Tables........................................................................................................................................ 16

**Chapter II: Changes in Pine Bark During the Aging Process I: Physical Properties** ............ 17

Abstract ......................................................................................................................................... 18

Introduction ................................................................................................................................. 20

Materials and Methods .............................................................................................................. 27

Results and Discussion ............................................................................................................... 33

Summary...................................................................................................................................... 39

Literature Cited ............................................................................................................................. 40

Tables........................................................................................................................................ 45

Figures......................................................................................................................................... 50

**Chapter III: Changes in Pine Bark During the Aging Process II: Hydrologic Properties** .... 60

Abstract ......................................................................................................................................... 61

Introduction ................................................................................................................................. 63

Materials and Methods .............................................................................................................. 68

Results and Discussion ............................................................................................................... 74

Summary ...................................................................................................................................... 79

Literature Cited ............................................................................................................................. 80
Chapter IV: Changes in Pine Bark During the Aging Process III: Chemical Properties ....97

Abstract................................................................. 98

Introduction ................................................................. 99

Materials and Methods ....................................................... 102

Results and Discussion ..................................................... 107

Summary ............................................................................... 110

Literature Cited .................................................................. 111

Appendices ........................................................................... 122

Appendix A ........................................................................... 123

Appendix B ........................................................................... 124

Appendix C ........................................................................... 125

Appendix D ........................................................................... 126
LIST OF TABLES

Chapter I: Introduction and Literature Review

Table 1.1 Scientific literature containing pine bark of specific ages........................................16

Chapter II: Changes in Pine Bark During the Aging Process I: Physical Properties

Table 2.1 Turning and sampling dates, and sample age for pine bark piles over the course of one year. ........................................................................................................45
Table 2.2 Color of PB over 12 months of aging using Munsell Soil Color.........................46
Table 2.3 Particle size distribution of pine bark over 12 months of aging...............................47
Table 2.4 White wood content of pine bark over twelve months of aging.........................48
Table 2.5 Sand content of pine bark (*Pinus palustris* Mill.) at 0, 3, 6, 9, and 12 months of aging.................................................................................................................49

Chapter III: Changes in Pine Bark During the Aging Process II: Hydrologic Properties

Table 3.1 Table 3.1. Dimensions of commonly used nursery containers which were used to calculate ECV models. ........................................................................................................86
Table 3.2 Particle size distribution of pine bark (*Pinus palustris* Mill.) over twelve months of aging.................................................................................................................87
Table 3.3 Saturated hydraulic conductivity ($K_{sat}$)values for pine bark (*Pinus palustris* Mill.) aged 0, 3, 6, 9, and 12 months............................................................................................................88
Table 3.4 Physical and hydrologic properties of pine bark (*Pinus palustris* Mill.) at 0, 3, 6, 9, and 12 months of aging.................................................................................................................89
Table 3.5 Physical properties of pine bark aged 0, 3, 6, 9, 12 months, derived from modeling equilibrium capacity variable values.................................................................90
Table 3.6 Sand content of pine bark (*Pinus palustris* Mill.) at 0, 3, 6, 9, and 12 months of aging..........................................................................................................................91

Chapter IV: Changes in Pine Bark During the Aging Process III: Chemical Properties
Table 4.1  Available nutrients of pine bark over twelve months aging..........................114

Table 4.2  pH buffering capacity, cation exchange capacity, exchangeable cations exchangeable acidity, and anion exchange capacity of pine bark after 0, 3, 6, 9, and 12 months of aging........................................................................................................115

Table 4.3  Elemental carbon, nitrogen, lignocellulose, and ash content and ratios of pine bark over 0, 3, 6, 9, and 12 months of aging........................................................................................................116

Table 4.4  Heavy metal detection of pine bark (Pinus palustris Mill.) over twelve months of aging.................................................................................................................................117

Table 4.5  Seedling germination and dry weights of three species grown in pine bark after 0, 3, 6, 9, and 12 months of aging........................................................................................................118

Table 4.6  Cucumber germination assay from pine bark water extracts over twelve months of aging.................................................................................................................................119
LIST OF FIGURES

Chapter II: Changes in Pine Bark During the Aging Process I: Physical Properties

Figure 2.1  Installation of the 12 month pine bark aging study. A) Fresh bark was brought from the lumber company and on the research site for further processing. B) The bark was ground and C) screened through a 1.27 cm screen into three fractions: nuggets, mini nuggets, and fines. D) The fines were placed in three piles (replications) of approximately 191 m3 each, with dimensions of approximately 16.8 m x 10.1 m x 3.2 m.................................50

Figure 2.2  Sampling schematic to illustrate height zones, conceptual grid for randomized sampling within height zone, and sampling depth. A) Stratified subsamples were taken from 9 locations on each pile at three different height “zones” (top, middle, and bottom). Three locations within each height were randomly selected by conceptually dividing the pile into a grid. B) At each sample location a hole was dug and were taken from horizontal depths of 0.3, 0.6, 0.9, and 1.2 m.........51

Figure 2.3  Example of a Munsell Soil Color card. Three coordinates – hue (A), value (B), and chroma (C) describe possible colors. Hue refers to the dominant spectral or rainbow color, value indicates the degree of lightness or darkness of a color, and chroma is the purity or saturation of a color. Samples are read in the order of hue value/chroma, e.g. 5YR 3/2...........................................52

Figure 2.4  Average pile temperatures (all heights and depths combined) for each sample date, average monthly temperatures, and accumulated precipitation (secondary Y axis)...............................................................53

Figure 2.5  Temperature measurements at depths of 0.3, 0.6, 0.9, and 1.2 m at three different height zones (top, middle, and bottom). Data are averaged between piles........54

Figure 2.6  Color changes of bark at (A) 0 months, (B) 2 months, (C) 3 months, (D) 4 months, (E) 5 months, (F) 6 months, (G) 7 months, (H) 8 months (I) 9 months, (J) 10 months, (K) 11 months, and (L) 12 months of aging using a Munsell Soil Color chart..........................................................55

Figure 2.7  Percentage of extra-large (>6.3 mm), large (2.0 to 6.3mm), medium (0.5 to 2.0 mm) and fine (< 0.05 mm) particles over twelve months of aging...............56

Figure 2.8  Percent differences in particle size fractions between 0 and 3, 0 and 6, 0 and 9, and 0 and 12 months of aging......................................................57

Figure 2.9  Total porosity (TP), container capacity (CC), air space (AS) and bulk density (BD) of pine bark over twelve months of aging...............................58
Figure 2.10  Average volume of pine bark piles and pile bulk density calculated from moisture content at time of sampling.

Chapter III: Changes in Pine Bark During the Aging Process II: Hydrologic Properties

Figure 3.1  Saturated hydraulic conductivity ($K_{sat}$) unit of Drzal (1994). Four 347.5 mL aluminum cores are fitted with individual funnel adaptor rings on the bottom and a plastic cylinder with a spout on the top. Each core assembly is secured onto an individual stationary funnel. A constant head reservoir, mounted to laboratory jacks, is filled with water and maintained at a constant level by a recirculation system. Rubber tubing is connected from the reservoir to the bottom of the stationary funnel, allowing water to saturate each core unit from the bottom. A hydraulic head is created by raising the reservoir a measured distance above the sample. Piezometers mounted to the reservoir and each core assembly are used to precisely measure hydraulic head differences.

Figure 3.2  Hydration efficiency curves of pine bark (Pinus palustris Mill.) over twelve months of aging at 50% and 25% moisture content by weight, with container capacity for each moisture content represented as solid lines.

Figure 3.3  Saturated hydraulic conductivity ($K_{sat}$) of pine bark (Pinus palustris Mill.) aged 0, 3, 6, 9, and 12 months.

Figure 3.4  Moisture retention curves and Van Genuchten models (log scale) of pine bark (Pinus palustris Mill.) at 0, 3, 6, 9, and 12 months of aging.

Figure 3.5  Pore fraction volume analysis.

Chapter IV: Changes in Pine Bark During the Aging Process III: Chemical Properties

Figure 4.1  pH and EC of pine bark samples over twelve months of aging. Three samples were measured from each pile with a handheld pH meter (HI 9811, Hanna Instruments, Ann Arbor, MI) using the 1:1 dilution method (Fonteno and Harden, 2010).

Figure 4.2  Composite pine bark samples from combined piles at 0, 3, 6, 9, and 12 months of aging were amended with pulverized dolomitic limestone (#100; Rockydale Quarries Corporation, Roanoke, VA) at rates of 0, 2.37, 4.75 kg/m3 (0, 4, and 8 lbs/yd3. Subsamples were taken each treatment on 1, 3, 5, 7, 14, and 21 days and substrate solution pH was measured at 0, 1, 3, 5, 7, 14, 21, 28 days using the 1:1 extraction method and a handheld pH meter (HI 9811, Hanna Instruments, Ann Arbor, MI).
Appendices

Appendix A  Processing of pine bark. Raw pine bark (A) is passed through a hammer-mill (B), and screened into three fractions: nuggets (C), mini-nuggets (D), and fines (less than or equal to 1.27 cm)..............................................................123

Appendix B  A drone with GPS mapping technology was used to create a site map using DroneDeploy 3-D mapping software (A). This 3-D map can be used to calculate the volume of inventory piles.............................................................................................124

Appendix C  Thermal imaging was used as potential technique to measure pile surface temperature (A), inner pile temperature during select sampling dates (B), and during turning on 13 Nov. 2015 (C).............................................................................................................125

Appendix D  Channeling and uneven moisture distribution of PB at 25% moisture content by weight after 10 hydration events (right) as compared to 50% moisture content by weight (left).............................................................................................................126
CHAPTER 1

Literature Review

History of Container Production and Organic Substrates

Many think of container production as a modern practice, but growing plants in containers has been documented at various points throughout history, dating back to the Egyptians almost 4,000 years ago. Wall paintings found in the temple of Deir el Bahari depict what appear to be mature trees being grown and transported in large containers (Raviv and Lieth, 2008). In the seventeenth century, exotic plants began to be cultivated indoors in orangeries to provide fruits, vegetables, and aesthetic value to the wealthy outside of the normal growing seasons or climates. This can be thought of as the first documented example of a container-growing system, although mineral soil was mostly used in these containers (Raviv and Lieth, 2008).

Composting is thought to be as old as agriculture itself, and was practiced by Chinese farmers and gardeners almost 4,000 years ago (King, 1927). The earliest known written reference to composting is found on clay tablets dated back to the Akkadian Empire, 2700 BC (Fitzpatrick et al., 1998). The noun “compost” first appeared in the English language in 1587, and the use of compost as a verb appeared in 1757. At the beginning of the eighteenth century we begin to see detailed descriptions of soil amendments with organic materials. Peat moss was suggested as an additive for soils with a high clay content, and animal dung was suggested for sandy soils. There is also documentation of an early hotbed that used partially fermented cow manure under a frame covered with glass. John Claudius Loudon, author of “Encyclopedia of Gardening” in 1839, listed many organic materials that could be used as plant growth media in greenhouses, and gave
detailed recipes for their composting, as well as suggestions for combining different ingredients for better plant growth (Raviv et al., 1986). Older references exist that describe the process and end-product of composting, but without the modern terms that we currently use, such as processing, aging, and stabilization. While the use of compost and organic matter in plant production are practices that have been employed for thousands of years, until recently, the reported benefits of these practices have been mostly anecdotal rather than the result of controlled scientific experimentation. The earliest scientific reference to aging/composting time is from Howard and Wad (1931), who suggested an optimum time of approximately six months for mixtures containing at least 75% high carbon material, such as plant debris (Fitzpatrick et al., 1998).

In 1934 a potting compost known as the John Innes mix was introduced. This mix consisted of “7 parts medium loam, by volume, 3.5 parts peat, by volume and 3.5 parts sand, by volume. To each cubic yard add 2 pounds of hoof and horn meal, 2 lbs superphosphate, 1 lb of sulfate of potash and 1 pound of chalk (calcium carbonate).” This mix was primarily for greenhouse use (Whitcomb, n.d.). The University of California and Cornell University are often credited with the initial development of soilless substrates for container production. This is a result of a greater understanding of plant nutritional requirements and greater control of plant pathogens in growing container crops, as well as the increased drainage and decreased transportation costs associated with the higher porosity of soilless media (Nelson, 2012). In the 1940’s and 50’s scientists at the University of California began researching container media in greater depth, and created the U.C. mix which consisted of 50% fine sand and 50% peat, with
various chemical additives listed for specific crops. Redwood sawdust was mentioned as a substitution option for part of the peat (Whitcomb, n.d.).

The profitability of crops grown in soilless substrates is higher than of those grown in soil due to improved physical and chemical properties of substrates and their low infestation rate with pathogens (Raviv et al., 2008). Soilless substrates consist of a variety of organic and inorganic components. Many different organic substrate components can be utilized in container production if the proper management is provided (Ingram, et al. 1993). The most popular of these components is peat. Within the last two decades the use of peat has been reevaluated since it is a non-renewable resource that plays a major role in atmospheric CO2 sequestration, influences groundwater quality, and as an essential habitat for plants and animals in those ecosystems. Chief among replacement materials for peat have been wood products, especially bark (Raviv and Lieth, 2008).

Pine Bark Structure

Bark is a generic term that includes the inner bark (living phloem) and rhytidome (outer bark, including cork cells) of a tree. Bark formation is initiated by the process of cell division at the cambium, which produces xylem on the woody (interior) side, and phloem, the primary bark tissue, on the exterior bark side. Phloem tissue contains phloem parenchyma, lignified fibers, companion cells, and sieve elements. The sieve elements are the main channel for downward movement of sap and nutrients from the leaves. The inner bark is the layer of physiologically active tissue adjacent to the cambium, and is generally thinner and lighter colored than the outer bark. As subsequent layers of phloem are produced each year, the outer layers become
compressed, and the sieve elements and surrounding structures collapse. This collapsed tissue ceases to take part in active physiological processes and is transformed into the relatively inert, dark colored outer bark that comprises the bulk of most tree barks. The structure of bark also contains a second cambial layer within the bark called the phellogen or cork cambium. Cork, or periderm, is produced by the cork cambium and contributes to the structure of the outer bark. (Harkin and Rowe, 1971).

Bark consists mainly of two contrasting groups of chemical substances: inert, materials including waxes (together with less-durable cellulose), cork, and lignin, and chemically active water-soluble extractives, mainly tannins and sugars (Aaron, 1976). Bark usually forms 6-16% of the volume of a tree, with 10% being the average value by weight or by volume of the harvested tree (Bunt, 1988; Aaron, 1991).

**History of Pine Bark as a Horticultural Substrate**

Often bark intended for horticultural use is a secondary product from lumber production and pulp and paper mill industries. It was formerly considered a waste product and disposed of by burning (Bunt, 1988), but there is considerable cost in burning bark at debarking stations, including the cost and cleaning of the incinerators, and the resulting air pollution. Some mills burn bark for energy for drying kilns, recovery boilers, and steam turbines; however, there are issues when mills do not burn as much bark as they produce and the bark accumulates in large quantities when left on site, where it can become a fire and environmental hazard (Van Schoor et al., 1990). Laws were enacted that prevented the producers of wastes from dumping or
burning bark residues (Gartner et al., 1973), therefore other uses for bark began to be investigated.

Research on PB for use as a horticultural substrate began in the US in the late 1960s. One of the first studies was conducted in the late 1960’s and published in 1971 and 1972 by Dr. Carl Whitcomb, in an effort to create a more porous potting mix for container grown *Carissa macrocarpa* Eckl. that were grown in the U.C. mix at a Florida nursery. During a Nursery Management and Production class that Dr. Whitcomb was teaching, it was mentioned that the U.C. manual had noted that redwood sawdust could be substituted for part of the peat. The class then discussed the use of ground PB instead of redwood sawdust. A student’s father worked at a pulp mill in Jacksonville, FL that burned large quantities of PB, and a truckload of the PB was obtained for a study. The coarse particles were screened out, and the remaining bark was used in several test mixes. The results of the study showed that plants grown in PB mixes were larger, and were the only plants in the study to display healthy, white root growth to the bottom of the container. While some plants displayed symptoms of nutrient deficiencies, indicating a need for more research, interest from nursery growers skyrocketed after the results of the studies were published (Whitcomb, n.d.). Concurrent PB research and growth trials were also conducted during this time at the University of Texas (1968-1971) involving shredded or ground PB (DeWerth, 1971.), and at the University of Georgia under Dr. Pokorny. Trials utilizing bark from softwood species in the Pacific Northwest were also being conducted in Oregon during this time (Bollen, 1969).

Research on utilizing softwood bark waste was also being carried out in Europe during the 1960s. The Norwegian Forest Research Institute began researching ways to utilize bark waste
beginning in the early 1960s (Solbraa, 1974). In England, there were several small-scale attempts to use bark for horticultural purposes. A firm of match manufacturers marketed shredded poplar bark, which was tested by the Parks Department of the former London County Council with unsatisfactory results, which was hypothesized to be the result of not applying nitrogen fertilizer. A timber merchant in Chester, England marketed bagged, coarse coniferous bark for horticultural use. Other uses for coniferous bark included composting and mulching trials by the Research Branch of the Silviculture (South) Division of the Forestry Commission; research involving extractives by the Commission’s Utilization Development Section, which resulted in the use of Sitka spruce tannins for upholstery; and the use of bark as fuel. In 1967, the Forestry Commission resumed work on bark with a concentrated effort on using it for horticultural purposes. By 1969 it was determined that a wide range of ornamental plants could be grown in a fertilized, bark-only mix, and the concurrent studies in the US further demonstrated the use of PB for both propagation and potting media. In 1970, the development in bark substrates in England was exhibited in England at the Chelsea Flower Show, where for the next four years a variety of plants and even fungi and forced bulbs were displayed. During this period, the first horticultural bark manufacturing company was established, which ultimately expanded to become the largest bark producer in England, and other bark operations soon followed (Allen, 1991).

The species of tree most commonly used for softwood bark substrates depends on the country and the region. In Europe, the principal countries that utilize softwood bark in substrates are France and Spain, with >1.2 Mm$^3$ and 0.5 Mm$^3$ of maritime pine (Pinus pinaster Aiton) used, respectively, in 2005 alone. In Australia, maritime and slash pine (Pinus elliottii Engelm.) are
primarily used, and bark from the Monterey pine (*Pinus radiata* D. Don) is the principal organic growing media component in New Zealand. (Bunt 1988) In the US, Douglas-fir (*Pseudotsuga menziesii* Mirb.) is the principal source of bark for substrates in the pacific northwest (Carlile, year), and loblolly pine (*Pinus taeda* L.) in the east. In the eastern US, PB often comprises as much as 75 to 100 percent (by volume) of container substrates (Wenliang et al., 2006). In the southeastern US, loblolly pine, shortleaf pine (*Pinus echinata* Mill.), slash pine, and long leaf pine (*Pinus palustris* Mill.) are the predominant species used for PB substrates (Bunt, 1988).

**Variability in Pine Bark Substrates**

Pine bark can be utilized as a fresh, aged, or composted product, with all three having the potential to provide excellent results as a substrate; however, PB is a naturally variable material, due to both biological and processing factors, and therefore not all bark supplies will be the same. Fresh PB refers to material that is ground, screened, and sold/used soon afterwards (Buamscha et al., 2007). Aging refers to the stockpiling and weathering of bark prior to its use, in open air piles established by the bark producer or commercial grower (Pokorny, 1979). No adjustments are made to pH or moisture content, and fertilizer is not added. Composting is the biological degradation of PB under carefully controlled conditions, where fertilizer, particularly N, is added, and pH, moisture, and aeration are carefully regulated. The advantages of composting are in the reduction of the carbon to nitrogen ratio, which reduces the potential for nitrogen immobilization, and in the build-up of heat which kills pathogenic organisms (Pokorny, 2002). However, composting is much more labor intensive to manage than aging, and unlike hardwood bark, mandatory composting of PB is not necessary (Pokorny, 1979). Aged bark is
most commonly used in the Southeastern U.S, but there is increasing interest in fresh bark because of its lower transportation costs and perception that it might be more consistent from batch to batch.

The storage and management of organic substrate components, including PB, can greatly affect the physical, chemical, hydrologic, and biological properties of a substrate (Handreck and Black, 2010). Bulk materials for substrates should ideally be consistent in properties from batch to batch (Carlile et al., year). Pokorny (1979) reported that of milled bark from six commercial nurseries, none of the barks tested were similar in particle size distribution or physical properties, which means that different cultural programs are needed to grow a uniform crop in each different medium.

Much of the variability in PB substrates are due to differences in environmental, mechanical, and processing/handling factors (Jackson, 2014). Environmental factors include the species of pine from which the bark is taken, the thickness of the bark at harvest, where the tree is harvested, and the soil type the trees were grown in. Mechanical factors include the method of bark removal, and hammer-milling of the bark. During the debarking process, large slabs and pieces of bark are removed from a log. Sawmills use ring debarkers or Rosserhead debarkers (Van Schoor et al., 1990), while most wood-panel and pulp and paper mills use drum debarkers. Ring, drum, and flail debarking machinery have different mechanisms of removing the bark, and some may rip and/or tear the bark more than others. These large pieces of bark are then delivered to the bark supplier, and processed by hammer-milling and screening to reduce the particle size to one which is suitable for plant growth (Pokorny, 1979). The hammer-milling of PB can be greatly influenced by the moisture content of the bark at the time of grinding, the thickness of the
bark, the screen size in the hammer-mill, and the horsepower, speed, torque, feeding consistency, hammer type and amount of wear, hammer tip speed, etc. Processing factors refer to the ways in which bark may be processed (hammer-milled, screened) and aged. Fresh bark may be aged without being hammer-milled and separated into different particle sizes first, while some suppliers may separate bark nuggets from fines and age only the fines for use in growing media. Aging times may vary between suppliers, or even for the same supplier at different times of the year. The resulting end-products are all sold to the consumer as “aged” PB.

The largest cost factor involved in using bark as a growing medium is transport (Van Schoor et al., 1990). Interest in fresh PB has increased because of its lighter weight, which reduces transportation costs, as well as potential for improved consistency (Fields et al., 2012), but little research has been done to investigate the differences between PB of various ages. Many of the differences are well known to growers and PB suppliers, but have not been documented or published in the literature. What has been published contains discrepancies about the effect aging has on PB substrates, and the effective use of fresh PB. Laiche (1974), however, reported reduced plant quality in fresh PB due to difficulty maintaining adequate moisture levels during the first three months after transplanting, and experienced difficulty in hydrating fresh PB. Self and Pounders (1974) have shown that plants can be grown successfully in fresh PB. Airhart et al. (1978) stated that both fresh and aged PB have been shown to become hydrophobic when moisture content falls below 34% (by weight). Self (1978) stated that aging of PB is unnecessary except for stockpiling inventory and improving wettability. Nichols (1981) found that seedlings grown in fresh *Pinus radiata* D. Don bark exhibited symptoms of toxicity when compared to seedlings grown in aged bark. Cobb and Keever (1984) observed equal or better plant growth in
fresh PB as compared to aged, with no difficulty hydrating the fresh PB or symptoms of phytotoxicity, and the plants grown in fresh bark had equal or higher tissue levels of N, phosphorus (P), calcium (Ca), and magnesium (Mg). Whitcomb and Appleton (1985) found that four different woody ornamental species grew similarly in fresh and one-year old aged PB with or without starter N. The Silviculture (South) Division of the Forestry Commission’s Research Branch in England observed that fresh PB severely inhibited the growth of container-grown plants as well as mushrooms, although this effect varied among PB from different species (Aaron, 1991). Svenson and Witte (1992) found no differences between fresh PB and PB that had been composted for four months in terms of plant available nutrients, shoot dry weight, pH, physical properties, and particle size. Harrelson (2004) reported reduced growth of *Cotoneaster dammeri* ‘Skogholm’ in fresh PB vs aged, due to lower container capacity and available water. Yap and Jackson (2012) showed that root growth of *Thuja* ‘Green Giant’ was not noticeably different in fresh vs. aged PB. In a study cited by Krewer and Ruter (2012), fresh PB had a water holding capacity of about 13% (by vol), while aged PB held 21% water (by vol). Bilderback et al. (2013) found that PB is generally considered to be non-phytotoxic and can be used without aging or composting but aging is preferred because fresh PB often has less than 20-30% fine particles, which results in reduced water holding capacity. Aged bark should theoretically hold more water due to an increase in the percentage of fines, increased uniformity of particle sizes, and decreased hydrophobicity due to the decomposition of wood and cambium (Bilderback, 2002; Bilderback, et al., 2005; Jackson, 2014).

Research on the efficient utilization of PB is often carried out with insufficient knowledge of the material and its variability (Van Schoor et al., 1990). Of over 80 articles that
the authors reviewed that involve PB substrates, we found 10 that list the specific age of the bark (Table 1), and of those 10 articles four of them are studies that look at differences in bark throughout the aging/composting process (Table 1.1). One of these papers is by Cunha-Queda et al. (2006) looking at the biochemical and microbial population changes in bark throughout a composting process. Two are by C.L. Davis et al. (1992 a,b) looking at microbial population numbers, physiology, and microbe speciation using plating techniques, SEM, and enzymatic methodology from bark sampled from a commercial composting operation. The most in depth article about documenting the changes during the composting process is by Guedes de Carvalho et al. (1991), who measured a number of physical, chemical, and biological parameters in a small-scale composting study. To our knowledge there is no commercial-scale research that has been conducted to investigate the physical, hydrologic, and chemical changes throughout the aging process of PB. Bark substrates are evaluated for readiness of use by growers based on physical factors (dark color, earthy odor, temperature decline of piles), and chemical tests (C/N ratio, pH) (Jorba and Trillas, 1983).
Literature Cited


Bilderback, T.E. Managing Container Substrates.


Whitcomb, C. N.D. Where did that originate? Ground pine bark in container growth media. 

Table 1. Scientific literature that state the specific age of bark materials used, and the parameters being investigated.

<table>
<thead>
<tr>
<th>Year</th>
<th>Author</th>
<th>Bark type/species</th>
<th>Aged or composted</th>
<th>Age of bark(s)</th>
<th>Properties measured</th>
</tr>
</thead>
<tbody>
<tr>
<td>2007</td>
<td>Cunha-Queda et al.</td>
<td>PB, EB, 60:40 PB/EB</td>
<td>Composted</td>
<td>0, 7, 65, 130, 180 d</td>
<td>Microbiological populations and biochemistry changes throughout composting process</td>
</tr>
<tr>
<td>1985</td>
<td>Whitcomb and Appleton</td>
<td>PB</td>
<td>Aged</td>
<td>Fresh, one-year old</td>
<td>Plant growth quality, starter nitrogen and activated charcoal effects</td>
</tr>
<tr>
<td>1974</td>
<td>Laiche</td>
<td>PB</td>
<td>Composted</td>
<td>Fresh (less than 2 weeks), 7 months, 10-15 years</td>
<td>Plant growth quality, fertilizer effects</td>
</tr>
<tr>
<td>1984</td>
<td>Cobb and Keever</td>
<td>PB (P. taeda, P. elliottii)</td>
<td>Aged</td>
<td>Fresh, one year</td>
<td>Plant growth and nutrient content, effect of supplemental N</td>
</tr>
<tr>
<td>1981</td>
<td>Nichols</td>
<td>PB (P. radiata)</td>
<td>Aged</td>
<td>6 weeks (dry) 6 weeks (3 dry, 3 wet) 6 weeks (wet)</td>
<td>Phytotoxicity, effects of age and moisture</td>
</tr>
<tr>
<td>1983a, b</td>
<td>Gutser et al.</td>
<td>70:30 SB/PB 85:15 SB/PB CB SB</td>
<td>Aged and composted</td>
<td>8 month aging, 3 month composting (70:30 SB/PB) 8 month aging, 2.5 month composting (85:15 SB/PB) 9 month composting (CB) 20-35 years (CB) 40 minutes, 5 weeks “post-composting” (SB) 1 week composting, 6 months storage (SB) 1 week composting, 4 weeks storage (SB)</td>
<td>Nitrogen dynamics, effect of composting method, duration, type, and tree – incubations and pot plant trials</td>
</tr>
<tr>
<td>2004</td>
<td>Harrelson et al.</td>
<td>PB</td>
<td>Aged</td>
<td>Fresh (one year)</td>
<td>Plant growth, fertilization rates</td>
</tr>
<tr>
<td>1992a, b</td>
<td>Davis et al.</td>
<td>PB (mixed P. patula, P. elliottii, P. taeda)</td>
<td>Aging followed by composting</td>
<td>Aged for 90 days, composted for 100 days; Aged for 2 weeks, composted for</td>
<td>Microbial populations and electron microscope physiology during composting</td>
</tr>
<tr>
<td>2007</td>
<td>Buamscha et al.</td>
<td>DFB</td>
<td>Fresh and aged</td>
<td>Fresh (48 h) Aged (approximately 7 mos.)</td>
<td>Nitrogen availability, physical properties, nutrient content</td>
</tr>
</tbody>
</table>

&Pine bark
& Eucalyptus bark
& Spruce bark
& Conifer bark
& Douglas fir bark
CHAPTER 2

Changes in Pine Bark During the Aging Process I: Physical Properties
Abstract.

Pine bark (PB) is one of the most commonly used horticultural substrates, and may be used as a fresh, aged, or composted product. Aged PB, which is the process of leaving PB inventories outdoors for a variable period of time with no moisture, pH, or nitrogen adjustments, is what is most commonly used; however, fresh PB may also be used successfully, but there is little research conducted that investigates the difference between bark over a monitored aging process. A twelve month study was initiated to better understand the effects of aging and pile management on PB substrates. The study was conducted at TH Blue, Inc., a PB supplier in Eagle Springs, North Carolina. Fresh PB was processed and the fines were placed in three pile replications of 191 cubic meters each. Piles were turned monthly. Beginning at time 0 and every 4 to 5 weeks, one week after turning, stratified subsamples were taken from each pile at three different heights (top, middle, and bottom) at horizontal depths within pile of 0.3 - 1.2 m to account for variations in pile depth and height. The stratified subsamples were mixed into one composite sample for each pile replication and physical properties of the bark were tested. Physical properties included pile volume, temperature, particle size distribution (PSD), total porosity (TP), container capacity (CC), air space (AS), bulk density (BD), white wood content (WW), color, and sand content. At the beginning of the aging process there was a rapid increase in pile temperature with a 4-month thermophilic phase ranging from 52.7 to 57.3 °C, after which temperatures decreased during months 5 and 6 and reflected changes in ambient conditions during the remainder of the study. Color changes followed a similar trend as the temperature data, with PB darkening during the first 6 months and appearing to stabilize with only slight changes of color throughout the last 6 months of the study. There was a decrease in PSD between
4.75 and 9.5 mm and an increase in PSD between 0.25 and 1.0 mm from months 0 through 12. There were no differences in TP after month 0 with values around 83-85% from months 2 through 12, while CC increased from 34.5 to 51.2 %, AS decreased from 42.5 to 32.2%, and BD increased from .18 to .20 g/cm³, further illustrating the shift to greater amounts of fine particles over time, resulting in smaller pore sizes within the same pore volume. White wood content decreased from 3.4 to 2.2% in weight and 6.4 to 3.8% by volume, and sand content increased from 2.8 to 20.2% by weight and 0.43 to 3.19% by volume. Pile volume decreased from 74.7 m³ to 51.9 m³ while pile BD increased from 365 to 537 kg/m³. The results from these tests indicate that the bark stabilized around month 6.
Introduction

Bark, especially softwood or pine bark (PB), is one of the most commonly used substrate components for both greenhouse and nursery crop production. It is obtained as a byproduct of the timber industry when it is stripped off logs after harvest, and is then hammer milled and screened to obtain a desirable particle size for plant growth. The advantages of using PB as a horticultural substrate are numerous: the fact that it is 1) a waste product; 2) a renewable resource; 3) it is less expensive than peat moss; 4) processing PB by hammer-milling and screening can allow for reproducibility and consistency; 5) PB substrates can have good drainage and aeration; and 6) PB has been shown to contain most minor elements essential to plant growth (Pokorny, 1979; Van Schoor et al., 1990). It also decomposes at a slower rate than hardwood bark and other composts/green waste, which can help maintain a stable air-water relationship in the substrate and reduce the risk of poor drainage and overwatering (Pokorny, 1979).

Pine bark is used as a composted, fresh, or aged product (Bilderback, 2002); however, some disagreement exists as to whether fresh PB can be used as successfully as aged or composted PB (Bunt, 1988). Composted PB refers to the end product of a carefully monitored process in which moisture content and pH are adjusted, piles are turned frequently, and nitrogen (N) is added to stimulate microbial populations and reduce the cellulose content of fine PB particles and fiber to humus through decomposition (Self, 1978). Fresh PB refers to material that is ground, screened, and sold/used soon afterwards (Buamscha et al., 2007). Aging refers to the stockpiling and weathering of bark prior to its use with no adjustments made to pH or moisture content, and no nitrogen additions (Pokorny, 1979). During the aging process piles should be
turned to provide aeration and consistency, but the frequency of turning may vary among suppliers. Aged PB may be referred to as “composted,” but in the strictest sense, unless it is amended with a nitrogen source, moistened, and turned on a regular basis, it may not truly compost (Bilderback, N.D.). Aging times can vary between suppliers, or even for the same supplier, depending on inventory and the time of the year (Jackson, 2014). The composting process, while producing a high-quality end product, is used less frequently due to the fact that it is a more labor and resource intensive process. Aged PB is most commonly used in container production; however, there is interest and demand for fresh PB due to its lighter weight (resulting in lower freight costs), quick turnaround time for the supplier, and perception by some growers that it is a more consistent product than aged PB.

The storage and management of PB, such as duration of aging, pre-processing conditions, and manufacturing methods can have a great effect on altering the physical properties of a container mix (Bilderback, 2002). The desirable physical characteristics of container media can be described in terms of their bulk density (BD), total porosity (TP), container capacity (CC), and air space (AS; Bunt, 1988). Bulk density of a substrate is described as its dry mass per unit of volume (Raviv and Lieth, 2008). TP is defined as the combined volume of the aqueous and gaseous phases of the substrate, not occupied by mineral or organic particles (Bunt, 1988), and is related to the shape, size, and arrangement of substrate particles. In many cases, TP and BD are inversely related (Raviv and Lieth, 2008). AS is the volumetric percentage of the substrate that contains air at the end of free (gravitational) drainage (Raviv and Lieth, 2008). Container capacity (CC) is defined as the total amount of water present after the medium in a container has been saturated and allowed to drain, and is conceptually analogous to field capacity in mineral
soils; however, the amount of water retained in a horticultural substrate will be greater than that present in a mineral soil at field capacity (Bunt, 1988). Fresh PB has been shown to have higher AS, lower container capacity CC, and lower available water content when compared to aged bark, which could require changes in irrigation management (Bilderback et al., 2005). Container weight is a function of a substrate’s BD and CC. Heavier containers are less likely to blow over in a nursery, but their shipping costs will be higher. In addition, some insecticide applications use rates based on substrate BD (Buamscha et al., 2007)

Particle size distribution (PSD) affects, and is used to make inferences on, the physical properties of a substrate, including air and water relationships (Bilderback et al., 2005; Tilt et al., 1987), because of its influence on surface area to volume ratios and pore sizes (Bollen, 1969). The ratio of small, medium, and large particles determines the percent pore space at CC and moisture retention of the substrate. Large particles should be small enough to avoid irrigation issues, but large enough to ensure a high porosity, and an adequate number of fines are needed to raise or maintain adequate moisture holding properties (Hoitink and Poole, 1980). Fine particles less than 0.5 mm are generally considered responsible for moisture retention in containers (Bilderback et al., 2013). Van Schoor et al. (1990) found that the percentage of fine particles (<0.5mm) in PB substrates is highly linearly correlated to AS, with AS decreasing with higher proportions of fine particles. An adequate proportion of fine particles is also needed to increase cation exchange capacity (CEC); Altland et al. (2014) found that CEC of PB substrates increased with decreasing particle size, and Daniels and Wright (1988) reported a drop in CEC among PB particles greater than 2.38 mm.
As PB ages, there will be an increase in smaller particles over time due to mechanical breakdown from turning and microbial activity. During this degradation process, the mass and volume of the material decreases due to decomposition by oxidation of organic carbon to CO₂, and the breakdown of structural organic components to form a matrix of smaller particles with a greater BD (Breitenbeck et al., 2004). Breitenbeck (2004) found that volume reduction of PB piles during a composting process was due as much, if not more, to reductions in particle size and pore volume as to mass loss from microbial activity. This increase in BD over the course of aging may result in higher freight costs when compared to fresh PB.

During the debarking process pine wood (xylem) may accompany the bark, and this wood is referred to as white wood (WW). WW content in PB substrates is variable, and influenced by many factors including tree species, time of harvest, bark thickness at time of removal, variations in processing (including aging of piles), and debarking method (Jackson, 2014; Solbraa 1974). Ring and drum debarkers remove little wood from logs and generally produce bark with a wood content of less than 10% (Hoitink and Poole, 1980). Fresh PB tends to have more white wood than aged PB because it has had less time to break down; however, even after aging much of the white wood may remain, but it is stained and more difficult to see (Jackson, 2014). The method of debarking also affects white wood content. There are no official standards in place for white wood content in PB substrates; however, a general suggestion is that 10-15% WW is acceptable (Jackson, 2014; Mulch and Soil Council, 2007). In the past, growers preferred PB with little to no white wood; however recent research has shown that white wood is not detrimental to the quality of PB and the resulting crop growth (Owen, 2014). Jackson (2014), in a study investigating differences in the properties of PB substrates from different suppliers,
found that physical properties did not change significantly after the removal of WW from bark. White wood content of 10-15% in a bark substrate may not affect physical properties if the particle size distribution of WW is like that of the bark, but as little as 5% WW content with a large particle size (length or diameter) can greatly change the physical properties of PB substrates (Jackson, 2014). Fields et al. (2012) found that WW content of aged PB was less than half of that of fresh PB from the same supplier, but no significant change was seen in any physical property after the removal of WW from any of the fresh or aged PB samples that were tested. The BD of WW (approximately 0.14 g/cm$^3$) is lower than that of bark, which indicates that the addition of WW may lower the weight of PB without altering other physical properties (Fields et al., 2012). In terms of pile management, it has been reported that wood and cambium in fresh PB may stimulate rapid decomposition, moisture loss, and heat in inventories (Bilderback, N.D).

Temperature profiles during the aging process are characterized by 1) a rapid initial increase in temperature, 2) a sustained high-temperature period (thermophilic phase), and 3) a decline to near-ambient temperatures. The initial rise and high temperature period is caused by exothermic reactions associated with the microbial degradation of the readily biodegradable organic matter in PB, and the duration of the subsequent thermophilic phase is determined by the cellulose content (Hoitink, 1980). The decrease in temperature after the thermophilic phase ideally occurs due to a decrease in microbial activity as the easily biodegradable materials are consumed and more resistant compounds such as cellulose and lignin remain (Wichuk and McCartney, 2010). Therefore, it has been suggested that a decline to near-ambient temperatures may indicate that the PB is approaching a stable/mature state, provided that reheating does not
occur upon turning the pile. However, pile temperature may be affected by ambient conditions (particularly in cold climates), pile size, extreme temperatures during the thermophilic phase, low AS, oxygen depletion, drying, and excessively high moisture (Wichuk and McCartney, 2010). Ambient conditions such as low temperatures and high winds can lead to heat loss and premature pile cooling, and smaller piles tend to heat up and cool more rapidly than large piles under similar environmental conditions, since their higher surface area to volume ratio provides less thermal insulation. As a result, in certain circumstances a small pile that has cooled may not be mature, while a large pile that is still warm may actually be stable (Wichuck and McCartney, 2010). There is also a strong temperature gradient within a pile. Temperatures will be lower at the bottom and higher at the top, creating a ‘flue’ effect which draws air into the bottom of the pile (Van Schoor et al., 1990).

The heat created during the thermophilic phase of the decomposition process causes the color of the stockpiled PB to become darker (Self, 1978). Initially the PB may be a lighter brown or reddish brown, and as the biological activity declines over time the compost will become a dark brown or black as the rate of change in biological activity changes imperceptibly (Thompson, 2002). Color is a sensory parameter that is frequently used anecdotally in the industry when discussing age and usability of PB inventories or substrates. The measurement of color changes has been suggested as a parameter to determine stability in composting systems (Thompson, 2002; Wichuk and McCartney, 2010), but has not been quantified in the literature for PB.

Pine bark substrates may contain a significant amount of sand from turning and other handling operations. This can be beneficial in maintaining long term integrity of the substrate,
through improved water holding capacity and reduced water infiltration rates are, which promote better hydration (Krewer and Ruter, 2012). Brown and Pokorny (1975) found that amending PB with sand decreased percolation rate and increased bulk density. Niemiera et al. (1994) reported that PB substrates with 9:1 and 5:1 bark/sand ratios had decreased drainage, TP, AS, and unavailable water at 1.5 kPa tension; and increased AW, BD, and solids content. Greater concentrations of small pores in the 5 PB: 1 sand substrate allowed for less percolation and pore dispersion of solution than 100% PB, which demonstrated greater channeling and less retention of water/nutrient solutions (Niemiera et al., 1994). Sand also increases the proportion (by weight) of smaller particle size fractions (Brown and Pokorny, 1975; Niemiera et al., 1994). This can increase available water due to the addition of sand created micropores that hold water less strongly than the surface and intraparticle water adsorption sites of PB particles.

The objective of this research was to investigate and quantify the changes in the physical properties of PB during 12 months of managed aging. While both aged and fresh PB can be used successfully, little research has been conducted to investigate the differences in physical properties between fresh and aged PB, or PB of specific ages. Additionally, the actual age of PB used for research in the literature is usually not reported and is either listed as “fresh” or “aged” (if age is mentioned at all), making it hard to extrapolate anything more than general trends. PB that has been aged for three months may be very different from bark that has been aged for twelve months, and both may be reported in the literature as aged, or sold to a grower as aged, but with different results. This is a factor that leads to high variability in PB substrates, which can be a concern for growers and the substrate industry. Another concern is substrate transportation costs, which will determine the feasibility and success of any substrate or substrate
component. As a result, there has been a greater interest in developing and using locating and
using substrates from local materials, such as loblolly or longleaf PB in the southeast, to reduce
transportation costs (Jackson and Fonteno, 2014) and/or potentially grow and create on-site.
Assessing the properties of PB of different ages will help to understand variability in PB
substrates, and aid in the further development and optimal use of these local and sustainable
materials to grow horticultural crops.

Materials and Methods

Sampling. The study was implemented on 20 Aug. 2015 at a commercial PB supplier (TH Blue
Inc, Eagle Springs, NC). Longleaf PB (*Pinus palustris* Mill.; Appendix A.A) from two lumber
companies, Jordan Lumber Company (Mt. Gilead, NC) and Troy Lumber Company (Troy, NC)
was obtained the week of 17 Aug. 2015 and 15 truckloads were unloaded on the research site for
further processing (Fig. 2.1A). The bark was passed through a hammer-mill (Fig. 2.1B;
Appendix AB) and screened (Powerscreen Chieftan, Duncannon, UK) into three fractions:
nuggets (Appendix AC), mini nuggets (Appendix AD), and fines (Fig. 2.1C, Appendix AE).
Fines were passed through a 1.27 cm screen. Nuggets and mini-nuggets are sold as mulch, and
fines are stockpiled and aged for eventual use as a nursery substrate. The < 1.27 cm fraction was
placed in three piles of approximately 191 m³ each, with dimensions of approximately 16.8 m x
10.1 m x 3.2 m (Fig. 2.1D). These were treated as replications. Piles were turned every four to
six weeks (Table 2.1) using a front-end loader. The piles were sampled at the time of installation
and approximately one week after every turning, depending on weather and turning constraints,
for a period of 12 months (Table 2.1). Sampling and sample storage procedures were based on
the methods outlined in the US Composting Council’s Test Methods for the Examination of Composting and Compost (Thompson, 2002). At each sample date, stratified subsamples were taken from different heights (top, middle, and bottom) and horizontal depths (0.3, 0.6, 0.9, and 1.2 m) within each pile. Three locations within each height were randomly selected for sampling by conceptually treating the pile as a three-dimensional grid (Fig. 2.2A), equaling 9 sampling locations per pile. At each of the 9 sampling locations, subsamples were taken by digging a hole to a depth of 1.2 m, and removing 3 L of bark from the surface every 0.3 m (five subsamples; Fig. 2.2B). This sampling procedure accounts for variation within a pile and reduces possible errors due to stratification of constituents and conditions within the piles (Breitenbeck, 2004). All subsamples per sampling location were composited on-site by placing them into a 4-mil poly bag (61 cm x 76.2 cm; Consolidated Plastics, Stow OH) and sealing with a zip-tie to prevent moisture loss. Samples were transported to the Horticultural Substrates Laboratory (North Carolina State University, Raleigh, NC) for storage and subsequent analysis. For each pile, approximately 8 L of PB from each of the 9 sampling locations were mixed into a composite sample for testing. When not being used for testing, samples were stored indoors in sealed, 4 mil poly bags at 4°C to minimize any moisture and microbial changes during storage.

Dimensions and volume. The dimensions of each pile were measured manually at each sample date using a 60 m open reel fiberglass tape measure (Apex Tool Group LLC., Sparks, MD). Measurements taken included the length of the bottom of the pile (L), length of the top of the pile (L_{top}), width (W), height (H), and circumference. The volume of each pile was calculated by adding the volume of a triangular prism and the volume of a cone, where:

\[
\text{diameter of cone (CD)} = L - L_{top} + W / 2
\]
volume of cone = \(0.262 \times CD \times CD \times H\)

volume of a triangular prism = \(H \times W \times L_{\text{top}} / 2\)

**Drone imaging.** Drone technology is increasingly being utilized for three-dimensional mapping and inventory control of piles, and has the potential to quickly, efficiently, and cost effectively measure pile volume. To compare this technology to the method used above for measuring pile volume, the research site was mapped on 9 Sept. 2016 using a Phantom 4 drone (DJI, Shenzhen, China) equipped with 3D mapping software (DroneDeploy, San Francisco, CA). To create the map, multiple overlapping photos (80 to 90% overlap) of the site were taken as the drone flew along an autonomous programmed flight path called a waypoint. To create a waypoint, the area to be flown was outlined on a base layer map, and the software generated a flight plan. The entire mapping process was automated: following a safety check, the drone automatically took off and flew along the automated flight path and captured images, and then landed back at the starting point. 171 photos were taken from the flight and were uploaded to DroneDeploy to be processed. DroneDeploy’s cloud-based processing stitched the images together to create a model of the site. To determine the volume of the piles, the area we wished to measure was located in the “2D map” and under the “annotations and measurement” section, “volume” was selected, in which each pile was outlined, and the volume of the outlined area was calculated (Appendix B). This was done for each pile, and the resulting values were compared with the manual measurements for month 12.

**Temperature.** Pile temperatures were measured on-site at each sample date. Measurements were taken simultaneously at three different heights (top, middle, and bottom) at horizontal depths of 0.3, 0.6, 0.9, and 1.2 m using three 1.2 m compost monitoring temperature probes (Green
Mountain Technologies, Bainbridge Island, WA.). Three probes were inserted into the three different pile heights, starting at a depth of 0.3 m, and were manually pushed into the pile in 0.3 m increments allowed to stabilize for 5 to 15 min between readings (depending on the degree of change in temperature readings) at each depth until the full length of each probe (1.2 m) was inserted into the pile. Temperature data were analyzed as separate readings at each height and depth, as well as averaged across all pile heights and depths to give an average pile temperature per sample date.

Thermal imaging. The temperature of the pile surface, as well as PB temperature during pile turning on 13 Nov. 2015, were measured using a thermal imaging camera (FLIR C2, FLIR Systems, Wilsonville, OR). Surface temperature images were taken by placing the thermal imaging camera on a tripod at a distance of 19 m. Thermal imaging was used to measure pile temperature one day after installation (21 Aug. 2015) in the middle height zone of the piles at a depth of 1.2 m by digging holes into the piles and photographing the inside of the hole (Appendix CB). Periodically, thermal images were also taken of sampling sites at various depths to compare the camera readings with temperature probe data.

Color. Within 48 h after each sampling time, the color of samples was analyzed using a Munsell Soil Color Book (Munsell Color, Grand Rapids, MI) as described in the US Composting Council’s Test Methods for the Examination of Composting and Compost (Thompson, 2002). Munsell Soil Color consists of approximately 250 colored chips with corresponding apertures arranged on hue cards. Three coordinates - hue, value, and chroma - describe the possible colors (Fig. 3). Hue refers to the dominant spectral or rainbow color, with red (R), yellow (Y), yellow-red (YR), green-yellow (GY), gray (Gley), and white being the dominant hues in communicating
soil color. The hue cards include 10R, 10YR, 2.5Y, 2.5YR, 5Y, 5YR, 7.5YR, 10Y–5GY, Gley 1 and 2, 5R, 7.5R, and white. Value indicates the degree of lightness or darkness of a color on a gray scale ranging from black to white. In the Munsell Soil Color Book, value ranges from 0 (pure black) to 10 (pure white). Chroma is the purity or saturation of a color. Chroma ranges from 0 (neutral colors) to 8 (most vivid color) in the Munsell system. Samples are read as hue value/chroma, e.g. 5YR 3/2. Three 500 mL replications of PB from each pile replication per age (9 replications per sample date) were spread out on a small tray and measured indoors in bright, ambient cool white fluorescent lighting, at the moisture content of the material at the time of sampling, which was between 50-60%. It is important that samples are measured at the MC at which the color does not change with additional water additions. Color was analyzed by placing the sample directly behind the apertures on the hue card separating the closest matching color chips. In the case of color differences within samples, the dominant colors were used for measurement (Fig. 2.3).

*Particle size distribution.* From each composite pile sample per sample date, approximately 3 L of PB was spread out to a depth of 2 cm on a drying pan and placed in a forced air dryer for 48 h at 105 °C. Particle size distribution was determined by passing three 100 g replications of the dry PB through a column of twelve sieves (W.S. Tyler, Mentor, OH) with screen sizes of 12.5, 9.5, 6.3, 4.75, 2.8, 2.0, 1.4, 1.0, 0.71, 0.5, 0.25, and 0.106 mm, with a pan at the bottom to collect the particles that passed through the 0.106 mm sieve. The column of sieves was placed in a Ro-Tap Shaker (W.S. Tyler, model B; Mentor, OH) and shaken for five min. The sieves were separated and the contents of each sieve were weighed separately. Particle size is expressed as a percentage
of the total weight of the 100 g sample. Three replications were analyzed from each pile replication, equaling 9 total replications per sample date.

Porometer. Physical properties including AS, CC, TP and BD were determined at each sample date using the North Carolina State University Porometer method (Fonteno and Harden, 2010). Samples were tested at a moisture content of 60% as recommended by Fonteno and Harden (2010). Three replications were analyzed from each composite pile replication, equaling 9 total replications per sample date. Bulk density was also calculated at sample moisture content using the pack weight and core volume to give a representation of BD “as-is” on-site, to provide insight into freight costs for bark of different ages, and to investigate the relationship between pile BD and volume over time. While this is not the standard procedure to measure BD, which is measured on a dry weight basis, it may give us insights into transportation costs associated with PB of different ages.

White wood content. Approximately 3 L of PB from each composite pile sample from months 0, 3, 6, 9, and 12 were spread out to a depth of 2 cm on a drying pan and placed in a forced air dryer for 48 h. at 105°C. The dried bark was separated into three 200g replications per pile replication (9 replications total per sample age), and WW of 0.5 mm or greater in length were manually separated from the bark. WW and bark were weighed separately, and WW content is expressed as a percentage of the total weight of the 200 g sample. The percent volume was also calculated by placing the WW and bark in 50 and 1000 mL beakers, respectively, and recording the volume of each.

Sand content. Sand content of PB samples was determined based on a modified procedure of Robinson (1927). Approximately 3 L of PB from a composite sample of material from each pile
replication from months 0, 3, 6, 9, and 12 were spread out to a depth of 2 cm on a drying pan and placed in a forced air dryer for 48 h. at 105 °C. 100 g of dried PB was placed in a 2 L plastic beaker and the volume in mL was recorded. 1.5 L of tap water was added to the beaker, and the sample was agitated vigorously by hand using a glass stir rod for 2 min. Large PB particles floating on the surface of the slurry were removed, and the sample was stirred again and the excess water and floating particles were decanted, leaving behind a mixture of fine PB particles and sand. This mixture was rinsed with tap water and stirred to remove as much of the excess PB particles as possible, and the water and PB particles were decanted for a second time. The remaining mixture of sand and fine PB was transferred to a 250 mL plastic beaker and placed in a forced air dryer for 24 h. at 105°C. Once dry, 50 mL of 30% hydrogen peroxide was added, a glass petri dish was placed on top of the beaker to prevent evaporation of the hydrogen peroxide, and it was placed in a hot water bath at 90 °C and digested until no more bubbles evolved from the mixture. At this point, the samples were removed from the hot water bath, the hydrogen peroxide and any undigested PB particles were decanted. The remaining sand was rinsed with water and decanted again, and was placed in a forced air dryer for 24 h. at 105 °C. The dried sand was then weighed, and poured into a graduated cylinder to measure volume. Sand content is expressed as a percentage of the total weight and volume of the 100 g PB/sand sample. Three replications were measured per age.

_Climatological data._ Climatological data over the duration of the experiment were obtained from the Sandhills Research Station (Jackson Springs, NC), approximately ten miles from the research site. Daily averages for air temperature, relative humidity, wind speed, wind direction,
precipitation, and solar radiation data were retrieved online from the State Climate Office of North Carolina website.

Statistical analysis. Data were checked for normality and linearity assumptions, and analyzed using the mixed ANOVA in SAS. Treatment means were separated using Tukey’s honestly significant differences (HSD) test at p < 0.05 significance level. There was no significant pile x age treatment interaction for any of the measured variables; therefore, data from all piles at each sample date were combined for analysis.

Results and Discussion

Sampling. Except for the first turning and subsequent second sample date, piles were turned and sampled each month over the course of the study. For the purposes of this discussion, samples will be referred to by the month in which they are collected, even though PB collected in the second month after the initiation of the study was approximately 6 weeks old (Table 2.1).

Pile temperature. There was an initial increase in pile temperature from ambient temperatures at installation, marking a 4 month long thermophilic phase of the decomposition process. During months 2 through 4, piles maintained average temperatures between 52.7–57.3 °C (Fig. 2.4) before decreasing at month 5, which is consistent with the length of the thermophilic phase described by Van Schoor et al. (1990) for a PB aging/composting process. Thermal imaging data showed an average pile temperature of 58.5°C (Appendix CB), and while this data cannot be directly compared to the other temperature data from this study, it is useful in illustrating how rapidly the thermophilic phase of the aging process begins. Average pile temperatures decreased during months 5 and 6 as microbial activity and ambient temperatures decreased, then gradually
increased throughout the remainder of the study, presumably in response to warmer ambient temperatures throughout the spring and summer months (Fig. 2.4). The stratified temperature data (Fig. 2.5) illustrate gradients based on height and depth, consistent with the description of pile temperature of Van Schoor et al. (1990), with temperature readings taken at depths of 1.2m being the warmest for any of the depth measurements in the three heights, and temperatures taken at the top of the pile generally being warmer than those taken at the middle of the pile, with both top and middle temperatures being warmer than those at the bottom (Fig. 2.5). Temperature variations among sampling depths for each height were the greatest during the first six months aging, and decreased throughout the remainder of the study.

_Bark Color_. The color of the bark darkened over time (Fig. 2.6), beginning as a reddish brown with a Munsell color value of 2.5YR 3/4 and getting progressively darker through month 5, eventually stabilizing to a dark brown with a consistent Munsell color value of 10YR 2/1 for months 6 through 12. This “darkening” is illustrated in the decreasing value and chroma numbers of the Munsell color readings. Color changes were easily determined during the first 6 months using the Munsell Soil Color system. During months 6 through 12 the bark continued to slightly darken each month, which could be noted by careful observation, but these subtle changes could not be differentiated using the Munsell Soil Color system (Fig. 2.6). These color measurements demonstrate the same trend as the temperature data, with readings stabilizing after 6 months of aging, and are consistent with the darkening of PB due to heat created during the thermophilic phase of the aging process, as described by Self (1978).

_Particle size distribution_. There was a general decrease in percentages of extra-large (>6.3 mm) and large particles (2.0 mm to 6.3 mm), and an increase of medium (0.5 mm to 2.0 mm) and fine
particles (<0.5 mm) over the course of the study (Fig. 2.7), which is consistent with the results of Fields (2012) who reported the same PSD differences between fresh and aged PB from different suppliers. There was a decrease in particle size fractions between 4.75 mm and 9.5 mm, and an increase in particle size fractions between 0.25 mm and 1.0 mm over 12 months of aging (Table 3, Fig. 2.8). There were no changes between ages in particles 12.5 mm or greater, between 1.4 mm to 2.8 mm, or 0.106 mm or less, or (Table 2.3). Cobb and Keever (1984) reported similar trends in PSD for fresh and aged bark, with a 16.3% decrease between fresh and aged bark for particles 4.76 mm or greater, a 0.02% decrease in particles between 2.38 to 4.76 mm, a 0.05% decrease in particles between 2.0 to 2.38 mm, a 0.04% increase in particles 1.0 to 2.0 mm, a 0.03% increase for 0.84 to 1.0 mm, a 1.8% increase for 0.60 to 0.84 mm, a 2.7% increase for 0.42 mm to 0.60 mm, and an 11.8% increase for the pan (less than 0.42 mm). Bilderback (2002) reported PB PSD values of 9.0% (fresh) and 13.3% (aged) for 6.3 mm and greater, 47.3% (fresh) and 36.2% (aged) for 2.0 – 6.3 mm, 25.7% (fresh) and 23.2% (aged) for 0.7 mm, 6.8% (fresh) and 7.3% (aged) for 0.50 mm, 7.6% (fresh) and 10% (aged) for 0.25 mm, 2.8% (fresh) and 7.2% (aged) for 0.106 mm, and 0.8% (fresh) and 2.8% (aged) for the pan (less than 0.106 mm).

PB from months 8 through 12 of this study provide PSD distributions closest to Pokorný’s recommendation of 70-80% coarse particles (0.6 to 9.5mm) and 20-30% fine particles (less than 0.6mm; Pokorný, 1979).

Physical properties. Over the course of the aging process, CC and BD increased while AS decreased (Fig. 2.9). TP increased from 77% to 83.5% between months 0 and 2, but there were no differences throughout the remainder of the study, with 12 month old PB having a TP value of 83.4%. From month 0 to 6, AS decreased while CC increased. CC, AS, and TP did not change
during months 6 to 12. The changes in CC, AS, and BD correspond to the increase in the percentage of smaller particles as the bark aged, resulting in a shift to smaller pore sizes in the same total pore volume. Container capacity increased from 36% at month 0 to 51% at month 12, but did not change during months 6 to 12 (Fig. 2.9), while AS decreased from 43% at month 0 to 32% at month 12. BD increased from 0.18 g/cm³ at month 0 to 0.20 g/cm³ at month 12, changing over months 0 through 10 and stabilizing after that. These findings are consistent with what has been reported in the literature for fresh and aged PB. Fields (2012) showed that there were no differences in TP between fresh and aged PB from two different suppliers (80% and 81.9% for fresh and aged, respectively from supplier 1; 82.5% and 82.8% for fresh and aged, respectively, for supplier 2), with an increase in CC (48.5% and 62.1% for fresh and aged, respectively, for supplier 1; and 33.8% and 49.6% for fresh and aged, respectively, for supplier 2) and a decrease in AS (33.8% and 17.9% for fresh and aged, respectively, for supplier 1; 48.7% and 33.3% for fresh and aged, respectively, for supplier 2) and a decrease in AS (33.8% and 17.9% for fresh and aged, respectively, for supplier 1; 48.7% and 33.3% for fresh and aged, respectively, for source 2). BD showed no differences, with 0.19 g/cm³ for fresh and aged for source 1, and 0.18 g/cm³ fresh 0.19 g/cm³ aged for source 2 (Fields et al., 2012). Bilderback (N.D.) reported values of 44% CC, 42% AS, 86% TP, and 0.19 g/cm³ BD for fresh PB; and 51% CC, 31% AS, 82% TP, and 0.19 g/cm³ BD for aged PB. Harrelson (2004) reported 49% CC, 39.3% AS, 88.3% TP, and 0.17 g/cm³ BD for fresh PB; and 61.1% CC, 25.2% AS, 87.3% TP, and 0.19 g/cm³ BD for aged PB. Cobb and Keever (1984) reported BD values of 0.30 g/cm³ for fresh PB, and 0.31 g/cm³ for aged, which are higher than our results and those of the studies cited above.

The Southern Nursery Association provides suggested ranges for the physical properties of horticultural substrates for nursery production (Bilderback et al., 2013). The recommended
ranges for PB are 50 to 85% for TP, 10 to 30% for AS, 45 to 65% for CC, and 0.19 to 0.24 g/cm$^3$ for BD. Average TP throughout all 12 sample dates ranged from 77% to 85.8%, which is within these guidelines. Air space values over 12 months ranged from 45.4% to 31.7%, all of which are higher than the recommended guidelines, but bark from 6 to 12 months had AS values closest to the guidelines. CC values ranged from 34.5% to 52.6%, with PB from 5 to 12 months having CC values within the suggested BMP range. BD ranged from 0.17 g/cm$^3$ to 0.21 g/cm$^3$ with bark from 6 to 12 months having BD values within recommended guidelines.

_Pile volume and bulk density._ Average BD as calculated for the PB piles on-site increased over 12 months from 365 kg/m$^3$ at month 0 to 537 kg/m$^3$ at month 12 (Fig. 2.10). Oriol et al. (1978) and Harkin and Rowe (1976) reported volume weights of approximately 200-250 kg/m$^3$ and 480.55 kg/m$^3$ respectively for fresh PB. Average pile volume decreased from approximately 74.7 m$^3$ at month 0 to 51.9 m$^3$ at month 12 (Fig. 2.10). This translates to a 30.5% decrease in volume over time with a 47% increase in BD. Van Schoor (1990) reported a decrease in volume up to 20% for PB in a composting system due to the mineralization of carbon by microorganisms to CO$_2$. Other possible factors that could have contributed to the volume loss in our system on a small scale were monthly turning, and the amount of material taken from each pile during sampling.

_White wood content._ The WW content of PB sampled 0, 3, 6, 9, and 12 months was all below the suggested maximum content of 10-15% (by vol), with 6.3% by volume and 3.48% by weight at month 0, to 3.8% by volume and 2.18% by weight at month 12 (Table 2.4). White wood content decreased after six months, which is consistent with the suggested aging time of six to eight months to allow for the degradation of wood within PB piles, as described by Bilderback et al.
Fields et al. (2012) also found decreases in WW content between fresh and aged PB from two different suppliers, with 29.25% WW (fresh) to 13.4% WW (aged) for supplier 1, and 11.4% WW (fresh) to 4.4% WW (aged) for supplier 2. These percentages are all higher than what was found in the present study, and could be due to differences in initial processing.

**Sand content.** Sand content increased from 2.8% by weight and 0.4% by volume at month 0 to 20.1% by weight and 3.2% by volume at month 12 (Table 2.5). Bulk density for bark at 0, 3, 6, 9, and 12 months were 0.18, 0.17, 0.19, 0.21, and 0.20 g/cm$^3$, respectively. If the weight of the sand is subtracted from the bark, the BD values are 0.17, 0.16, 0.17, 0.18, and 0.16 g/cm$^3$ for months 0, 3, 6, 9, and 12, respectively, which may indicate that much of the increase in bulk density over the aging process in this experiment was due to an increase in sand content.

These data suggest that PB suppliers with inventory piles on sandy surfaces may not need to add additional sand to mixes to increase water holding capacity.

**Summary**

The results from this study illustrate the changes in physical properties that occur in a monitored aging process over the course of a year. These data, while consistent with previously reported data on fresh and aged PB, provide information for PB at specific ages of a year-long aging process. Bark managed under these conditions stabilized around 6 months of aging, as shown by decreases in average and stratified pile temperatures, a decrease in pile volume, color darkening, and decreased WW content. Concomitantly, there was a shift to higher percentages of fine particles within the same total pore volume, an increase in CC and subsequent decrease in AS, and an increase in BD. It should be noted that processing influences results, and that bark
handled and treated in other ways may yield different end products that can be used successfully, but may not be identical to what is presented here.

**Literature Cited**


Bilderback, T. Managing container substrates. n.d.


www.mulchandsoilcouncil.org


Table 2.1. Turning, sampling dates, and sample age for a pine bark piles over the course of 12 months in 2015 and 2016.

<table>
<thead>
<tr>
<th>Turning date</th>
<th>Sampling date</th>
<th>Sample age (Days after initiation)</th>
<th>Sample age (Months after initiation)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Installation</td>
<td>21 Aug. 2015</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>2 Oct. 2015</td>
<td>10 Oct. 2015</td>
<td>52</td>
<td>2</td>
</tr>
<tr>
<td>5 Nov. 2015</td>
<td>13 Nov. 2015</td>
<td>84</td>
<td>3</td>
</tr>
<tr>
<td>2 Dec. 2015</td>
<td>11 Dec. 2015</td>
<td>112</td>
<td>4</td>
</tr>
<tr>
<td>8 Apr. 2016</td>
<td>17 Apr. 2016</td>
<td>243</td>
<td>8</td>
</tr>
<tr>
<td>11 May. 2016</td>
<td>23 May 2016</td>
<td>275</td>
<td>9</td>
</tr>
<tr>
<td>16 June 2016</td>
<td>23 June 2016</td>
<td>306</td>
<td>10</td>
</tr>
<tr>
<td>16 July 2016</td>
<td>21 July 2016</td>
<td>334</td>
<td>11</td>
</tr>
</tbody>
</table>

*Pinus palustris* Mill.
Table 2.2. Color of PB over 12 months of aging using Munsell Soil Color

<table>
<thead>
<tr>
<th>Age (day)</th>
<th>Hue</th>
<th>Value</th>
<th>Chroma</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>2.5 YR</td>
<td>3</td>
<td>4</td>
</tr>
<tr>
<td>52</td>
<td>5 YR</td>
<td>3</td>
<td>4</td>
</tr>
<tr>
<td>84</td>
<td>2.5 YR</td>
<td>2.5</td>
<td>2</td>
</tr>
<tr>
<td>112</td>
<td>5 YR</td>
<td>2.5</td>
<td>2</td>
</tr>
<tr>
<td>146</td>
<td>2.5 YR</td>
<td>2.5</td>
<td>1</td>
</tr>
<tr>
<td>173</td>
<td>5 YR</td>
<td>2.5</td>
<td>1</td>
</tr>
<tr>
<td>211</td>
<td>10 YR</td>
<td>2</td>
<td>1</td>
</tr>
<tr>
<td>243</td>
<td>10 YR</td>
<td>2</td>
<td>1</td>
</tr>
<tr>
<td>275</td>
<td>10 YR</td>
<td>2</td>
<td>1</td>
</tr>
<tr>
<td>306</td>
<td>10 YR</td>
<td>2</td>
<td>1</td>
</tr>
<tr>
<td>334</td>
<td>10 YR</td>
<td>2</td>
<td>1</td>
</tr>
<tr>
<td>372</td>
<td>10 YR</td>
<td>2</td>
<td>1</td>
</tr>
</tbody>
</table>

Values for color determined using Munsell Soil-Color Charts (Munsell Color, Grand Rapids, MI). Samples were assessed indoors in bright, ambient lighting at 60% moisture.

Samples taken one week after turning of piles.

Hue refers to the dominant spectral or rainbow color, with red (R), yellow (Y), yellow-red (YR) green-yellow (GY), gray (Gley), and white being the dominant hues in communicating soil color. The Munsell soil hue cards include 10R, 10YR, 2.5Y, 2.5YR, 5Y, 5YR, 7.5YR, 10Y – 5GY, Gley 1 and 2, 5R, 7.5R, and white.

Value indicates the degree of lightness or darkness of a color on a gray scale ranging from black to white. In the Munsell color charts, value ranges from 0 (pure black) to 10 (pure white).

Chroma is the purity or saturation of a color. In the Munsell color charts, chroma ranges from 0 (neutral colors) to 8 (most vivid color).
Table 2.3 Particle size distribution of pine bark over 12 months of aging.\textsuperscript{z}

<table>
<thead>
<tr>
<th>Age (mo.)</th>
<th>12.5</th>
<th>9.5</th>
<th>6.3</th>
<th>4.75</th>
<th>2.8</th>
<th>2</th>
<th>1.4</th>
<th>1</th>
<th>0.71</th>
<th>0.5</th>
<th>0.25</th>
<th>0.106</th>
<th>&lt;0.106</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>X-large\textsuperscript{x}</td>
<td>Large\textsuperscript{y}</td>
<td>Medium\textsuperscript{w}</td>
<td>Fines\textsuperscript{v}</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>0</td>
<td>1.2a</td>
<td>6.7a</td>
<td>18.1a</td>
<td>12.3a</td>
<td>20.7a</td>
<td>9.4a</td>
<td>7.0c</td>
<td>5.3b</td>
<td>5.3b</td>
<td>4.9b</td>
<td>5.6c</td>
<td>2.7b</td>
<td>0.8c</td>
</tr>
<tr>
<td>2</td>
<td>0.4a</td>
<td>3.6b</td>
<td>12.1bc</td>
<td>9.7ab</td>
<td>19.5abc</td>
<td>10.3a</td>
<td>8.4a</td>
<td>7.3a</td>
<td>8.1a</td>
<td>7.5ba</td>
<td>8.5bc</td>
<td>3.5ab</td>
<td>1.0bc</td>
</tr>
<tr>
<td>3</td>
<td>0.7a</td>
<td>4.4ab</td>
<td>13.4abc</td>
<td>10.5ab</td>
<td>19.4abc</td>
<td>10.0a</td>
<td>7.8abc</td>
<td>6.5ab</td>
<td>7.5ab</td>
<td>7.6ab</td>
<td>8.1abc</td>
<td>3.3ab</td>
<td>0.9c</td>
</tr>
<tr>
<td>4</td>
<td>0.5a</td>
<td>4.0b</td>
<td>13.6abc</td>
<td>10.5ab</td>
<td>19.8ab</td>
<td>10.1a</td>
<td>8.1ab</td>
<td>6.5ab</td>
<td>7.5ab</td>
<td>7.1ab</td>
<td>7.8bc</td>
<td>3.2ab</td>
<td>1.2bc</td>
</tr>
<tr>
<td>5</td>
<td>0.7a</td>
<td>3.8b</td>
<td>13.1abc</td>
<td>10.3ab</td>
<td>18.8abc</td>
<td>9.6a</td>
<td>7.7abc</td>
<td>6.4ab</td>
<td>8.0a</td>
<td>7.9ab</td>
<td>9.3abc</td>
<td>3.5ab</td>
<td>1.0bc</td>
</tr>
<tr>
<td>6</td>
<td>0.5a</td>
<td>4.4ab</td>
<td>14.8ab</td>
<td>10.2ab</td>
<td>20.0ab</td>
<td>10.2a</td>
<td>7.4abc</td>
<td>6.1ab</td>
<td>7.1ab</td>
<td>6.5ab</td>
<td>7.9bc</td>
<td>2.8b</td>
<td>1.9abc</td>
</tr>
<tr>
<td>7</td>
<td>0.3a</td>
<td>3.8b</td>
<td>11.9bc</td>
<td>9.4ab</td>
<td>18.0abc</td>
<td>9.7a</td>
<td>7.4abc</td>
<td>6.5ab</td>
<td>8.3a</td>
<td>8.3ab</td>
<td>10.5abc</td>
<td>3.7ab</td>
<td>2.2abc</td>
</tr>
<tr>
<td>8</td>
<td>0.4a</td>
<td>3.3b</td>
<td>10.7bc</td>
<td>8.2b</td>
<td>17.2abc</td>
<td>9.2a</td>
<td>7.4abc</td>
<td>6.6ab</td>
<td>8.7a</td>
<td>9.1a</td>
<td>11.7ab</td>
<td>4.5ab</td>
<td>2.9a</td>
</tr>
<tr>
<td>9</td>
<td>0.4a</td>
<td>2.7b</td>
<td>9.4c</td>
<td>7.6b</td>
<td>15.6c</td>
<td>9.0a</td>
<td>7.2abc</td>
<td>6.7a</td>
<td>9.2a</td>
<td>10.1a</td>
<td>13.7a</td>
<td>6.0a</td>
<td>2.4ab</td>
</tr>
<tr>
<td>10</td>
<td>0.4a</td>
<td>3.4b</td>
<td>11.8bc</td>
<td>8.0b</td>
<td>16.1bc</td>
<td>9.1a</td>
<td>7.1abc</td>
<td>6.5ab</td>
<td>8.6a</td>
<td>9.1a</td>
<td>12.5ab</td>
<td>5.5ab</td>
<td>2.1abc</td>
</tr>
<tr>
<td>11</td>
<td>0.4a</td>
<td>3.2b</td>
<td>9.7bc</td>
<td>7.6b</td>
<td>16.8abc</td>
<td>9.6a</td>
<td>7.6abc</td>
<td>7.2a</td>
<td>9.2a</td>
<td>9.9a</td>
<td>11.9ab</td>
<td>4.8ab</td>
<td>2.0abc</td>
</tr>
<tr>
<td>12</td>
<td>0.5a</td>
<td>2.8b</td>
<td>11.7bc</td>
<td>8.8b</td>
<td>16.8abc</td>
<td>9.6a</td>
<td>7.4abc</td>
<td>6.7a</td>
<td>8.9a</td>
<td>9.0a</td>
<td>11.3ab</td>
<td>4.5ab</td>
<td>1.9abc</td>
</tr>
</tbody>
</table>

\textsuperscript{z}Particle size distribution data were collected from three samples from each pile replication per age (9 samples total per age) and are represented as a mean percent by weight of the samples. Analysis performed using Ro-tap Shaker (Model B, W.S. Tyler, Mentor, Ohio).

\textsuperscript{x}X-large particles are greater than 6.3 mm in diameter.

\textsuperscript{y}Large particles are less than 6.3 mm and greater than 2.0 mm in diameter.

\textsuperscript{w}Medium particles are less than 2.0 mm and greater than 0.5 mm in diameter.

\textsuperscript{v}Fines particles are less than 0.5 mm in diameter.

\textsuperscript{a}Means separated within column by Tukey’s HSD with $\alpha \leq 0.05$. Means followed by the same letter are not significantly different.
Table 2.4. White wood content of pine bark (*Pinus pallustris* Mill.) over 0, 3, 6, 9, and 12 months of aging.\(^z\)

<table>
<thead>
<tr>
<th>Age (months)</th>
<th>% Volume(^y)</th>
<th>% Weight(^x)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>6.36a(^w)</td>
<td>3.48a</td>
</tr>
<tr>
<td>3</td>
<td>5.80a</td>
<td>3.24a</td>
</tr>
<tr>
<td>6</td>
<td>5.69a</td>
<td>3.19ab</td>
</tr>
<tr>
<td>9</td>
<td>4.22b</td>
<td>2.63ab</td>
</tr>
<tr>
<td>12</td>
<td>3.80b</td>
<td>2.18b</td>
</tr>
</tbody>
</table>

\(^a\)White wood data were collected from three 200g dry samples from each pile replication per age (9 samples total per age).
\(^y\)Percent white wood by volume per 200g dry PB
\(^x\)Percent white wood by weight per 200g dry PB
\(^w\)Means separated within column by Tukey’s HSD with \(\alpha \leq 0.05\). Means followed by the same letter are not significantly different.
Table 2.5. Sand content of pine bark (*Pinus palustris* Mill.) at 0, 3, 6, 9, and 12 months of aging.\(^z\)

<table>
<thead>
<tr>
<th>Age (months)</th>
<th>% Volume(^y)</th>
<th>% Weight(^x)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>0.43d(^w)</td>
<td>2.80c</td>
</tr>
<tr>
<td>3</td>
<td>1.00cd</td>
<td>5.87bc</td>
</tr>
<tr>
<td>6</td>
<td>2.02bc</td>
<td>13.01ab</td>
</tr>
<tr>
<td>9</td>
<td>2.25ab</td>
<td>14.05a</td>
</tr>
<tr>
<td>12</td>
<td>3.19a</td>
<td>20.15a</td>
</tr>
</tbody>
</table>

\(^z\)Sand content data were collected from three 100g dry samples from a composite sample from all pile replications per age (3 samples total per age).

\(^y\)Percent sand by volume per 100g dry PB

\(^x\)Percent sand by weight per 100g dry PB

\(^w\)Means separated within column by Tukey’s HSD with \(\alpha \leq 0.05\). Means followed by the same letter are not significantly different.
Figure 2.1. Installation of the 12 month pine bark aging study. A) Fresh bark was brought from the lumber company and on the research site for further processing. B) The bark was ground and C) screened through a 1.27 cm screen into three fractions: nuggets, mini nuggets, and fines. D) The fines were placed in three piles (replications) of approximately 191 m$^3$ each, with dimensions of approximately 16.8 m x 10.1 m x 3.2 m.
Figure 2.2. Sampling schematic to illustrate height zones, conceptual grid for randomized sampling within height zone, and sampling depth. A) Stratified subsamples were taken from 9 locations on each pile at three different height “zones” (top, middle, and bottom). Three locations within each height were randomly selected by conceptually dividing the pile into a grid. B) A diagrammatic cross section illustrating each sample location where a hole was dug and were taken from depths (horizontal) of 0.3, 0.6, 0.9, and 1.2 m.
Figure 2.3. Example of a Munsell Soil Color card. Three coordinates – hue (A), value (B), and chroma (C) describe possible colors. Hue refers to the dominant spectral or rainbow color, value indicates the degree of lightness or darkness of a color, and chroma is the purity or saturation of a color. Samples are read in the order of hue value/chroma, e.g. 5YR 3/2.
Figure 2.4. Average pile temperatures (all heights and depths combined) and average monthly ambient temperatures for pine bark (*Pinus palustris* Mill.) over twelve months of aging. Temperature readings were taken approximately one week after turning.
Figure 2.5. Temperature measurements at horizontal depths of 0.3, 0.6, 0.9, and 1.2 m at three different height zones (top, middle, and bottom). Data are averaged among piles.
Figure 2.6. Color changes of bark at (A) 0 months, (B) 2 months, (C) 3 months, (D) 4 months, (E) 5 months, (F) 6 months, (G) 7 months, (H) 8 months (I) 9 months, (J) 10 months, (K) 11 months, and (L) 12 months of aging using a Munsell Soil Color char
Figure 2.7. Percentage of extra-large (>6.3 mm), large (2.0 to 6.3 mm), medium (0.5 to 2.0 mm) and fine (< 0.05 mm) particles over twelve months of aging.
Figure 2.8. Percent differences in particle size fractions between 0 and 3, 0 and 6, 0 and 9, and 0 and 12 months of aging.
Figure 2.9. Total porosity (TP), container capacity (CC), air space (AS) and bulk density (BD) of pine bark (*Pinus pallustris* Mill.) over twelve months of aging. TP = CC + AS.
Figure 2.10. Average volume of pine bark piles and pile bulk density calculated from moisture content at time of sampling.
CHAPTER 3

Hydrologic Properties
Abstract. Pine bark (PB) is one of the most commonly used horticultural substrates, and may be used as a fresh, aged, or composted product. Aged PB is most commonly used; however, there is interest in fresh PB due to its lighter transportation costs and perception that it might be a more a consistent product, due to variability in aged PB substrates. While both fresh and aged PB have been shown to grow plants successfully, there are some concerns about the water holding capacity and hydrophobicity of fresh PB. There is little research conducted that investigates the changes of PB during a managed aging process. To better understand the effects of aging and pile management on PB substrates, a twelve-month study was initiated at a local PB supplier in NC. Fresh PB was processed and the fines (less than or equal to 1.27 cm) were placed in three pile replications of 191 cubic meters each. Piles were turned monthly. Beginning at time 0 and every 4 to 5 weeks for a period of twelve months, stratified subsamples were taken from each pile one week after turning at three different heights (top, middle, and bottom) at horizontal depths within pile of 0.3-1.2 m to account for variations in pile depth and height. The stratified subsamples were mixed into one composite sample for each pile replication to test the hydrologic properties of the bark. Hydrologic properties included wettability, saturated hydraulic conductivity ($K_{sa}$), moisture retention curves (MRC), ECV models for nursery containers, and pore fraction volume analysis. There was a decrease in extra-large and large particles and an increase fines from months 0 through 12. There were no differences in TP after month 0 with values around 77.9 to 82.2% from months 2 through 12, while CC increased from 40.3 to 55.5 %, AS decreased from 37.6 to 26.8%, and BD increased from .18 to .21 g/cm$^3$, further illustrating the shift to greater amounts of fine particles over time, resulting in smaller pore sizes within the same pore volume. Sand content increased over time, and could have an influence on the physical properties. Data for saturated hydraulic conductivity showed high variability within
age, likely due to high variation in the particle shapes of bark materials, resulting in a non-uniform pore distribution that effected the rate of percolation in certain samples. However, a decreasing trend in saturated hydraulic conductivity could be seen with age in response to the increasing percentage of fines in the older bark. Despite the shift to a larger amount of fine particles, and increase in sand, and a stabilization of PB physical properties after 6 months, there were no differences in wettability at 50 and 25% gravimetric water content, moisture retention curves, or unavailable water values. These data indicate that Fresh PB may display more percolation, however this is a possibility with all PB due to its coarse nature, but there are little differences between fresh and aged in terms of hydration or water holding capacity under the aging and pile management conditions of this study.
Introduction

Container production accounts for nearly 66% of the total nursery crop production within the United States (U.S. Department of Agriculture, 2015). Currently, approximately 70% of the freshwater consumed in the world is used for agricultural purposes, and nearly 40% of the freshwater used in the US is used to irrigate crops (Kenney et al., 2005). During the growing season, a container nursery may consume upwards of 177.3 m$^3$ of water per ha each day (Fulcher and Fernandez, 2013). Containerized crop producers are becoming increasingly conscious of water use due to economic decisions, governmental restrictions, and/or increased environmental sustainability concerns (Fulcher et al., 2016). Freshwater is a finite resource that is rapidly becoming more scrutinized in agricultural consumption, and ornamental crop producers must continually improve production sustainability with regards to irrigation in order to continue to stay economically viable.

Because of the coarse nature of PB substrates, frequent watering is required. Hoskins et al. (2014) demonstrated that irrigation water has a tendency to preferentially flow through certain portions of a PB substrate in nursery containers, leading to uneven post-irrigation water distribution and unnecessary loss of water and leaching of fertilizers. This may be of concern in fertigation systems, as fertilizers may quickly leach and not reach the entire root zone. Moisture content (MC) plays an important role in this preferential flow – at lower MCs an infiltrating channel of water will be relatively narrow and fast moving, whereas at high MCs it will be relatively wide and slow moving (Hoskins and Owen, 2014). This is due to the effect that moisture content has on the hydration efficiency, or wettability (degree of hydrophilicity/hydrophobicity) of a substrate. Wettability characterization is important in horticulture because of the widespread use of organic materials as primary components in
growing media, and the variation of water and air availability in the limited volumes of containers (Michel, 2015). In addition to issues involving preferential water and irrigation flow, hydrophobic properties of PB substrates may negatively affect the physical properties of the media, and may result in a need for increased rewetting times between irrigations after excessive drying. (Michel, 2015). Beardsell (1982) found that hydrophobic properties of bark increase with drying, and that once dry, PB substrates may be especially difficult to rehydrate. In addition to its texture, numerous sites exist on the internal surfaces of the bark particles for the absorption of water and nutrients. These small cellular connections may be a reason why bark is difficult to hydrate (Thomas and Perry, 1980).

The term available water (AW) in reference to soilless substrates is the amount of water held between CC and permanent wilting point (PWP; -1.5 MPa). Container capacity (CC) and AW of a substrate vary with different container geometries (Bilderback and Fonteno, 1987; Milks et al., 1989). Unavailable water (UW) is defined as the water that is adsorbed to the particle surface of the substrate held at a tension equal to or greater than PWP, making it unavailable for plant use. As more water is removed from the substrate, greater suctions are needed to remove the remaining water. Permanent wilting point varies with the substrate and the crop being grown; therefore, there the potential at which water in the substrate ceases to be available is not always the same (Fields, 2014). In organic substrates, the volumetric fraction of UW is larger than that of mineral soils, due to the increased surface area of organic particles. In PB, UW has been reported to be near 27%. High unavailable water content is assumed to be tightly bound around bark particles and in vesicles inside bark particles even at low tensions, such as after drainage. Much of the water in PB substrates may be tightly bound to mineral crystals, organic compounds, or in occluded pores (Hanson et al., 2004).
Saturated hydraulic conductivity ($K_{\text{sat}}$) is one of the most important metrics for soil-water-plant interactions, as well as water solute movement and retention through the soil profile (Deb and Shukla, 2012). Saturated hydraulic conductivity refers to the steady infiltration rate at which water percolates through the substrate after a head of water has accumulated on the surface and free drainage is occurring from the bottom (Handreck and Black, 2010). It is a result of the cohesive and adhesive nature of water in that when the volumetric water content increases there are higher proportions of capillary water, which fills more pores and allows for more paths in which water can move (Fields, 2016). An understanding of $K_{\text{sat}}$ can help growers to make more informed irrigation decisions. Raviv et al. (1999) discussed the importance of measuring in situ hydraulic conductivity and deemed decreasing $K_{\text{sat}}$ as the primary limiting factor for water uptake by roots in soilless substrates.

An important technique for determining how a substrate will retain and release water is the determination of a moisture retention curve (MRC). MRCs describe the changes in substrate water content with increasing matric suction, and the percent water (by volume). Using the differences in water content at specific tensions on an MRC, easily available water (EAW), which is defined as water held at tensions between -1.0 to -5.0 kPa, and water buffering capacity (WBC), water held at tensions between -5.0 and -10.0 kPa (de Boodt and Verdonck, 1972), can be measured. Substrate water is not held evenly throughout the entire range of its availability. Instead different amounts are released under different pressures (Karlovich and Fonteno, 1986), and these values can accurately predict how a substrate will hold and release water at over broad tension ranges. Moisture retention curves may also be used to describe the structural effects of substrates on air and water relationships, and can be used to model these relationships in containers of different sizes (Tilt et al., 1987, Milks et al., 1989, Fonteno, 1989, and Drzal et al., 1994). This is accomplished using Van Genuchten models derived from MRC data. This is a non-linear, five parameter model (Van
Genuchten and Nielsen, 1985) which was adapted for horticultural substrates by Milks et al. (1989a) as follows:

\[ \Theta = \Theta_r + \frac{(\Theta_s - \Theta_r)}{[1+(\alpha h)^n]^m} \]

where \( \Theta \) is volumetric water content, \( \Theta_s \) is the volumetric water content at saturation (0 kPa), \( \Theta_r \) is the residual water content (-30 kPa), \( h \) is the height of the column or moisture tension, \( \alpha \) is the inverse of the “air entry value,” and \( n \) and \( m \) are curve fitting parameters. \( \Theta_s \) and \( \Theta_r \) values can be used in an equilibrium capacity variable (ECV) model to predict TP, AS, and CC of a substrate in containers of different sizes and shapes (Bilderback and Fonteno, 1987; Milks et al., 1989c). Container size has been shown to influence substrate physical properties, which in turn will influence irrigation management of container crops (Bish et al., 1997; Milks et al., 1989c; Owen and Altland, 2008).

Moisture retention curves are directly related to the pore size distribution of a horticultural substrate (Milks et al., 1989a). Pore size distribution analysis allows for inferences into the internal structure of the substrate and may allow one to visualize ‘internal’ structure of the substrate (Drzal et al., 1999). Pore fraction analysis as described by Drzal et al. (1999) is separated into four distributions. Macropores are pore sizes > 416 \( \mu \); pores within this range cannot hold water under tension induced by gravity when allowed to drain after saturation. Mesopores are in the range of 416 to10 \( \mu \). Micropores are categorized into the range of 0.2 to 10 \( \mu \), and are equivalent to volumes of water held between 30 kPa and 1.5 mPa. The water in these pores may be viewed as water not commonly used under normal irrigations, but as a stress buffer extracted by plant roots when suctions exceed 30 kPa. Ultramicropores have effective pore diameters of < 0.2 \( \mu \), and hold water at suctions > 1.5 mPa. Water found in ultramicropores would be considered unavailable to plants (Drzal et al., 1999). The air and water relationship in a container medium is dynamic, and can be effected by substrate physical and hydraulic properties (and the interactions between the two),
irrigation method, plant species, and container geometry (Drzal, 1994). Relating physical and hydraulic properties such as pore size and hydraulic conductivity may assist in understanding water transport characteristics of substrates.

As discussed in Chapter 2, the processing and handling of PB may cause PB substrates to contain a significant amount of sand. This can improve water holding capacity and reduced water infiltration rates of substrates, resulting in better hydration (Kreweer and Ruter, 2012). Brown and Pokorny (1975) found that amending PB with sand increased bulk density, which decreased K_{sat}. Niemiera et al. (1994) reported that PB substrates with 9:1 and 5:1 bark/sand ratios had decreased drainage and UW values at 1.5 kPa tension, and increased solids, AW, and bulk density. Greater concentrations of small pores of the 5 PB: 1 sand substrate allowed for less percolation and pore dispersion of solution than 100% PB, which demonstrated greater channeling and less retention of water/nutrient solutions (Niemiera et al., 1994). Sand also increases the proportion (by weight) of smaller particle size fractions (Brown and Pokorny, 1975; Niemiera et al., 1994). This increases available water due to the addition of sand created micropores that hold water less strongly than the surface and intraparticle water adsorption sites of PB particles.

The differences in the physical properties of PB of different ages as discussed in Chapter 2, especially particle size distribution (PSD), will have a great impact on the hydrologic properties of PB substrates of different ages. Age may also influence the hydrophobicity of PB due to resins and waxes (Bunt, 1988; Pokorny, 1979) in fresh PB that have not had time to degrade over an aging process. Differences in the hydrologic properties of fresh and aged PB have been investigated in the literature, but the authors were unable to find any data for PB of specific ages. Airhart et al. (1978) stated that both fresh and aged PB have been shown to
become hydrophobic when moisture content falls below 34%. Self (1978) stated that aging of PB is unnecessary except for stockpiling inventory and improving wettability. Laiche (1974) reported reduced plant quality in fresh PB due to difficulty maintaining adequate moisture levels during the first three months after transplanting, and experienced difficulty in hydrating fresh PB. Harrelson (2004) reported reduced growth of *Cotoneaster dammeri ‘Skogholm’* in fresh PB vs. aged, due to lower container capacity and available water. Bilderback et al. (2013) stated that aged PB is preferred because fresh PB often has less than 20-30% fine particles, which results in reduced water holding capacity.

The objective of this research was to investigate and quantify the changes in the hydrologic properties of PB during 12 months of managed aging, in order to better understand the aging process and utilize PB at various degrees of age and stability. The variation of bark materials in horticultural substrates is due in large part to the lack of understanding of processing and handling of these materials. Water is the greatest limiting factor in plant growth (Tilt, et al. 1987), therefore understanding the hydrologic properties of substrates is essential in order to efficiently manage irrigation and fertilization in the nursery industry. Assessing the potential of PB substrates of different ages can aid in the optimal use of resources such as water to improve efficiency and sustainability in container production.

**Materials and Methods**

*Sampling.* Sampling was conducted as described in Chapter 2.

*Particle size distribution.* From each composite pile sample per sample date, approximately 3 L of PB was spread out to a depth of 2 cm on a drying pan and placed in a forced air dryer for 48 h. at 105°C. Particle size distribution (PSD) for four fractions (X-large: > 6.3 mm, large: between
6.3 mm and 2.0 mm, medium: between 2.0 mm and 0.5, and small: < 0.5 mm) was determined by passing three 100g replications of the dry material through a column of three sieves (W.S. Tyler, Mentor, OH) with screen sizes of 6.3, 2.0, and 0.5 mm, with a pan to collect the particles that passed through the 0.5 mm sieve. The column of sieves was placed in a Ro-Tap Shaker (W.S. Tyler, model B; Mentor, OH) and shaken for five min. The sieves were separated and the contents of each sieve were weighed separately. Particle size was expressed as a percentage of the total weight of the 100g sample. Particle size is expressed as a percentage of the total weight of the 100 g sample. Three replications were analyzed from each pile replication, equaling 9 replications per sample date.

*Hydration efficiency.* For each sample date, 3 L bark of approximately 60% MC by weight was spread on two trays to a thickness of approximately 1.5 cm. The trays were left out uncovered to air dry and MC was monitored until the trays were at 50% and 25% MC by weight. After the desired MC was obtained, the hydration efficiency of the bark samples was conducted following the procedures described by Fields et al. (2014). For each sample date, four samples (replications) of bark per pile were packed into transparent cylinders fitted with a mesh screen on the bottom for drainage. Samples were packed to a height of 10 cm. and a weight within 5% of the other three replications. A diffuser was placed on top of the packed cylinder to simulate a drip irrigation system and placed under a separatory funnel. A hydration event was performed by placing 200 mL of tap water into the funnel and slowly passing it through the diffuser and onto the substrate. Due to the hydrophobicity of bark at 25% MC, the water flow rate from the funnel into the diffuser was adjusted with the funnel stopcock to prevent ponding on top of the substrate. Water that was not absorbed by the substrate during each hydration event flowed through the screen at the bottom of the cylinder and was collected into a beaker. Samples were allowed to equilibrate until dripping ceased (approximately 3 to 5 min.) and the effluent in the
beakers was recorded. The amount of water retained by the sample was calculated by subtracting the effluent volume from the total water applied. A second hydration event was performed with a new 200 mL of water applied through the same funnel and subsequent effluent collected. This was repeated for 10 hydration events. After 10 hydration events, the cylinders were weighed and sample height measured to account for any changes in volume due to shrinking or swelling.

To determine maximum hydration, container capacity was measured by placing the cylinders into a Buchner funnel and saturating as described in the North Carolina State University Porometer Manual (Fonteno and Harden, 2010). Samples were saturated from below by adding water to stoppered funnels in a step-wise fashion, increasing the water level by 1/3 every 2 min until it was even with the top of the substrate (10 cm from bottom of cylinder). After equilibrating for 15 min, the stopper was removed from the bottom of the funnel and the sample was allowed to drain for 30 min. The saturated samples were reweighed and heights were measured again to observe any changes in volume. Samples were dried in a forced-air drying oven at 105 °C for 48 h. Once dry, samples were weighed and dry weight was used to determine MC and the total water retained in the material. Four replications from each pile were analyzed, giving a total of 12 replications per sample date.

*Saturated hydraulic conductivity.* Saturated hydraulic conductivity ($K_{\text{sat}}$) was determined for PB aged 0, 3, 6, 9, and 12 months using the procedure and equipment described by Drzal (1994; Fig. 3.1). For each sample date, four replications of bark from each pile were packed into four 347.5 mL aluminum cores (7.6 cm x 7.6 cm; twelve replications per sample date) as described in the North Carolina State University Porometer method (Fonteno and Harden, 2010), and each core was fitted with an individual funnel adaptor ring on the bottom and a plastic cylinder with a spout on the top. Each core assembly was secured onto an individual stationary funnel mounted on the
$K_{\text{sat}}$ unit. A constant head reservoir, mounted to laboratory jacks, was filled with water and maintained at a constant level by a recirculation system. Rubber tubing was connected from the reservoir to the bottom of the stationary funnel, allowing water to saturate each core unit from the bottom. A hydraulic head was created by raising the reservoir a measured distance above the sample. Saturation of the samples was achieved by slowly raising the reservoir in 1 cm increments from the base of the sample until the water level in the reservoir was at an equal level with the sample surface. The samples were then allowed to equilibrate for 15 min before initiating flow. Flow was initiated by imposing a hydraulic head of 0 cm, followed by a hydraulic head of 2 cm which was used for $K_{\text{sat}}$ measurement. The system was allowed to equilibrate for 15 min at each selected hydraulic head. After equilibration, a volume of water was collected in a 1 L flask placed under the spout of each core assembly and the time taken for the water to reach that volume was recorded. Piezometers mounted to the reservoir and each core assembly were used to precisely measure hydraulic head differences ($\Delta H$). $K_{\text{sat}}$ was determined using Darcy’s Law equation:

$$Q = K_{\text{sat}} \frac{dH}{dx}$$

where $Q$ is the volume flux density (the volume of liquid phase passing through unit cross-sectional area of soil in unit time), $dH$ is the head difference, and $dx$ is the distance.

Unavailable water. Moisture retention curves and physical properties were determined for bark aged 0, 3, 6, 9, and 12 months. Due to similarity in the physical properties between different piles, equal parts of material from each of the three piles were combined into one composite sample for each age. For each sample date, bark was hydrated to 66% MC by weight and sealed in 4 mil poly bags (61 cm x 76.2 cm; Consolidated Plastics, Stow OH) for 12 h to equilibrate. Samples of bark from each age were then spread to a thickness of approximately 1.5 cm on a
tray, which remained uncovered to air dry. Moisture content was monitored until the trays were at 40% MC by weight. At this point, X-large size particles (>6.3 mm) were removed from each sample by shaking the material by hand for 1 min through a 6.3 mm sieve (W.S. Tyler, Mentor, OH), and five 3.29 mL subsamples of bark were measured using a 2/3 teaspoon measuring cup, and placed in 1.1 x 3.7 cm stainless steel cups (Decagon; Pullman, WA). Plastic lids were placed on the cups and sealed with Parafilm M® (American Can Co.; Greenwich, CT) before measurement to prevent evaporative water loss. The water potential of the subsamples were then individually tested using a dewpoint potentiometer (WP4C, Decagon; Pullman, WA). The material on the tray was allowed to continue to dry down, and samples were taken continuously to maximize the amount of replications with readings between -1.0 to -2.0 mPa. Data points between -0.5 and -2.5 mPa were plotted, and the regression equation was used to calculate MC at -1.5 mPa.

*Moisture retention curves.* Moisture retention curves and physical properties were determined for bark aged 0, 3, 6, 9, and 12 months. Due to similarity in the physical properties between different piles, equal parts of material from each of the three piles were combined into one composite sample for each age. Physical properties (TP, CC, AS, BD) were determined using the North Carolina State University Porometer method, and MRCs were obtained using procedures in the North Carolina State University Horticulture Substrates Laboratory Manual (Fonteno and Harden, 2010). Five replications of each age were packed in 347.5 mL aluminum cores (7.6 cm x 7.6 cm) to the same bulk densities as determined for each age using the porometer. The packed cores were placed in volumetric pressure plate extractors (VPPE) fitted with 50-kPa ceramic plates on the bottom of the unit (Soilmoisture Corp., Santa Barbara, CA) in a controlled-temperature chamber held at 22 °C (North Carolina State University Horticultural Substrates Laboratory). Samples were saturated by adding tap water to the VPPEs in a stepwise fashion, and were allowed to
equilibrate for 48 h before drainage. Samples were covered to prevent evaporation, drained for an additional 48 h, and water effluent volumes were recorded. Metal lids were fitted onto the top of each VPPE and pressures of 1.0, 2.0, 4.0, 5.0, 7.5, 10, 20, and 30 kPa were applied over the course of 16 days. Each pressure was applied for 48 h, and drainage from each bark sample was recorded. After drainage from the last applied pressure was recorded, the cores were removed from the VPPEs, weighed, and placed forced air dryer for 48 h. at 105 °C. The dried samples were weighed, and Θ was calculated.

Moisture retention curve plots and modeling. A scatterplot was created for each age to show the relationship between the suctions and their corresponding Θ values. Values for 0 kPa (saturation) and 0.38 kPa (initial drainage) were under-represented using VPPE values, so TP and CC values derived from the porometer data were used to provide a more accurate estimate of TP and CC during free drainage (Milks et al. 1989). Values for EAW and WBC, which categorize a substrate’s water-holding ability, were calculated using the method described by de Booit and Verdonck (1972), from points on the curve (1.0 to 5.0 kPa for EAW and 5.0 to 10.0 kPa for WBC). Values for residual water at -10 kPa (RW10) and -30 kPa (RW30) were determined as described by Fields et al. (2014). Proc NLIN (Version 9.2; SAS Institute, Cary, NC) was used to create the five parameter Van Genuchten model for horticultural substrates (Milks et al., 1989a).

Equilibrium capacity variable models. Equilibrium capacity variable modeling was performed for PB aged 0, 3, 6, 9, and 12 months to determine simulated physical properties of the substrate in different sized and shaped containers. This was achieved using values obtained by modeling the MRC curves with the Van Genuchten models (Milks et al., 1989b). These models use Θs and Θr, the curve fitting parameters (a, n, and m), and specific container dimensions to describe TP,
CC, AS, AW and UAW of the substrate in each individual container. The containers chosen for the ECV models are those commonly used in nursery production with PB substrates (Table 3.1).

**Pore volume fraction analysis.** Pore size distribution curves for PB aged 0, 3, 6, 9, and 12 months were derived directly from the MRC data, as described by Drzal et al. (1999). The effective diameter of the largest water-filled pore at each tension was calculated as follows:

\[ d_p = \frac{4\sigma x 10^5}{\rho_w gh} \]

where \( \sigma \) is surface tension (J∙m\(^{-2}\)) and \( \rho_w \) is the density (Mg∙m\(^{-3}\)), \( g \) is gravitational acceleration (m∙s\(^{-2}\)), and \( h \) is the matric suction (cm of water). The pore size distribution was divided into four groups: macropores, mesopores, micropores and ultramicropores. Macropores had a minimum pore diameter of 300 µM (0 to 10 cm tension). Mesopores were pores with diameters between 300 and 30 µM (10 to 100 cm tension), and micropores had diameters between 30 and 0.2 µM (300 to 15,000 cm tension). Ultramicropores had diameters <0.2 µM (>15,000 cm tension) and may include internal pores that are not normally available to plants (UW).

**Sand content.** Sand content was determined as in Chapter 2.

**Statistical analysis.** Data were checked for normality and assumptions and analyzed using the mixed procedure in SAS, and means were separated using Tukey’s HSD with \( \alpha = 0.05 \) to observe differences between PB of different ages. Table 3.4 was produced from mathematically derived modeling algorithms, which required means of repetitions to produce curve fitting parameters for use in the ECV models. To validate the model, means of moisture retention data were compared with points of corresponding \( \theta \) and to model predictions using the 7.6 cm aluminum core as the container.
Results and discussion

Particle size distribution. The percentage of extra-large (>6.3 mm) and large (2.0 to 6.3 mm) particles decreased over the course of the study (Table 3.2). The percentage of medium (0.5 to 2.0 mm) particles increased between 0 and 11 months, although month 12 showed no difference compared to the other months. The percentage of fines (<0.5 mm) increased to month 11, with month 12 again showing no difference compared to the other months.

Wettability. The initial moisture contents of 50% and 25% for this experiment were determined by mass, which is the industry standard when testing bulk materials with no specific container (Fields et al., 2014); however, the hydration efficiency curves are determined using volumetric water content. The volumetric water content values of the curves at 0 hydration events are the initial moisture content values by weight (50 or 25%), expressed volumetrically. At 50% MC, the bark hydrated similarly for all ages. At 25% MC, only 5 mo. aged bark reached CC; however, values for volumetric water content for 6 mo. were within the standard deviation for CC (Fig. 3.3). This is consistent with the work of Airhart et al. (1978), where it was shown that MC over 34% was needed to hydrate PB. All PB samples at 25% MC exhibited channeling and preferential flow (Appendix D), with large areas of the substrate never hydrating at all (even after complete saturation in some cases). This is consistent with what has been reported in terms of uneven water distribution and preferential flow/channeling at lower MCs by Hoskins and Owen (2014). At 25% MC, all samples showed less water uptake than at 50% MC, regardless of sample date. Hydrophobicity was not improved with age.

Hydraulic conductivity. The data obtained from this experiment displayed high dispersion, resulting in no differences between ages when the complete data set was analyzed. The authors hypothesize that the variability occurred due to high variation in the particle shapes of bark
materials, resulting in a non-uniform pore distribution that affected the rate of percolation in certain samples. To reduce the influence of unrepresentative values that did not describe the characteristic of the datasets, means were analyzed using data from within the interquartile range. Means run on data within the IQR were the closest to the means of the original data set (Table 3.3), and we felt that it best expressed the trends that we observed as the tests were being conducted. When analyzed in this fashion, \( K_{\text{sat}} \) values for PB decreased with age, with 119.3 cm/min for month 0, 90.9 cm/min for month 3, 80.38 cm/min for month 6, 73.5 cm/min for month 9, and 54.9 cm/min for month 12 (Fig. 3.1). This can be explained by reductions in particle size over time due to mechanical breakdown by turning and microbial degradation, as well as an increase in sand content, which will be further discussed below. The smaller particles result in smaller pore sizes and an increased bulk density. Larger pore spaces created by a greater percentage of larger particle size fractions in fresh PB should permit greater \( K_{\text{sat}} \) under saturated conditions.

*Physical properties of composited pile reps for SMT.* Values for TP were similar across all ages (Table 3.4). CC increased from 0 to 12 months, and AS decreased. BD increased over time, from 0.18 g/cm\(^3\) at month 0 to 0.21 g/cm\(^3\) at month 12. There were no differences in EAW among the different ages, and there were no obvious trends in terms of age for WBC, RW10, and RW30.

*Moisture retention curves.* Moisture retention curves were similar for months 0, 3, 6, 9, and 12 (Fig. 3.4), and were similar to MRC values of de Boodt and Verdonck (1972). These similar trends, despite changes in PSD and physical properties with age, could be a result of the technique used to determine the MRC values. Fields et al. (2016), found that MRC values from pressure extraction analyses become constant at a given \( \Theta \) between -0.65 and -1.0 kPa (which can be seen in the data produced from our study [Fig. 3.4]), while MRC values from an evaporative
method continue to experience a decrease in Θ as suction increases. It has been hypothesized that these differences are due to loss in hydraulic connectivity between coarse PB particles and the ceramic plate within the VPPE. Other researchers have observed similar trends with coarse, highly porous substrate materials or soil (Fields et al., 2014; Fonteno and Bilderback, 1993; Gee et al., 2002; Stevenson, 1982). Our results may further support the concept that substrates with high proportions of coarse particles may lose the connection of water between the sample and the pressure plate at low pressures.

**Unavailable water.** There were no differences in UW for PB aged 0, 3, 6, 9, and 12 months (Table 3.2), with average θ values at -1.5 MPa of 6.90, 6.90, 6.86, 7.29, and 8.50 % for months 0, 3, 6, 9, and 12, respectively. Our results are similar to those described by Fields (2014), who obtained θ values between 7.1 and 7.9 % using dewpoint potentiometry and linear regression predictions. Our values are much lower than UW values previously reported in the literature: Bilderback (n.d.) reported UW values of 31% for fresh PB and 30% for aged, and Harrelson et al. (2004) reported values of 39.2% for fresh PB and 35.8% for aged. Wright and Browder (2005) reported UW of 26.6 % for PB. These differences are likely due to differences in measurement techniques obtained using pressure plates as compared to dewpoint potentiometry. When pressure is applied to samples during VPPE analysis, some pores may trap water in what become inaccessible pores that would normally be drained at a given applied pressure, resulting in the assumption that these pores are smaller than their actual size and never drain, underestimating available water and overestimating UW.

**ECV models.** ECV models showed a change in CC and AS with changing container size for all ages, with changes between the containers being the greatest for PB aged 12 months (Table 3.5). Total porosity of each age was unchanged regardless of container size, because as long as BD is
constant, TP will remain the same (Milks et al., 1989b). For month 0, there was a 3.3% decrease in CC and increase in AS between the #1 and #25 containers, and in month 12 there was an 7.3% decrease in CC and increase in AS. The decrease in CC values as container size increases, which can be thought of as an increase in drainage, is due to the increased gravitational forces drawing more water out of the smaller pores in taller containers (Spomer, 1974), as well as increased hydraulic head in taller containers which creates more positive pressure on water in the pore spaces (Fields et al., 2014b). Taller containers will have higher total AS and lower CC compared with smaller containers (Owen and Altland 2008). The increase in CC and general decrease in AS was more pronounced between PB of different ages as container size increased. Owen and Altland (2008) found that AS increased linearly and CC decreased linearly in douglas fir bark of two different textures (medium :<2.2 cm, and fine:<0.9 cm) with increasing height of the cores used to measure physical properties. Differences in CC and AS values were greatest between container sizes for fine bark (Owen and Altland, 2008), which is consistent with the data presented here, indicating that bark age may have a greater influence on irrigation requirements for plants grown in larger containers.

**Pore fraction analysis.** Changes in the pore fractions of bark from 0, 3, 6, 9, and 12 months of aging were minimal (Fig. 3.5). Solids ranged from 22.1, 16.5, 16.6, 14.6, and 17.8 % for months 0, 3, 6, 9, and 12, respectively. Utramicropores, which are the same as the θ values for UW, increased slightly, from 6.90, 6.90, 6.86, 7.29, and 8.5 % for 0, 3, 6, 9, and 12 months, respectively, but as seen with the UW data, there were no statistical differences. The percentage of micropores were 23.4, 25.0, 23.6, 22.4, and 20.6 % for months 0, 3, 6, 9, and 12, respectively. Mesopore percentages ranged from 3.6, 8.5, 4.5, 5.5, and 3.6 %, and macropore values increased slightly from 44.0, 43.1, 48.4, 50.2, and 49.5 % for months 0, 3, 6, 9, and 12, respectively.
Just as there were minimal differences in the SMT curves, the differences in these data are minimal as well, which may be in part due to the technique used to determine MRCs as discussed above. Moisture retention is due in large part to particle/pore distributions, so one would expect to see the same trends represented in both data sets, especially as the pore volume fraction data is derived from the MRC results. These data show that the majority of the volume in a container will be taken up by macropores, followed by micropores, solids, ultramicropores, and mesopores. Because these pore fractions are based on intended use, the range of pores within a given pore fraction can vary since the average effective suction will change with the height of the substrate and container geometry (Drzal et al., 1999).

Sand content. Sand content increased over the course of the aging process (Table 3.6), from 5.05, 8.52, 11.29, 16.10, and 15.90 % by weight and 0.79, 1.31, 1.75, 2.70, and 2.64 % by volume for months 0, 3, 6, 9, and 12, respectively. If we subtract the weight of the sand from the BD values that were calculated, the differences between the BD values are minimized, ranging from 0.17 g/cm$^3$ for months 0 and 3, and 0.18 g/cm$^3$ for months 6, 9, and 12. These sand additions over time could potentially increase the water holding abilities of PB substrates.

Summary

Despite decreases in extra-large and large particle fractions and increases in fine particles, as well as increases in BD and CC, there were no differences in wettability, moisture retention curves, or unavailable water for any ages of bark. We did observe a decreasing trend in $K_{sat}$ (if using IQR data), due to a shift to smaller particle sizes, which could indicate that fresh bark might need to be watered more frequently, however the coarse nature of PB particles may result in variability in hydraulic conductivity, regardless of age. The hydration efficiency data indicate that younger PB will not be more difficult hydrate than aged; however, all PB samples were
difficult to hydrate at 25% gravimetric moisture content, and displayed preferential water flow/channeling.

**Literature cited**


Bilderback, T. Managing container substrates. n.d.


Owen, J.S., Jr. and J.E. Altland. 2008. Container height and douglas fir bark texture affect 

29:484-495


Spomer, L.A. 1974. Two classroom exercises demonstrating the pattern of container soil water 

Stevenson, D.S. 1982. Unreliabilities of pressure plate 1500 kilopascal data in predicting soil 
419.

Thomas, S. and F.B. Perry, Jr. 1980 Ammonium nitrogen accumulation and leaching from an all 

Tilt, K.M., T.E. Bilderback, and W.C. Fonteno. 1987. Particle size and container size effects on 

Online_Resources/Census_of_Horticulture_Specialties/HORTIC.pdf>.

Van Gencuchten, M.T. and D.R. Nielsen. 1985. On describing and predicting the hydraulic 

bark as a growing medium for plants. University of Natal, Pietermaritzburg.


Table 3.1. Dimensions of commonly used nursery containers which were used to calculate ECV models.

<table>
<thead>
<tr>
<th>Container</th>
<th>Bottom radius (cm)</th>
<th>Top radius (cm)</th>
<th>Height (cm)</th>
<th>Vol.(L)</th>
</tr>
</thead>
<tbody>
<tr>
<td>#1(^y)</td>
<td>6.50</td>
<td>10.00</td>
<td>17.78</td>
<td>3.86</td>
</tr>
<tr>
<td>#2(^x)</td>
<td>9.00</td>
<td>12.07</td>
<td>21.90</td>
<td>7.70</td>
</tr>
<tr>
<td>#3(^w)</td>
<td>10.50</td>
<td>13.97</td>
<td>24.13</td>
<td>11.25</td>
</tr>
<tr>
<td>#5(^v)</td>
<td>11.25</td>
<td>15.08</td>
<td>26.04</td>
<td>14.30</td>
</tr>
<tr>
<td>#7 squat(^a)</td>
<td>14.50</td>
<td>19.05</td>
<td>31.75</td>
<td>28.28</td>
</tr>
<tr>
<td>#10(^t)</td>
<td>16.00</td>
<td>20.96</td>
<td>36.20</td>
<td>39.13</td>
</tr>
<tr>
<td>#15(^s)</td>
<td>18.00</td>
<td>23.34</td>
<td>38.42</td>
<td>51.93</td>
</tr>
<tr>
<td>#25(^r)</td>
<td>23.00</td>
<td>30.16</td>
<td>46.04</td>
<td>102.95</td>
</tr>
</tbody>
</table>

\(^z\)ECV models are calculated from container dimensions and moisture retention curves to determine simulated physical properties of the substrate.

\(^y\)Trade 1-gallon container.

\(^x\)Trade 2-gallon container.

\(^w\)Trade 3-gallon container.

\(^v\)Trade 5-gallon container.

\(^a\)Trade 7-gallon container.

\(^t\)Trade 10-gallon container.

\(^s\)Trade 15-gallon container.

\(^r\)Trade 25-gallon container.
Table 3.2. Particle size distribution of pine bark (*Pinus palustris* Mill.) over twelve months of aging.\(^z\)

<table>
<thead>
<tr>
<th>Age</th>
<th>Extra large(^y)</th>
<th>Large(^x)</th>
<th>Medium(^w)</th>
<th>Fines(^v)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>21.3a(^a)</td>
<td>41.6a</td>
<td>24.9b</td>
<td>12.2c</td>
</tr>
<tr>
<td>2</td>
<td>18.5ab</td>
<td>38.3ab</td>
<td>28.7ab</td>
<td>14.5bc</td>
</tr>
<tr>
<td>3</td>
<td>18.2ab</td>
<td>39.8ac</td>
<td>29.4ab</td>
<td>12.7c</td>
</tr>
<tr>
<td>4</td>
<td>18.1ab</td>
<td>39.0abd</td>
<td>29.3a</td>
<td>13.6c</td>
</tr>
<tr>
<td>5</td>
<td>18.0ab</td>
<td>39.1ab</td>
<td>29.6a</td>
<td>13.4c</td>
</tr>
<tr>
<td>6</td>
<td>18.5ab</td>
<td>39.9ab</td>
<td>28.4ab</td>
<td>13.3c</td>
</tr>
<tr>
<td>7</td>
<td>18.8ab</td>
<td>38.6ab</td>
<td>29.1ab</td>
<td>13.5c</td>
</tr>
<tr>
<td>8</td>
<td>15.4ab</td>
<td>34.7bcde</td>
<td>31.0a</td>
<td>18.9abc</td>
</tr>
<tr>
<td>9</td>
<td>14.5ab</td>
<td>33.2de</td>
<td>31.7a</td>
<td>20.6abc</td>
</tr>
<tr>
<td>10</td>
<td>14.4b</td>
<td>32.3e</td>
<td>32.1a</td>
<td>21.2a</td>
</tr>
<tr>
<td>11</td>
<td>14.9b</td>
<td>31.9e</td>
<td>31.5a</td>
<td>21.6a</td>
</tr>
<tr>
<td>12</td>
<td>17.6b</td>
<td>34.8bde</td>
<td>28.6ab</td>
<td>18.9abc</td>
</tr>
</tbody>
</table>

\(^a\)Particle size determined using a Ro-tap shaker for 5 min.

\(^y\)Extra large particles = 6.3 mm diameter or larger.

\(^x\)Large particles = 2.0 to 6.3 mm diameter.

\(^w\)Medium particles = 0.5 to 2.0 mm diameter.

\(^v\)Fine particles = less than 0.5 mm diameter.

\(^u\)Means separation down column using Tukey’s HSD with \(\alpha = 0.05\).
Table 3.3 Saturated hydraulic conductivity (K\text{sat}) values for pine bark (\textit{Pinus palustris} Mill.) aged 0, 3, 6, 9, and 12 months.\textsuperscript{z}

<table>
<thead>
<tr>
<th>Age</th>
<th>All data\textsuperscript{y}</th>
<th>IQR\textsuperscript{x}</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>111.7a\textsuperscript{w}</td>
<td>119.3a</td>
</tr>
<tr>
<td>3</td>
<td>91.4a</td>
<td>90.9ab</td>
</tr>
<tr>
<td>6</td>
<td>82.5a</td>
<td>80.4b</td>
</tr>
<tr>
<td>9</td>
<td>74.1a</td>
<td>73.5b</td>
</tr>
<tr>
<td>12</td>
<td>55.6a</td>
<td>54.9c</td>
</tr>
</tbody>
</table>

\textsuperscript{z}K\text{sat} values determined using the methods of Drzal (1994).
\textsuperscript{y}Means separation with 12 data points
\textsuperscript{x}Means separation using data from within the inter-quartile range
\textsuperscript{w}Means separation down column for individual properties using Tukey’s HSD with \( \alpha = 0.05 \).
Table 3.4. Physical and hydrologic properties of pine bark (*Pinus palustris* Mill.) at 0, 3, 6, 9, and 12 months of aging.\(^z\)

<table>
<thead>
<tr>
<th>Age</th>
<th>TP(^y)</th>
<th>CC(^x)</th>
<th>AS(^w)</th>
<th>BD(^v)</th>
<th>EAW(^u)</th>
<th>WBC(^t)</th>
<th>RW10(^s)</th>
<th>RW30(^r)</th>
<th>UW(^p)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>77.9ab</td>
<td>40.3b</td>
<td>37.6ab</td>
<td>0.18c</td>
<td>3.1a</td>
<td>0.5b</td>
<td>30.3b</td>
<td>30.0ab</td>
<td>6.90a</td>
</tr>
<tr>
<td>3</td>
<td>83.5ab</td>
<td>43.8b</td>
<td>39.7a</td>
<td>0.19bc</td>
<td>6.9a</td>
<td>1.6a</td>
<td>31.9a</td>
<td>31.2a</td>
<td>6.90a</td>
</tr>
<tr>
<td>6</td>
<td>83.4ab</td>
<td>52.5a</td>
<td>30.9bc</td>
<td>0.20ab</td>
<td>3.6a</td>
<td>0.9ab</td>
<td>30.5ab</td>
<td>29.7ab</td>
<td>6.86a</td>
</tr>
<tr>
<td>9</td>
<td>85.4a</td>
<td>54.1a</td>
<td>31.3bc</td>
<td>0.21a</td>
<td>4.6a</td>
<td>0.9ab</td>
<td>29.7b</td>
<td>28.7bc</td>
<td>7.20a</td>
</tr>
<tr>
<td>12</td>
<td>82.2ab</td>
<td>55.5a</td>
<td>26.8c</td>
<td>0.21a</td>
<td>2.7a</td>
<td>0.9ab</td>
<td>29.1b</td>
<td>28.3c</td>
<td>8.50a</td>
</tr>
</tbody>
</table>

\(^z\)Physical and hydrologic properties determined using the methods of Fonteno and Harden (2010).

\(^y\)TP = total porosity; total percentage of pore volume (TP = CC + AS).

\(^x\)CC = container capacity; maximum water content after free (gravitational) drainage.

\(^w\)AS = air space; total percentage of pore space not filled with water at CC.

\(^v\)BD = bulk density; substrate dry weight/total sample volume.

\(^u\)EAW = easily available water; water lost between -1 kPa and -5 kPa.

\(^t\)WBC = water-buffering capacity; water lost between -5 kPa and -10 kPa.

\(^s\)RW10 = residual water (volumetric water content) of sample at -10 kPa.

\(^r\)RW30 = residual water (volumetric water content) of sample at -30 kPa.

\(^p\)UW = unavailable water; residual water (volumetric water content) of sample at -150kPa.

\(^o\)Means separation down column for individual properties using Tukey’s HSD with \(\alpha = 0.05\).
Table 3.5. Physical properties of pine bark aged 0, 3, 6, 9, 12 months, derived from modeling equilibrium capacity variable values.  

<table>
<thead>
<tr>
<th>Age</th>
<th>Container size</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>#1&lt;sup&gt;y&lt;/sup&gt;</td>
</tr>
<tr>
<td></td>
<td>CC&lt;sup&gt;q&lt;/sup&gt;</td>
</tr>
<tr>
<td>0</td>
<td>36.8</td>
</tr>
<tr>
<td>3</td>
<td>38.5</td>
</tr>
<tr>
<td>6</td>
<td>41.5</td>
</tr>
<tr>
<td>9</td>
<td>41.8</td>
</tr>
<tr>
<td>12</td>
<td>36.3</td>
</tr>
</tbody>
</table>

<sup>q</sup>Properties derived by modeling Van Genuchten curve fitting parameters from moisture retention curves.
<sup>y</sup>Trade 1-gallon container, commonly used in nursery production (3.9 L).
<sup>x</sup>Trade 2-gallon container, commonly used in nursery production (7.7 L).
<sup>z</sup>Trade 3-gallon container, commonly used in nursery production (11.4 L).
<sup>v</sup>Trade 5-gallon container, commonly used in nursery production (14.3 L).
<sup>t</sup>Trade 7-gallon container, commonly used in nursery production (28.3 L).
<sup>f</sup>Trade 10-gallon container, commonly used in nursery production (39.1 L).
<sup>s</sup>Trade 15-gallon container, commonly used in nursery production (51.9 L).
<sup>r</sup>Trade 25-gallon container, commonly used in nursery production (103.0 L).
<sup>Container capacity.</sup>
<sup>p</sup>Air space.
Table 3.6. Sand content of pine bark at 0, 3, 6, 9, and 12 months of aging.\textsuperscript{z}

<table>
<thead>
<tr>
<th>Age (months)</th>
<th>% Volume\textsuperscript{y}</th>
<th>% Weight\textsuperscript{x}</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>0.79c\textsuperscript{w}</td>
<td>5.05c</td>
</tr>
<tr>
<td>3</td>
<td>1.31bc</td>
<td>8.52bc</td>
</tr>
<tr>
<td>6</td>
<td>1.75b</td>
<td>11.29b</td>
</tr>
<tr>
<td>9</td>
<td>2.70a</td>
<td>16.10a</td>
</tr>
<tr>
<td>12</td>
<td>2.64a</td>
<td>15.90a</td>
</tr>
</tbody>
</table>

\textsuperscript{z}Sand content data were collected from three 100g dry samples from a composite sample from all pile replications per age (3 samples total per age).

\textsuperscript{y}Percent sand by volume per 100g dry PB

\textsuperscript{x}Percent sand by weight per 100g dry PB

\textsuperscript{w}Means separated within column by Tukey’s HSD with $\alpha \leq 0.05$. Means followed by the same letter are not significantly different.
Figure 3.1. Saturated hydraulic conductivity ($K_{sat}$) unit of Drzal (1994). Four 347.5 mL aluminum cores are fitted with individual funnel adaptor rings on the bottom and a plastic cylinder with a spout on the top. Each core assembly is secured onto an individual stationary funnel. A constant head reservoir, mounted to laboratory jacks, is filled with water and maintained at a constant level by a recirculation system. Rubber tubing is connected from the reservoir to the bottom of the stationary funnel, allowing water to saturate each core unit from the bottom. A hydraulic head is created by raising the reservoir a measured distance above the sample. Piezometers mounted to the reservoir and each core assembly are used to precisely measure hydraulic head differences.
Figure 3.2. Hydration efficiency curves of pine bark (*Pinus palustris* Mill.) over twelve months of aging at 50% and 25% moisture content by weight, with container capacity for each moisture content represented as solid lines.
Volumetric water content (%) vs Hydration events for different months:

- **0 months**:
  - 50% MC
  - 25% MC

- **2 months**:
  - 50% MC
  - 25% MC

- **3 months**:
  - 50% MC
  - 25% MC

- **4 months**:
  - 50% MC
  - 25% MC

- **5 months**:
  - 50% MC
  - 25% MC

- **6 months**:
  - 50% MC
  - 25% MC
Figure 3.3. Saturated hydraulic conductivity ($K_{sat}$) of pine bark (*Pinus palustris* Mill.) aged 0, 3, 6, 9, and 12 months. Data taken from within the interquartile range of the data set to reduce unrepresentative values.
Figure 3.4. Moisture retention curves and Van Genuchten models (log scale) of pine bark (*Pinus palustris* Mill.) at 0, 3, 6, 9, and 12 months of aging.
Month 0

Van Genuchten model
VPPE values

Month 3

Van Genuchten model
VPPE values

Month 6

Van Genuchten model
VPPE values
Figure 3.5. Pore volume fraction analysis for pine bark (*Pinus palustris* Mill.) after 0, 3, 6, 9, and 12 months of aging.
CHAPTER 4

Chemical Properties
Abstract. Pine bark (PB) is one of the most commonly used horticultural substrates, and may be used as a fresh, aged, or composted product. Aged PB is most commonly used; however, interest in and use of fresh PB is growing due to its lighter weight, resulting in lower transportation costs, and possible greater consistency in materials from batch to batch when compared to aged PB. While both fresh and aged PB may be used successfully in container production, there is little research conducted that investigates and quantifies the differences in bark of different ages over a monitored aging process. To better understand the effects of aging and pile management on PB substrates, a twelve-month study was conducted. Fresh PB was processed and the fines (less than or equal to 1.27 cm) were placed in three pile replications of 191 cubic meters each. Beginning at time 0 and every 4 to 5 weeks for a period of twelve months, piles were turned, and one week after turning stratified subsamples were taken from each pile at three different heights (top, middle, and bottom) at horizontal depths of 0.3-1.2 m to account for variations in pile depth and height. The stratified subsamples were mixed into one composite sample for each pile replication to test the chemical properties of the bark. Chemical properties included pH, EC, pH buffering capacity, CEC, AEC, plant available nutrients, C:N ratio, lignocellulose content, and seedling germination tests. With the conclusion of this study, the data generated can hopefully improve the understanding, product consistency and usability of PB substrates of different ages.
**Introduction**

Although much research has been conducted on irrigation and fertilization of substrates, little research exists regarding their chemical properties. Understanding chemical properties is important in terms of fertilization and nutrient retention. The main factor that distinguishes fertilization management of substrates used for container grown plants is the limited volume of the substrate in the container, which may result in a lower pH buffering capacity, and limited nutrient reserves (Raviv et al., 2008).

When discussing the pH of a substrate, it is the pH of the solution around the substrate that is being referred to, because the H\(^+\) protons are balanced with those fixed by the CEC. pH is very important for assimilation of major nutritional elements (Lemaire, 1995). Fresh PB is highly acidic, with a pH range of 4.1 to 5.0 (Pokorny, 1979). Several sources disagree whether the pH goes up or down slightly with decomposition (Krewer and Ruter, 2012), which may be due to the fact that pH may decrease if the piles have gone under anaerobic conditions. Harrelson et al. (2004) found no changes in substrate pH based on age.

Cation exchange capacity (CEC) is a term used to describe the attraction of cations in a substrate to negatively charged sites on substrate particles, and indicates the ability of a substrate to retain the mineral elements supplied by fertilization. Many nutrients required by plants are positively charged, and are attracted by these negatively charged sites (Lemaire, 1995). The CEC of horticultural substrates, which are composed primarily of organic matter, is highly pH dependent (Bunt, 1988). Most substrates possess negative permanently and/or temporarily charged surfaces, and surface charge properties of substrates have a great effect on the chemical reactions taking place in the rhizosphere, and on the availability and uptake efficiency of applied
Cations in the substrate solution such as will be attracted to the surface of PB particles and exchanged with other cations. The CEC of PB largely indicates the potential amounts of exchangeable divalent ions (Ca$^{2+}$ and Mg$^{2+}$); most of the monovalent cations such as NH$_4^+$ and K$^+$ are water soluble (Bunt, 1988). High CEC increases pH buffering capacity, which prevents wide variation in pH and availability of nutrients. Anion exchange capacity (AEC) of PB is poor (Buamscha et al. 2007). Pine bark has little ability to hold anions such as nitrates, phosphates or sulfates. Phosphates may be retained well in soils, but in PB they leach out rapidly (Krewer and Ruter, 2012).

Limestone is incorporated into substrates to neutralize the acidity of the substrate solution, increase pH buffering capacity, and provide additional Ca and Mg. When dolomitic lime is applied to acid organic substrates, H$^+$ is displaced from the organic functional groups (R-COO-H) and replaced with Ca and Mg. Dolomitic lime increases PB substrate pH up to a certain point, usually around 6.5, after which additional lime has little or no measurable impact. This is due to the solubility of dolomite – carbonate ions are weak bases, and have a very low solubility as pH increases above 6.5, after which very little change will occur, even if additional dolomitic lime is added (Altland and Jeong, 2016). Lime also enhances microbial activity within the media (Tucker, 1995).

The elemental composition of PB varies according to tree species, age, ecology, soil type, and season; but the concentration of elements in bark from the same tree species and geographic region is fairly stable (Van Schoor, 1990). Pokorny (1979) reported that milled PB is low in both macro and micro nutrients needed for plant growth, and a fertilization program providing adequate nutrients is needed to achieve optimum plant growth. Bark itself makes little
contribution to the nutrition of the plant being grown in a bark mix, but Ca, K, Mn, and Fe might be present in beneficial qualities, depending on the soil type in which the harvested tree originated (Aaron, 1991). Ogden et al. (1987) found that the average water-extractable micronutrient content of PB, with the exception of Fe, is similar to Hoagland’s micronutrient solution. Niemiera (1992) found that PB supplied sufficient amounts of Fe, Zn, Cu, and Mn. Pokorny (1979) reported the presence of B, Cu, Fe, Mn, and Zn in PB. One of the concerns related to the use of aged or composted waste products for use as horticultural substrates is the question of regulated elements/heavy metals. Some trace elements are essential micronutrients for plants at low concentrations, but may be toxic at higher concentrations (Naasz et al., 2009). The US Environmental protection agency has recommendations in place for the maximum permissible levels in compost products of As, Cd, Cr, Cu, Pb, Hg, Mo, Ni, Se, and Zn (Fitzpatrick et al., 1998). To detect phytotoxic substances in materials used for substrates, germination tests have been used to investigate delay of germination of seeds like cucumber. Loblolly, slash, and longleaf pine (*Pinus taeda* L., *Pinus elliottii* Engelm., *Pinus palustris* Mill.), the prominent pine species used for PB substrates over much of the eastern US, are considered to be non-phytotoxic and may be used without aging or composting with no phytotoxic effects (Bilderback et al., 2013).

The carbon to nitrogen (C:N) ratio is frequently used to indicate biostability – the higher the C:N ratio the slower the decay and the better the stability of the substrate. However, the chemical composition of the organic matter in terms of lignin will also effect the decay rate. Lignin is more resistant to decay than hemi-cellulose and cellulose (Lemaire, 1995). For most substrate components the C:N ratio should be 30:1, however PB will not have this low of a ratio.
since most of the PB is lignin and not cellulose (Bilderback, N.D.). Most softwood barks will have a C:N ratio around 200-400:1 (Van Schoor, et al., 1990).

Most barks contain slightly more lignin than cellulose (Van Schoor et al., 1990). Lignin is considered to be more stable than cellulose and hemicellulose (Verhagen, 2007), and because of its resistance to enzymatic degradation, lignin content of PB will have little variation over the course of aging (Van Schoor et al., 1990). During aging it is the cellulose that is most easily broken down by microbes, so it is the cellulose content that determines the nitrogen requirement during composting (Van Schoor et al., 1990). Bark from large tree specimens of loblolly and longleaf pine have been reported to contain little cellulose. The composting process comprises a complex series of biological events that remove mostly cellulose (wood and cambium) and various toxins from bark and leave humic acid, lignins, and a variety of microorganisms as major end products (Hoitink and Poole, 1980).

The objective of this research was to investigate and quantify the changes in the chemical properties of PB during 12 months of managed aging, in order to better understand and utilize PB at various degrees of age and stability. Understanding the nutrient holding capacities of PB of different ages may assist growers in improving fertilizer use efficiency, especially when combined with the physical and hydrologic properties of PB substrates.

Materials and Methods

Sampling. Sampling was conducted as described in Chapter 2.

pH/EC. pH and EC were measured each month within 48 h after sampling. Three samples per composite pile sample were measured, totaling nine replications per age. pH and EC were
measured with a handheld pH/EC meter (HI 9811, Hanna Instruments, Ann Arbor, MI) using the 1:1 dilution method (Fonteno and Harden, 2010). 100 mL PB was mixed with 100 mL deionized (DI) water, stirred, and allowed to equilibrate for 20 min before measurements were taken.

**pH buffering capacity.** PB samples of 0, 3, 6, 9, and 12 mos. of age were titrated based on the parameters proposed by Barnes (2013). Equal parts material from the three pile replications were combined into one composite sample per age. Three subsamples (1.5 g equivalent dry weight) per composite sample were diluted to 50 g with DI water and were titrated with 0.1 N hydrochloric acid (HCl) or 0.1 N sodium hydroxide (NaOH) using an automatic titrator (Titralab® 856, Radiometer Analytical SAS, Villeurbanne Cedex, France) at a rate of 0.3 mL·min⁻¹. Samples were taken through two titration cycles between pH 3 and 11. The first cycle, which hydrated the PB and exposed cation exchange sites, involved increasing the sample from its native pH to a pH of 11, then decreasing to a pH of 3. The process was repeated, bringing the sample to pH 11 and then back down to 3, and this second cycle was used to generate the pH buffering curve. pH buffering capacity was determined by averaging the amount of acid or base over the points of the titration curve between pH of 5.0 and 7.0. Two sets of samples were measured – one that contained an “as-is” particle size distribution, and another where particles were screened to 2.00 mm or less.

**Nutrient analysis.** PB samples of 0, 3, 6, 9, and 12 months of age were submitted to the North Carolina Department of Agriculture and Consumer Services Agronomic Division (Raleigh, NC) for nutrient analysis. Three samples were taken from each pile replication, totaling 9 samples per age. Plant available nutrients were determined by saturated media extract and vacuum filtration. The solution was analyzed for NO₃-N by nitratehydrazine reduction (Kempers, 1988), NH₄-N
was determined by a modified Berthelot reaction (Krom, 1980), and urea concentration was determined with the diacetyl monoxime thiosemicarbazide colorimetric method (Sullivan and Havlin, 1991) with an auto-flow spectrophotometric analyzer (San++ Segmented Flow Auto-Analyzer, Skalar Instruments; Breda, Netherlands). Total concentrations of P, K, Ca, Mg, S, Fe, Mn, Zn, Cu, B, and Na were determined from the saturated media extract solution with inductively coupled plasma-optical emission spectrometry (ICP-OES; Optima 8000 PerkinElmer, Waltham, MA).

*Elemental carbon and nitrogen.* Total elemental C and N were determined by the Environmental and Agricultural Testing Service laboratory (North Carolina State University, Raleigh, NC), on three samples from each composite pile sample at 0, 3, 6, 9, and 12 months of aging (nine replications per age). Samples were ground to a particle size of <2 mm before testing. Total elemental C and N were determined by total combustion method using a CHNS/O Analyzer (2400 Series II PerkinElmer, Waltham, MA).

*Cation exchange capacity.* Effective CEC of bark samples from 0, 3, 6, 9, and 12 months of aging were measured based on modified procedures described by Gillman (1979). Three replications from each pile were analyzed, equaling nine replications per age. Initially, 10 g of bark (4 g dry weight basis) and 50 mL of DI water were mixed and allowed to saturate overnight. The mixtures were then vacuum filtered (Whatman® Qualitative #1) and placed in a flask with 50 mL of 0.1 M barium chloride (BaCl₂) and mixed on an oscillating shaker (G10 Gyrotory® Shaker; New Brunswick Scientific Co., Edison, NJ) for 2 h. 20 mL of the slurry was filtered with 0.45-μm nylon filters (25mm syringe filters; Fisher Scientific, Waltham, MA) and sent to the Environmental and Agricultural Testing Service laboratory (North Carolina State University,
Raleigh, NC) for analysis. Exchangeable cations (Na, K, Mg, Ca) and exchangeable acidity (Al, Fe and Mn) displaced by Ba\textsuperscript{2+} were measured via ICP-OES (Perkin Elmar 8000, Waltham, MA). Effective CEC was calculated as the sum of the exchangeable cations and exchangeable acidity.

*Lignin, cellulose, and hemicellulose.* Pine bark samples of 0, 3, 6, 9, and 12 months of age were submitted to Cumberland Valley Analytical Services in Hagerstown, MD for cellulose and lignin determination. 3 samples per pile replication were analyzed, totaling 9 replications per age. Lignin was determined using the neutral detergent fiber (NDF) and acid detergent fiber (ADF) methods of Goering, H.K. and P.J. Van Soest (1970).

*Heavy metal content.* Heavy metals were analyzed using a Innov-X α-4000 X-ray fluorescence analyzer. One liter of sample material was placed into a ziplock bag and analyzed at 60% moisture content. Three reps per pile replication at each age, with six measurements taken per replication, totaling 36 readings per age.

*Lime incubation.* Composite samples (all piles mixed) of PB from 0, 3, 6, 9, and 12 months of aging were amended with two different lime rates to investigate effects of PB age on lime efficacy. Nine liters of each substrate were hydrated to a mass wetness of 2 (66% moisture) and sealed in plastic bags (size, make) for 24 h to equilibrate. Initial substrate pH was determined on all substrates prior to limestone incorporation using the 1:1 extraction method. Pulverized dolomitic limestone (#100; Rockydale Quarries Corporation, Roanoke, VA) was incorporated at rates of 0, 2.37, 4.75 kg/m\textsuperscript{3}, making for a total of 15 treatments (5 substrates x 3 lime rates). Substrates were sealed in plastic bags and stored at a constant air temperature 18.3°C. On 1, 3, 5, 7, 14, and 21 days after limestone amendment (DAM), three subsamples from each substrate
treatment were collected and substrate solution pH was measured at 0, 1, 3, 5, 7, 14, 21, 28 DAM using the 1:1 extraction method. Prior to subsampling, bags were thoroughly mixed/rotated.

Toxicity tests. At each month of sampling, PB was tested for seedling toxicity using method 05.05B from the TMECC manual (Thompson, 2002). For each age, four 100 mL PB samples from each pile replication (12 total replications per age) were mixed with 200 mL deionized (DI) water, stirred, and allowed to equilibrate for 2 h. Slurries were stirred every half hour during the 2 h soaking period, and then vacuum filtered through 9 cm filter paper (Whatman® Qualitative #1). Ten cucumber seeds (*Cucumis sativus* ‘Bush Champion’) were placed on two sheets of 9 cm filter paper in a 9 cm Pyrex® petri dish, and 7 mL of the extract was dispensed onto the filter paper using a 5 mL pipettor (Fisher Scientific, Waltham, MA) so that the filter paper was saturated with PB extract, with a thin layer of extract on the surface. As a control, distilled water was used instead of PB extract in four petri dishes. The petri dishes were placed in a well-lit, controlled-temperature chamber (NCSU Horticultural Substrates Laboratory) held at 22 °C for five days, after which the number of seeds germinated per plate were counted.

At 0, 3, 6, 9, and 12 months of aging, PB was tested for toxicity using the Mulch and Soil Council test protocols for landscape soils and soil amendments, and premium potting soils (Mulch and Soil Council, 2007). The studies took place in the Marye Ann Fox Teaching Laboratories Greenhouse at North Carolina State University (Raleigh, NC). Three plant species were used: radish, ‘Early Scarlet’ (*Raphanus sativus*); marigold, ‘Janie Flame’ (*Tagetes patula*); and tomato, ‘Better Boy’ (*Lycopersicum esculentum*). For each of the ages tested, four 10.16 cm square plastic pots per species were filled with 100% PB from each of the piles, a 50:50 blend of PB from each pile and sand. Five seeds of each species were placed on the substrate surface, one near each of the four corners of the pot and one in the center, and were covered with a light layer
of substrate. The pots were grouped in trays, irrigated with tap water, and placed under a mist system (30 seconds every hour). Two weeks after sowing, seedling survival data was collected by counting the number of seedlings per pot, and pots were thinned down to one plant per pot. Four weeks after sowing, each plant was harvested, placed into a paper bag (size, company), and the bags were placed in a forced air dryer at 105°C for five days, after which dry weights were recorded.

**Statistical analysis.** Data were checked for normality and linearity assumptions, and analyzed using the mixed ANOVA in SAS. Treatment means were separated using Tukey’s honestly significant differences (HSD) test at p < 0.05 significance level. For the lime incubation study, ANOVA was conducted on all data to determine if significant differences occurred in substrate pH or EC as a result of age, lime rate, and time. Additionally, the slice option in SAS (SAS 9.3; Institute, Cary, NC) was used to determine if significant differences occurred among all substrates amended at similar rates of dolomitic limestone. Means were separated by Tukey’s least significant differences at P ≤ 0.05.

**Results and discussion**

**pH/EC.** The pH increased over the course of twelve months from 4.2 to 4.5, with decreases occurring at months 7 and 8 (Fig. 4.1). This overall trend is consistent with the findings of Pokorny (1979), who did not report a substantial rise of pH with PB age. Lemaire (1980) stated that fine PB has a higher pH than coarse PB, which could explain the increase observed in our data, as particle size distribution shifted to a higher percent of fine particles. The average pH of all months were within the range of 4.1 – 5.0, as described by Pokorny (1979), except for month
8, which had an average pH of 4.0. Lemaire (1998) reported an increase in pH over the course of six months for PB compost, although it was unclear if the PB was truly composted instead of aged. There were no differences in EC among any of the ages, with values ranging from 0.01 to 0.06.

**pH buffering capacity.** There were no differences between months 0, 3, 6, 9, and 12 in pH buffering capacity for either the screened or unscreened (2.00 mm or less) samples. During the titration procedure, hysteresis and pH drift were observed for all samples. Hysteresis is a difference between forward (increasing pH) and backward (decreasing pH) titration curves. For our samples, the backward titration curves required more equivalents of acid to reach the pH 3 endpoint than forward titration curves required equivalents of base to reach pH 11. The hysteresis was the greatest between pH 5 and 8, where carboxylic acids are the principal organic functional groups that buffer pH. pH drift is a difference between pH endpoints between replicates for a given acid or base addition. These trends in our titration data are consistent with the findings of Barnes et al. (2013) for peat moss samples.

**Cation exchange capacity.** The effective CEC of bark and the exchangeable bases increased after month 0, while exchangeable acidity remained the same. Buamscha et al. (2007) and Wright (1988) found that CEC increases with decreasing particle size. Nash and Pokorny reported 96.6 meq/L. Altland et al reported 29.9 to 74.4 meq/L. Our findings were lower than what was reported in the literature, these differences are most likely due to the experimental procedure.

**Plant available nutrients.** There was a decrease in concentrations of K, Ca, Mg, S, Fe, Mn, Zn, Cu, B, and Na. There were no changes in concentrations of Cl, N (inorganic, organic, and urea), and there was an increase in P concentrations. Micro nutrient concentrations lower than what was
reported by Niemiera (1992), however this could be due to the extraction method. Aaron 1972 (from Van Schoor)- nutrient content (mg/kg): 5 NO3 (N 0.3%), 6 P, 155 K, 36 Mg
0.2 – 0.3% nitrogen, 0.33 mg/l water-extractable NH4 and 0.67 ng/L NO3 (Pokorny 1979)

C:N ratio. The C:N ratio decreased with time. For months 0, 3, 6, 9, and 12 our values were lower than what has been reported in the literature for PB. Krewer and Ruter (2012) reported a C:N ratio of 300:1 for white pine. Van Schoor et al. (1991) reported C:N ratios of 200 – 400:1 for softwood barks.

Lignocellulose. There were no changes in lignin content, with average values ranging between 52 to 55%, which is consistent lignin content of 52% for loblolly PB that was reported by Labosky (1979). Harkin and Rowe (1971) reported lignin values of 40-55% for PB of different species. The LCI decreased over time, signifying a slower decay rate after 6 months, but there was only a difference of .07 between ages, which illustrates the slower decay rate of softwood bark. The lignin:N ratio, which is also used as a decay parameter, was the same for all ages.

Liming incubation. Initial pH values before liming were similar for all ages (Fig. 4.2). P values indicate that age, lime rate, DAM, interaction of age and DAM, and interaction of lime rate and DAM were significant. Both lime rates had the greatest efficacy on fresh PB at month 0, with no other trends observed for age and lime rate.

Heavy metal content. The heavy metals that were detected in higher than recommended concentrations than the Mulch and Soil Council standards were Pb and Zn (Table 4.4) for months 4,5,6,9,10,11, and 12. Readings were between 6.5 and 9.1 ppm for Pb, and 6.3 and 7.2 for Zn. The frequency and concentrations of heavy metal detection did not appear to increase with age, and readings were not a great deal higher than the limit of 5 ppm.
Germination assays. There were no differences in the cucumber germination assays, with all months between 99 to 100% germination. The Mulch and Soil Council assay did not show any differences in seedling survival for any of the species in either the 100% PB or 50:50 PB/Sand mix, except for tomato in 100% PB at month 0, which had a lower germination rate. Dry weights increased with age for all species in 100% pine bark, which is presumably due to an increase in the amount of fine particles, which would increase the water holding capacity. Dry weights were higher for all species in the 50:50 PB/sand mix, with no differences among ages with the bark was mixed with sand.

Summary

The results of our study show a slight increase in pH over the course of twelve months of aging, with an increase in CEC after three months of aging. There were no differences in pH buffering capacity, but lime appeared to react more quickly when applied to fresh PB at rates of 2.37 and 4.75 kg/m$^3$. There was a decrease in plant available nutrient concentrations except for P, which increased over time, with some incidence of Pb and Zn concentrations slightly over the recommended limit of 5 ppm. No seedling toxicity was observed for bark of any age, which is consistent with the finding of Pokorny (2013) who stated that seedling toxicity is not an issue with southern pine species. There was a decrease in the C:N ratio after three months of aging, and a decrease in the lignocellulose index, which indicates a lower rate of decomposition.
Literature cited


Bilderback, T. Managing container substrates. n.d.


Table 4.1. Extractable nutrients of pine bark over twelve months of aging.\textsuperscript{z}

<table>
<thead>
<tr>
<th>Age</th>
<th>P</th>
<th>K</th>
<th>Ca</th>
<th>Mg</th>
<th>S</th>
<th>Fe</th>
<th>Mn</th>
<th>Zn</th>
<th>Cu</th>
<th>B</th>
<th>Cl</th>
<th>Na</th>
<th>Inorg.N</th>
<th>Org. N</th>
<th>Urea</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>2.83c\textsuperscript{y}</td>
<td>39.23a</td>
<td>3.09a</td>
<td>1.90a</td>
<td>7.33a</td>
<td>0.32a</td>
<td>0.15a</td>
<td>0.03a</td>
<td>0.01a</td>
<td>0.16a</td>
<td>8.54abc</td>
<td>1.99a</td>
<td>0.32a</td>
<td>0.00a</td>
<td>0.01a</td>
</tr>
<tr>
<td>3</td>
<td>6.06b</td>
<td>33.20a</td>
<td>3.33ab</td>
<td>1.67a</td>
<td>3.05b</td>
<td>0.28a</td>
<td>0.16ab</td>
<td>0.04a</td>
<td>0.01a</td>
<td>0.16a</td>
<td>7.24ab</td>
<td>1.48a</td>
<td>0.44a</td>
<td>0.00a</td>
<td>0.01a</td>
</tr>
<tr>
<td>6</td>
<td>9.23a</td>
<td>22.41b</td>
<td>1.55b</td>
<td>0.68b</td>
<td>0.29c</td>
<td>0.19b</td>
<td>0.04b</td>
<td>0.02bc</td>
<td>0.00b</td>
<td>0.16a</td>
<td>8.53a</td>
<td>0.95b</td>
<td>0.65a</td>
<td>0.00a</td>
<td>0.26a</td>
</tr>
<tr>
<td>9</td>
<td>9.93a</td>
<td>19.74b</td>
<td>1.05c</td>
<td>0.47bc</td>
<td>0.18cd</td>
<td>0.08c</td>
<td>0.04b</td>
<td>0.01bc</td>
<td>0.00b</td>
<td>0.15a</td>
<td>6.47b</td>
<td>0.78b</td>
<td>0.66a</td>
<td>0.00a</td>
<td>0.10a</td>
</tr>
<tr>
<td>12</td>
<td>9.66a</td>
<td>21.64b</td>
<td>1.22c</td>
<td>0.61c</td>
<td>0.13cd</td>
<td>0.10c</td>
<td>0.06b</td>
<td>0.00c</td>
<td>0.00b</td>
<td>0.12b</td>
<td>5.07c</td>
<td>0.75b</td>
<td>2.29a</td>
<td>0.00a</td>
<td>0.00a</td>
</tr>
</tbody>
</table>

\textsuperscript{z}Determined on \textit{Pinus palustris} Mill. Samples by saturated media extract and vacuum filtration with an auto-flow spectrophotometric analyzer.

\textsuperscript{y}Means separated within column by method by Tukey’s HSD at $P \leq 0.05$. Means followed by the same letter are not significantly different (n=9).
Table 4.2. pH buffering capacity, cation exchange capacity, exchangeable cations exchangeable acidity, and anion exchange capacity of pine bark after 0, 3, 6, 9, and 12 months of aging.\textsuperscript{z}

<table>
<thead>
<tr>
<th>Age</th>
<th>pH buffering capacity\textsuperscript{y} (mmol kg\textsuperscript{-1})</th>
<th>pH buffering capacity\textsuperscript{y} \textless 2.0 mm (mmol kg\textsuperscript{-1})</th>
<th>CEC\textsuperscript{x} (mg L\textsuperscript{-1})</th>
<th>Exchangeable cations\textsuperscript{w} (mg L\textsuperscript{-1})</th>
<th>Exchangeable acidity\textsuperscript{v} (mg L\textsuperscript{-1})</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>1.98a\textsuperscript{t}</td>
<td>2.03a</td>
<td>76.72b</td>
<td>69.13b</td>
<td>7.60a</td>
</tr>
<tr>
<td>3</td>
<td>2.66a</td>
<td>1.70a</td>
<td>90.67a</td>
<td>83.33a</td>
<td>7.63a</td>
</tr>
<tr>
<td>6</td>
<td>3.21a</td>
<td>1.80a</td>
<td>91.51a</td>
<td>84.34a</td>
<td>7.17a</td>
</tr>
<tr>
<td>9</td>
<td>1.98a</td>
<td>1.58a</td>
<td>99.81a</td>
<td>91.25a</td>
<td>8.56a</td>
</tr>
<tr>
<td>12</td>
<td>1.64a</td>
<td>1.58a</td>
<td>92.40a</td>
<td>83.99a</td>
<td>8.37a</td>
</tr>
</tbody>
</table>

\textsuperscript{z}Pinus pallestris Mill.
\textsuperscript{y}pH buffering capacity measured by titration and averaged over the points of the titration curve between pH of 5.0 and 7.0.
\textsuperscript{x}Determined from BaCl extractions based on the methods of Gillman (1979)
\textsuperscript{w}Sum of Ca, K, Mg, and
\textsuperscript{v}Sum of Fe, Al and Mn ions
\textsuperscript{t}Determined from BaCl extractions based on the methods of Gillman (1979)
\textsuperscript{a}Means separated within column by Tukey’s HSD at $P \leq 0.05$. Means followed by the same letter are not significantly different (n=12).
Table 4.3. Elemental carbon, nitrogen, and lignocellulose ratios of pine bark over 0, 3, 6, 9, and 12 months of aging.\(^z\)

<table>
<thead>
<tr>
<th>Age</th>
<th>C(^y)</th>
<th>N</th>
<th>C:N</th>
<th>Lignin(^x)</th>
<th>Cellulose</th>
<th>Hemicellulose</th>
<th>Lignocellulose index(^v)</th>
<th>Lignin:N</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>48.69a(^u)</td>
<td>0.25a</td>
<td>194.19a</td>
<td>52.14c</td>
<td>27.72d</td>
<td>1.99b</td>
<td>0.64a</td>
<td>207.96a</td>
</tr>
<tr>
<td>3</td>
<td>47.21a</td>
<td>0.31a</td>
<td>155.48b</td>
<td>55.86a</td>
<td>31.20cd</td>
<td>3.48ab</td>
<td>0.62a</td>
<td>186.83a</td>
</tr>
<tr>
<td>6</td>
<td>43.69ab</td>
<td>0.26a</td>
<td>165.28b</td>
<td>55.29ab</td>
<td>32.97bc</td>
<td>4.82a</td>
<td>0.59ab</td>
<td>211.12a</td>
</tr>
<tr>
<td>9</td>
<td>43.47ab</td>
<td>0.29a</td>
<td>152.25b</td>
<td>54.25abc</td>
<td>35.90ab</td>
<td>3.40ab</td>
<td>0.58b</td>
<td>197.43a</td>
</tr>
<tr>
<td>12</td>
<td>38.56b</td>
<td>0.25a</td>
<td>154.89b</td>
<td>53.06bc</td>
<td>36.88a</td>
<td>2.87b</td>
<td>0.57b</td>
<td>215.86a</td>
</tr>
</tbody>
</table>

\(^z\)Pinus pallustris Mill.
\(^y\)Total C and N determined by total combustion method and CHNS/O Analyzer (2400 Series II PerkinElmer, Waltham, MA).
\(^x\)Lignin, cellulose and hemicellulose determined using the neutral detergent fiber (NDF) and acid detergent fiber (ADF) methods of Goering, H.K. and P.J. Van Soest (1970).
\(^v\)Determined by dividing lignin content by the sum of the lignin, cellulose, and hemicellulose content
\(^u\)Determined by loss on ignition
\(^a\)Means separated within column by Tukey’s HSD at \(P \leq 0.05\). Means followed by the same letter are not significantly different (n=12).
Table 4.4. Heavy metal detection of pine bark (*Pinus palustris* Mill.) over twelve months of aging. *

<table>
<thead>
<tr>
<th>Age</th>
<th>Cu (ppm)</th>
<th>Cr (ppm)</th>
<th>As (ppm)</th>
<th>Pb (&gt; 5ppm)</th>
<th>Zn (&gt; 5ppm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>ND</td>
<td>ND</td>
<td>ND</td>
<td>ND</td>
<td>ND</td>
</tr>
<tr>
<td>2</td>
<td>ND</td>
<td>ND</td>
<td>ND</td>
<td>ND</td>
<td>ND</td>
</tr>
<tr>
<td>3</td>
<td>ND</td>
<td>ND</td>
<td>ND</td>
<td>ND</td>
<td>ND</td>
</tr>
<tr>
<td>4</td>
<td>ND</td>
<td>ND</td>
<td>ND</td>
<td>8.5 x</td>
<td>6.9</td>
</tr>
<tr>
<td>5</td>
<td>ND</td>
<td>ND</td>
<td>ND</td>
<td>6.5</td>
<td>6.3</td>
</tr>
<tr>
<td>6</td>
<td>ND</td>
<td>ND</td>
<td>ND</td>
<td>6.8</td>
<td>6.5</td>
</tr>
<tr>
<td>7</td>
<td>ND</td>
<td>ND</td>
<td>ND</td>
<td>ND</td>
<td>ND</td>
</tr>
<tr>
<td>8</td>
<td>ND</td>
<td>ND</td>
<td>ND</td>
<td>ND</td>
<td>ND</td>
</tr>
<tr>
<td>9</td>
<td>ND</td>
<td>ND</td>
<td>ND</td>
<td>7.2</td>
<td>7.2</td>
</tr>
<tr>
<td>10</td>
<td>ND</td>
<td>ND</td>
<td>ND</td>
<td>9.1</td>
<td>6.7</td>
</tr>
<tr>
<td>11</td>
<td>ND</td>
<td>ND</td>
<td>ND</td>
<td>8.9</td>
<td>6.8</td>
</tr>
<tr>
<td>12</td>
<td>ND</td>
<td>ND</td>
<td>ND</td>
<td>6.7</td>
<td>6.3</td>
</tr>
</tbody>
</table>

* Heavy metals were analyzed using a Innov-X α-4000 X-ray fluorescence analyzer. One liter of sample material was placed into a ziplock bag and analyzed at 60% moisture content. Three reps per pile replication at each age, with six measurements taken per replication, totaling 36 readings per age.

y ND = no detection

x Average of all readings for age
<table>
<thead>
<tr>
<th>Age</th>
<th>Pine bark&lt;sup&gt;x&lt;/sup&gt;</th>
<th>Control&lt;sup&gt;w&lt;/sup&gt;</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>99&lt;sup&gt;y&lt;/sup&gt;</td>
<td>100a</td>
</tr>
<tr>
<td>2</td>
<td>100a</td>
<td>99a</td>
</tr>
<tr>
<td>3</td>
<td>100a</td>
<td>99a</td>
</tr>
<tr>
<td>4</td>
<td>100a</td>
<td>100a</td>
</tr>
<tr>
<td>5</td>
<td>100a</td>
<td>100a</td>
</tr>
<tr>
<td>6</td>
<td>100a</td>
<td>100a</td>
</tr>
<tr>
<td>7</td>
<td>99a</td>
<td>99a</td>
</tr>
<tr>
<td>8</td>
<td>100a</td>
<td>100a</td>
</tr>
<tr>
<td>9</td>
<td>100a</td>
<td>100a</td>
</tr>
<tr>
<td>10</td>
<td>100a</td>
<td>100a</td>
</tr>
<tr>
<td>11</td>
<td>100a</td>
<td>99a</td>
</tr>
<tr>
<td>12</td>
<td>100a</td>
<td>100a</td>
</tr>
</tbody>
</table>

<sup>x</sup>100 mL PB samples were mixed with 200 mL deionized water, stirred, allowed to equilibrate for 2 h, and then vacuum filtered through 9 cm filter paper (Whatman® Qualitative #1).

<sup>y</sup>Percent cucumber seeds (*Cucumis sativus* ‘Bush Champion’) germinated out of 10 that were sown.

<sup>z</sup>*Pinus palustris* Mill. extracts

<sup>w</sup>Deionized water

<sup>v</sup>Means separated within column by Tukey’s HSD at *P* ≤ 0.05. Means followed by the same letter are not significantly different (n=9).
Table 4.6. Seedling germination and dry weights of three species grown in pine bark after 0, 3, 6, 9, and 12 months of aging.\textsuperscript{z}

<table>
<thead>
<tr>
<th>Age</th>
<th>100% Pine bark</th>
<th>50:50 Pine bark/sand</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Radish\textsuperscript{x}</td>
<td>Marigold\textsuperscript{w}</td>
</tr>
<tr>
<td></td>
<td>Survival\textsuperscript{a} (%)</td>
<td>Weight\textsuperscript{t} (mg)</td>
</tr>
<tr>
<td>0</td>
<td>86.7ab\textsuperscript{a}</td>
<td>2.00b</td>
</tr>
<tr>
<td>3</td>
<td>95.0ab</td>
<td>6.75a</td>
</tr>
<tr>
<td>6</td>
<td>96.7a</td>
<td>9.17a</td>
</tr>
<tr>
<td>9</td>
<td>71.7b</td>
<td>9.58a</td>
</tr>
<tr>
<td>12</td>
<td>95.0ab</td>
<td>11.42a</td>
</tr>
</tbody>
</table>

\textsuperscript{a}Germination test based on the procedures outlined by the Mulch and Soil Council for premium potting mixes and soil and landscape amendments.

\textsuperscript{b}For each of the ages tested, four 10.16 cm square plastic pots per species were filled with 100\% pine bark (\textit{Pinus palustris} Mill.) from each of the piles, a 50:50 blend of pine bark from each pile and sand.

\textsuperscript{c}\textit{Raphanus sativus} ‘Early Scarlet’

\textsuperscript{d}\textit{Tagetes patula} ‘Janie Flame’.

\textsuperscript{e}\textit{Lycopersicum esculentum} ‘Better Boy’

\textsuperscript{f}Average percent of seeds germinated out of five that were sown.

\textsuperscript{g}Dry weight of seedlings after being placed in a forced air dryer at 105\degree C for five days.

\textsuperscript{h}Means separated within column by Tukey’s HSD at \( P \leq 0.05 \). Means followed by the same letter are not significantly different (n=12).
Figure 3. pH and EC of pine bark samples over twelve months of aging. Three samples were measured from each pile with a handheld pH meter (HI 9811, Hanna Instruments, Ann Arbor, MI) using the 1:1 dilution method (Fonteno and Harden, 2010).
Figure 4. Composite pine bark samples from combined piles at 0, 3, 6, 9, and 12 months of aging were amended with pulverized dolomitic limestone (#100; Rockydale Quarries Corporation, Roanoke, VA) at rates of 0, 2.37, 4.75 kg/m³ (0, 4, and 8 lbs./yd³. Subsamples were taken each treatment on 1, 3, 5, 7, 14, and 21 days and substrate solution pH was measured at 0, 1, 3, 5, 7, 14, 21, 28 days using the 1:1 extraction method and a handheld pH meter (HI 9811, Hanna Instruments, Ann Arbor, MI).
Appendix A. Processing of pine bark. Raw pine bark (A) is passed through a hammer-mill (B), and screened into three fractions: nuggets (C), mini-nuggets (D), and fines (less than or equal to 1.27 cm).
Appendix B. A drone with GPS mapping technology was used to create a site map using DroneDeploy 3-D mapping software (A). This 3-D map can be used to calculate the volume of inventory piles.
Appendix C. Thermal imaging was used as potential technique to measure pile surface temperature (A), inner pile temperature during select sampling dates (B), and during turning on 13 Nov. 2015 (C).
Appendix D. Channeling and uneven moisture distribution of PB at 25% moisture content by weight after 10 hydration events (right) as compared to 50% moisture content by weight (left).