UTILIZATION OF PECAN SHELLS IN SMOKED CHICKEN PRODUCTS

by

WENQIAN FU

(Under the Direction of WILLIAM L. KERR)

ABSTRACT

This thesis explores the use of pecan shells as a low-cost alternative to pecan or other woods as a source of smoke flavors in chicken breast meat. Several physical properties related to quality were measured including moisture content, water activity, color, cook loss, water-holding capacity and shear force for chicken products smoked with four different varieties of sawdust (pecan shell, hickory, mesquite and apple tree wood). Sensory studies were carried out using descriptive and consumer evaluation. The phenolic compounds responsible for smoky flavor in the smoked chicken breasts were determined by SPME in combination with GC-MS. Of the physical properties, only the moisture content, pH and color were affected by wood species ($p<0.05$). Pecan shell smoked chicken was slightly darker than other samples ($L^*=72.86$), with a slightly redder color ($a^*= 5.71$, $b^*=25.70$). Based on descriptive sensory studies, only 'smoky' and 'hardness' attributes differed amongst the samples. Hickory smoked chicken breasts had the strongest smoky flavor (5.28), while the smoky flavor in both pecan shell (4.29) and apple tree wood (4.52) smoked chicken breasts was very similar. Consumer tests showed that pecan shell smoked chicken had scores near "like moderately" (6.20) for overall likeability. The species of woods had an influence on the concentration of phenolic compounds responsible for smoky flavor ($p<0.05$). The overall level of phenolic compounds related to smoky flavor was greatest in

hickory-smoked chicken (0.96 ppm) while pecan shell smoked chicken contained 0.44 ng phenolics/mg. In summary, chicken breast smoked with pecan shells had good acceptability and properties similar to chicken smoked with other woods.

INDEX WORDS: pecan shells, smoked meat, descriptive sensory analysis, consumer evaluation, phenolic compounds, solid phase microextraction, gas chromatography-mass spectrometry

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CHAPTER 1

INTRODUCTION

Pecan shell is a waste product resulting from the pecan shelling process. It is estimated that 52,416 tons of pecan shells were generated in 2012 from shelling operations in the United States (NASS 2015). Generally, this material is sent to landfills, although there has been some work in developing pecan shells as a source of biofuels. For many years, people in southeastern U.S. have used pecan *wood* for smoking or grilling meat. There has also been some anecdotal mentions of using pecan *shells* as a source of smoke. Thus, in this thesis we explore the possibility of using pecan shells as an economic alternative to pecan or other woods as a source of smoke flavors in products.

We examined the use of pecan shells for producing smoked chicken breasts. Chicken breast was chosen as it has a relatively mild flavor profile and might better highlight the impact of added smoke flavors. To process the products, the shells were ground to pieces that would suit for burning and smoke production. The smoked products were compared with those produced from other common sources including hickory, mesquite and apple tree wood. The basic properties of the shells or wood were examined including moisture content, ash and particle size. The basic physical properties of the meats after smoking were also determined including moisture content, pH, water activity, yield, water-holding capacity, color and textural attributes. Finally, we also studied the sensory attributes of the smoked meats, including the descriptive analysis of key attributes of the different smoked meats, as well as a consumer evaluation of the likeability of select products. In conjunction with these studies, samples were analyzed by GC-

MS to determined how selected aromatic compounds related to smoke flavor differed in the various samples.

The thesis consists of five chapters. The first introductory chapter outlines the purpose and scope of the work. The second chapter reviews the relevant literature related to the project background, chemical and physical analyses of smoke-producing sawdust, the smoking process, physical and chemical properties related to meat quality, sensory analyses of meat products, and determination of phenolic and other aromatic compounds in smoked meats. The third chapter focuses on the effects of pecan shell smoke on quality parameters of smoked chicken products including moisture content, water activity, color, yield loss, water-holding capacity and instrumental texture analysis, as compared with chicken breasts smoked with three other sawdust materials (hickory, mesquite and apple tree wood). Some basic analysis of the smoke sources were measured, including moisture content, ash content, and particle size distribution of these four varieties of smoke sources. The fourth chapter examines the comprehensive sensory properties of the smoked chicken breasts prepared from the four smoke sources. The sensory studies included both descriptive sensory and consumer evaluation study. The former investigates the influence of the different woods on the flavor and texture of smoked chicken, using trained panelists to describe the flavor and texture attributes of each sample, and to find correlations between texture sensory profiles with water-holding capacities and instrumental texture analysis (TPA). In this study, the Spectrum ^{TM} descriptive method based on 15-point numerical scales with references (where 0= "not detected" and 15 = "extremely strong") was performed to detect five flavor attributes (sweetness, salty, smoky, woody and earthy flavor) and four texture attributes (cohesiveness, hardness, juiciness and chewiness). We successfully developed reference compounds and intensity specifications for smoked chicken products. The

consumer evaluation allowed untrained panelists to determine acceptability for overall liking, flavor, texture and appearance of pecan shell smoked chicken products. In addition, this study also established a headspace sampling procedure, using SPME fiber and gas chromatography-mass spectrometry (GC-MS) for analysis of phenolic compounds related to smoky flavor, including guaiacol, 4-methylguaiacol, 4-ethylguaiacol and 2,6-dimethoxyphenol. The final chapter summarizes the conclusions of the entire study and makes recommendations for future work.

CHAPTER 2

LITERATURE REVIEW

2.1 Pecan Production

Pecan [*Carya illinoinensis* (Wangenh.) K. Koch] is a tree nut crop cultivated in the southern and eastern areas of the Unites States, Mexico, Israel and Australia. In 2014-2015, the USA and Mexico lead the world's production of pecans, growing over 93% of world's crop with totals of 60,185 and 40,823 metric tons, respectively. The next largest suppliers of pecans were South Africa (5,724 metric tons) and Australia (1,080 metric tons). Global production of pecans totaled more than 108,000 metric tons (kernel basis), which represents a 59% increase over the last ten years (INC 2015).

Pecans are the only native tree nut grown for commercial use in the United States, and are considered the most important tree nut crop due to their export potential (Wood et al. 1994). In 2013, pecan exports from the U.S. totaled almost 35,500 metric tons. This was 2% less than in 2012, but up 7% compared to 2004 values. Mexico and the U.S. were also the greatest exporters of pecans, accounting for 97% of all pecans exported in 2013. Interestingly, the U.S. was the main destination for Mexico's exports, while Canada was the principal destination for the United States 's exports (INC 2015).

Pecan varieties are generally classified as one of two types: improved and native/seedling varieties. As indicated by Hubbard (1987), Georgia has had a comparative advantage in pecan production because growers have been replacing traditional varieties with new improved ones. These new varieties produce a more consistent crop with desirable characteristics enhanced

through genetic manipulation, including size of kernel, color and resistance to disease or pests. New varieties can also be tailored for particular markets, such as large kernels targeted for the gift trade industry.

Most pecan tree varieties need approximately 205-233 frost-free days to reach maturity. Thus, the southern United States has become the most important region in commercial pecan tree growing. According to NASS (2015) data, nearly three-fourths of U.S. pecans are produced in Georgia, New Mexico and Texas. Of those, Georgia remains the top producer with production of all pecans (improved varieties, native/seedling in shell basis) reaching 100 million pounds from 2012 to 2014. Pecans are sold as an agriculture commodity either on an in-shell or shelled basis. They are usually divided into various grades using the following criteria: nut size, meat size, color of the meat and shelling ratio (SR) of the nut. Xu (2000) described the SR as the weight of the strands in the face divided by the total weight, with SR ranging from 0.0 to 1.0.

There are over 500 varieties of pecans existing today, however only three cultivars are regarded as standard in the U.S. pecan growing industry. ‘Stuart’, ‘Desirable’ and ‘Schley’ are most commonly planted in Georgia orchards, as they have been found to be productive over a wide range of growing conditions and produce high-quality nuts when the trees mature. The harvest season for pecan nuts is between mid-October through December. Once harvesting begins, a hydraulic shaker grips the trunk of the tree to dislodge nuts; sometimes a second shaking operation is needed to dislodge nuts of certain cultivars. The nuts are mechanically swept into rows called windrows, where a mechanical harvester will eventually pick them up.

2.2 Pecan Processing

Some nuts are kept in the shell to extend storage life. Others are shelled for immediate use. The hulls are separated from nuts at a hulling facility. In large-scale operations the nuts are cracked, the shells removed and the nuts sized in a rapid manner through a sequence of unit operations. As a first step the pecans are weighed, then sent through a cracking machine. These often consist of a series of grooved rollers with successively smaller spaces. The nuts drop to a conveyor that carries them to the sheller, which removes the shells. The shells may be removed by floating in a different density fluid or even by machine vision techniques. The pecans may be further treated in an air cleaner to remove as much shell and unwanted debris as possible.

Typically, about 20-25% of the initial weight is lost as loose shell. After the nuts have been cracked and stripped of shells, the remaining pecan nuts are passed over sieves with holes with progressively larger size. These sort the nuts according to size so that they can be classified as small, large, jumbo or chipped. In addition, size standards exist for half or whole pieces. The nuts are typically dried to a level of 4.5g/100g kernel moisture at 24 °C, with relative humidity below 60% and moderate airflow. The pecan pieces may be roasted in hot ovens or oil. The drying and roasting helps improve the appearance, aroma, flavor and texture of the nuts. Raw pecans may be treated with propylene oxide gas to pasteurize the nutmeat surface. The processed and graded nuts are then sent for packaging in plastic bags, paperboard cartons or glass jars.

2.3 Pecan Shells

It is estimated that 52,416 tons of pecan shells were produced in 2014 from shelling operations in the United States (NASS 2015). These are normally considered as waste products. In one application, the material may be sold to gardeners or landscapers for use as mulch or

decorative ground covering. Pecan shells can give the landscape a nice, earthy color and interesting texture. It also increases soil acidity, an advantage for acid-loving vegetation such as radishes, potatoes, and peppers (Brison 1974). However, recurrent use of pecan shells in soil will destroy plants that are less tolerant of low soil pH.

Several researchers and industries have begun to investigate the potential value and uses for pecan shells. For example, Babu et al. (2013) found that extracts prepared from pecan shells served as a natural antimicrobial, with demonstrated effect against *L. monocytogenes* and other *Listeria* spp. Pecan shells have also played an emerging role as a cheap energy source in the United States. For example, one of Proctor and Gamble's largest paper manufacturing plants in Georgia will soon burn pecan shells, along with other waste wood products (such as mill waste, discarded treetops and scrap wood) to create steam for it's plant in Albany, GA (Brunsman 2015). The company believes that the combined heat-and-power biomass unit would provide 100 percent of the steam, and up to 70 percent of the total electricity, needed for manufacture in its leading towel and tissue facility. In addition, pecan shells have been utilized in a variety of other applications, such as a source of activated carbon for water filtration (Bansode et al. 2003), as a sugar-refining agent (Rao et al. 2000), and for geosmin adsorption (Ng et al. 2002). To date, no known studies have been reported on the use of pecan shells for direct food use.

2.4 Physical Characteristics and Composition of Sawdust

Various woods have been used to impart desirable characteristics to foods subject to the smoking process. For this discussion, we include the use of nut shells as a potential smoke generator, as it is dry biomass that contains significant levels of cellulose, hemicellulose and lignin. In addition to imparting a unique and pleasing flavor, smoking helps retain natural juices

in meat products by contributing to a sealed surface during the cooking process. Burning of the wood creates numerous chemical compounds derived from cellulose, hemicellulose, and lignin pyrolysis (Guillén et al. 2004). Fengel and Wegener (1983) proposed that furan derivatives and various lactones formed during the degradation of hemicellulose contribute to the overall flavor and chemical properties of smoke. In addition, several reactive aldehydes result from cellulose degradation and are the primary color-forming reactants. Several phenolic compounds, the major contributors to smoky flavor, derive from lignin pyrolysis (Shahidi 1994).

The production of high-quality smoked foods is a delicate balance of art and technology. Obviously, the type of wood is important to the final flavor and properties of smoked foods, as well as how long and at what temperature the material is burned. While proper smoking can improve preservation, flavor and color of the food, improper smoking conditions can create undesirable and even toxic compounds, including polycyclic aromatic hydrocarbons (PAH). As noted above, one major determinant of the properties of smoke are the relative proportions of cellulose, hemicellulose and lignin. In addition, the moisture and ash content play a role in how the material burns and generates smoke compounds. Another factor, which determines how fast the wood burns and releases smoke compounds, is the particle size of the pieces. While whole pieces of wood (or shells) can be burned, it is common in the industry to prepare the material into sawdust, chips or pellets.

2.4.1 Moisture

The moisture content of wood or shell pieces is an important factor to be considered in the smoking process. Moisture affects the bulk, tap and particle density as well as the porosity of

the material. Normally, wood used for smoking should have less than 20g/100g moisture, and ideally it should be dried to between 8 and 15g/100g moisture for best smoke production.

Several researchers have shown that the types of wood, as well as the moisture content, determine the composition liquid smokes (Guillén and Ibargoitia 1999; Cadwallader 2007). In this process, the burning wood produces smoke which is condensed with the addition of water. In particular, the moisture content affects the pyrolysis temperature and the duration of the smoke generation. With increasing wood moisture content, the combustion efficiency becomes lower and smoke formation increases. Hitzel et al. (2012) showed that higher moisture wood chips resulted in lower mean smoke temperatures. Between 12 and 25g/100g moisture, the levels of PAH4 (benz[a]anthracene, chrysene, benzofluoranthene and benzo[a]pyrene) generated during smoking increased linearly with the level of moisture in the wood.

Overly low moisture content can also be a problem for smoking. Low moisture content causes the wood to burn faster and leads to incomplete combustion. This, in turn leads to elevated formation of PAHs (Simon et al. 2005). While smoking with woods with intermediate moisture levels (8 to 15g/100g) results in the lowest levels of PAH compounds, it has also been found to result in the best organoleptic properties in the finished food. For example, it was shown that when hickory was burned at 250 or 350 °C, different types of pyrazine compounds were formed (Chen and Maga 1995). These compounds contribute to cooked, smoky and roasted flavors. In conjunction, when the moisture content of the sawdust was increased from 12 to 20 or 30g/100g, key flavor compounds such as 2-methoxy-3-methylpyrazine were diminished.

The timber industry generally uses an oven-drying method for determining moisture content in wood. While this approach is generally an accurate way of measuring moisture, errors can occur if the material contains excessive volatiles, which are other than the moisture that is

expected to evaporate from the sample. For best results, a well-ventilated oven with temperatures between 101 and 105 °C are used to dry the sample. Drying is allowed to continue until a constant mass is reached, that is when the weight does not change by more than 0.1 g between successive weightings. Typically, drying occurs within 18-24 h. The moisture content is calculated as:

$$\text{Moisture Content } \left(\frac{\text{g}}{100\text{g}}\right) = \frac{\text{Initial mass} - \text{Dry mass}}{\text{Initial mass}} \times 100$$

2.4.2 Particle Size Distribution

Particle size is one of the primary physical properties of bulk solids, and for combustible materials is a major determinant of the burning efficiency (Vítěz and Trávníček 2010). There are various approaches to measuring and describing particle size. For agricultural materials containing irregular solid pieces, a common approach is to pass the pieces through a series of sieves with different screen sizes, and then weigh the material collected in each fraction. For wood and shell pieces, the common screen sizes are: #8 (2.36 mm aperture size), #10 (2.00mm), #14 (1.40 mm), #16 (1.18 mm), #20 (0.850 mm), #30 (0.600 mm), and #40 (0.425 mm), plus a pan that collects the final fraction. Approximately 100 g of wood sample is used and placed in the top sieve, which is then shaken in a Ro-Tap sieve shaker for 15 min. After the shaking period each fraction is weighed, and the size distribution reported in grams, by weight percent in each fraction, and as percent cumulative weight.

2.4.3 Ash Content

Most wood dust contains common constituents, including cellulose, hemicelluloses, lignin, ash and a small amount of other materials. Ash content is a measure of the mineral content, sometimes considered as impurities in the wood dust. An important aspect of the ash content is that it influences the burning efficiency. One measure of this is the "calorific value", defined as the amount of heat energy released, as wood or other fuel is completely combusted, and measured in units such as kJ/kg. (Spinelli et al. 2011). In a study on 89 piles of wood pieces remaining from logging, Gruduls et al. (2013) showed that pieces with higher ash content had lower calorific value. Also, higher ash content correlated with moisture content and bulk density.

Ash content of wood products is determined by methods described by the National Renewable Energy Laboratory (NREL 2005). Before analyses, the samples are ground into a fine powder and 3-5 g are placed in pre-dried crucibles. The samples are flamed in a Bunsen burner until thoroughly charred, then placed in a muffle furnace. Ashing is carried out at 525 °C for 18 h. The sample is then allowed to cool to 250 °C, before being quickly transferred to a desiccator for cooling overnight. Percent ash is calculated by:

$$\text{Ash}\left(\frac{\text{g}}{100\text{g}}\right) = \frac{\text{Weight after ashing} - \text{Tare weight of crucible}}{\text{Original sample weight} \times \text{Dry matter coefficient}} \times 100$$

2.4.4 Chemical Composition

A majority of wood pieces and nut shells is composed of cellulose, hemicelluloses and lignin. In addition, wood contains a variety of minor compounds such as terpenes and related compounds, fatty acids, and other carbohydrates (Guillén and Ibargoitia 1999). Wood smoke

flavor is generated by the controlled pyrolysis of these major components of wood (Figure 2.1) (Cadwallader 2007).

Cellulose is the major part of most woods. Hence, its pyrolysis products are significant both in number and in abundance in the smoke (Cadwallader 2007). Much of the aliphatic acids and aldehydes are the major volatile products in the pyrolysis of cellulose (Shafizadeh 1984). Cellulose decomposes at 350 °C and evolution of volatile organic compounds continues to temperatures up to 450 °C (Tzamtzis et al. 1997). Unlike cellulose, hemicellulose is a heterogeneous branched polysaccharide (Cadwallader 2007). The pyrolysis of hemicellulose is very complicated and in general hemicelluloses are less heat stable than cellulose. The thermal decomposition of hemicellulose produces furans, anhydroglucose, and carbonyl-containing compounds, and these chemicals are the major volatile components of liquid smoke flavorings (Kim et al. 1974).

Lignin is a relatively high molecular weight and cross-linked polymer, which is comprised of an irregular array of hydroxy- and methoxy-substituted phenylpropane units. It decomposes with heat to phenols and phenol ethers, such as methoxyphenols and syringols, and these compounds are felt to be of considerable importance to smoky flavor (Cadwallader 2007). The thermal degradation of lignin takes place at the temperatures exceeding 400 °C.

The composition of wood used, especially the relative amounts and structure of the major components, is an important factor affecting the volatile composition of the smoke and its subsequent contribution to flavor (Simon et al. 2005). Generally, percentages of cellulose, hemicelluloses and lignin can vary significantly between materials. The percentages of each fraction of several types of materials are shown in Table 2.1.

2.5 Smoked Meat

The smoking of foods, and in particular meats, has been practiced for thousands of years. Smoking contributes a characteristic flavor, aroma and color to foods. In addition, smoking has a preservative effect on food, which increases its shelf life. This occurs through the combined effects of dehydration, along with the antimicrobial and antioxidant activity of the chemical volatile compounds that are created, including formaldehyde, carboxylic acids, and phenols (Baltes et al. 1981; Pszczola 1995). Smoke can inhibit the growth of several microorganisms depending upon the chemical constituents that develop in the smoking process (Ciecierska and Obiedziński 2007). One important fraction is the phenolic compounds including simple phenols, flavonoids, hydrocinnamic acids and phenolic acids. Carbonyl compounds such as formaldehyde and acrolein may also have antimicrobial activity, although the efficacy in smoke has been less well documented. Organic acids such as acetic acid, benzoic acid and propionic acid can also develop, and may provide a portion some antimicrobial effect in smoked foods.

Historically, smoking has been applied to meat products such as ham, sausage and bacon, but it has also been applied to foods such as fish and cheese. In the past few decades, a variety of liquid smoke flavorings have been developed that can be used to give a consistent smoky flavor and color to foods. Liquid smokes have some advantages to burning wood in a smokehouse. They can be applied as sprays in specific amounts to provide uniform dosage, are easy to use, and contribute little to on-site atmospheric pollution (Knowles et al. 1975). An important advantage is that one can directly limit the amount of known toxic compounds in the smoke, so as to decrease the content of certain polycyclic aromatic hydrocarbons (Simon et al. 2005).

The smoking process is carried out using either hot smoking or cold smoking. The key difference between them is that the former involves cooking by heat as the smoke is applied, but

the latter does not. The cold smoking process consists of three steps: salting, drying and smoking, and the operating temperature is controlled between 32 and 43 °C (Sigurgisladottir et al. 2000). The process can take days or weeks, as the smoke slowly penetrates the meat without heat. In contrast to cold smoking, hot smoking is actually a pasteurizing process, and its preservative effect relies on the composition and preparation of raw material, temperature, relative humidity, density and composition of the smoke as well as the smoking time (Doe 1998; Kolodziejska et al. 2002). Usually, the temperature of hot smoking ranges from 66 to 120 °C or even higher, and it is much more effective at preserving meat than cold smoking. Cooking is often done simultaneously with the smoking of meat. In fact, cooking is often more important than smoking in meat processing because it requires careful control of smoking and heating to deliver desired products (Pearson et al. 1996). Federal regulations specify that a final internal temperature of fully cooked processed meat items should be above 65 °C.

The equipment used for smoking, whether hot or cold smoking, involves one of three types of smokehouse, namely natural air circulation, forced air, or continuous flow smokehouse. There are also several variations based on these three types of smokehouse. An illustration of the air-conditioned or forced-ventilation smokehouse is shown in Figure 2.2. These units can permit precise control and regulation of smoking, allowing cooking and smoking in a series of steps, each with specific temperature, humidity and air velocity (Pearson and Gillett 1996).

There are several factors affecting the amount and rate of smoke deposition, including smoke density, smokehouse air velocity, relative humidity, the type of casing (if used), and the surface of the product being smoked. Specifically, high humidity helps smoke deposition, however, it limits color development. Instead, low humidity will cause a dry surface. A relative humidity of approximately 40% in the smoking process is often optimal for smoke penetration

through cellulose-type casings. Furthermore, a rapid movement of air will bring more smoke into contact with the meat surface; however, it makes it difficult to simultaneously maintain high density. Hence, manufacturers generally make a compromise between these parameters and consideration of the smoke uptake and color development needed to obtain satisfactory products (Pearson and Gillett 1996).

With respect to combustible materials used to generate smoke, hickory is perhaps the standard of excellence. It is a hardwood that burns well and creates pleasing smoke flavors. Other hardwoods are also acceptable including mesquite, oak, pecan and apple woods. Softwoods, such as pine or spruce are largely avoided for smoking as creosote and resins result in the harsh flavored meat. Often, the sawdust used for smoking is actually a mixture of hardwoods. The optimal quality wood smoke is produced at a combustion temperature of 343 °C, and at an oxidation temperature of 199-249 °C to minimize the production of carcinogenic substances (Pearson and Gillett 1996).

2.5.1 Water Activity and Moisture Content Analysis

Water is a major constituent of most food products, playing important roles in solubilizing food components, influencing structure-function relationships and plasticizing food macromolecules. Water/moisture content is a measurement of the total water contained in a food based on mass. It is usually defined as a percentage of the total weight:

$$\text{Moisture } \left(\frac{\text{g}}{100\text{g}}\right) = \frac{\text{wt of wet sample} - \text{wt of dry sample}}{\text{wt of wet sample}} \times 100$$

Moisture content in meats is often determined by forced-air convection oven drying of approximately 5 g of grounded sample at 103 °C until a constant weight is obtained.

Moisture content does not measure the functions or the many types of environments or structural domains in which water is present. Thus, water may be present in or outside of cells, trapped within capillaries or void spaces, closely associated with macromolecules or bound at hydrophilic groups of various molecules. Some have postulated the existence of "bound" and "free" water; although others have doubted that water is more tightly bound to molecules such as proteins and carbohydrates, as compared to say other water molecules. At the very least, different domains of water may exist with different degrees of motional freedom.

However, moisture content itself is not always a reliable indicator of food microbial or chemical stability (Damodaran et al. 2007). For example, the critical moisture content for microbial growth may be 30 g/100g in one product and 15 g/100g in another. An alternative measure of water, which seems to correlate better with stability issues, is the water activity. Most directly, water activity is related to the mole fraction of water (X_w) in the food, times an activity coefficient (g) that accounts for non-ideal solution behavior. That is:

$$a_w = \gamma X_w$$

Water activity is often expressed in terms of relative vapor pressure, as this is the way in which it is usually measured. That is, one measures the vapor pressure above the food, assuming it is in equilibrium with the water in the food. Thus,

$$a_w = \frac{P}{Po}$$

where p is the vapor pressure above the food, and p_o the vapor pressure of pure water at the same temperature. In practice, real foods may have water in various capillaries or small droplets that

influence the measured vapor pressure, so that the measured water activity may be an effective one.

Some have posited that water activity is a measure of the water available for biological reactions, thus can determine the ability of microorganisms to grow. Others have suggested that this is an overly simplistic viewpoint of complex phenomena. However, in many cases, water activity does seem to correlate with the ability of various microorganisms to grow. In fact, a_w has a regulatory status. Products with a_w>0.85 must be pasteurized or sterilized by an appropriate method to ensure it is safe for consumption. Dried foods such as beef jerky must have a_w<0.85 to ensure safety and shelf stability.

In general, no specific relationship exists between moisture content and water activity. The relationship must be determined for each food in terms of moisture isotherms, and these can differ whether the moisture is attained through adsorption or desorption. In most practical situations, a_w is measured through the vapor pressure above the food in equilibrium with a small air space, and this is most often detected by measuring the dewpoint of the air.

2.5.2 pH and Color Measurement

As indicated previously, one of the primary purposes of smoking meat is to develop the aroma and color. Smoking and cooking are simultaneously involved in color formation. Furthermore, the browning or Maillard reaction produces the characteristic brown color on the surface of smoked products, and involves the interaction of free amino groups from proteins or other nitrogenous compounds and carbonyl groups from reducing sugars and other carbohydrates (Pearson and Gillett 1996). The Malliard reaction is usually divided into three main stages. The initial stage starts with a condensation reaction resulting in formation of an *N*-glycosylamine.

The next stage proceeds with a Schiff base formation and Amadori rearrangement to release the amino group and initiate sugar fragmentation. The final step leads to formation of furfurals or hydroxymethyl furfural, which are brown or black in color. A proposed scheme of the Maillard reaction is given in Figure 2.3. The high amount of total reactive carbonyls in wood smoke and presence of amino groups are big contributors to browning during smoking of meat.

Browning is typically studied by use of colorimetry, which typically give some measure of lightness, hue, and color saturation. Many scales exist for describing color, usually in terms of two or three parameters. These include, for example, the HSL, CYMK, or LCH systems. One popular scale is the CIE L*a*b* system. Here, L* represents the degree of lightness 0 (black) to 100 (white). The a* axis ranges from -127 (green) to +127 (red), while the b* axis ranges from -127 (blue) to 127 (yellow). While unique color can be described in terms of specific L*, a* and b* values, sometimes other derivative measures can be useful. For example, the browning index (BI) has been developed to describe the degree of browning, and is given by:

$$BI = \left[\frac{100(x - 0.31)}{0.17}\right]$$

where

$$x = \frac{(a + 1.75L)}{(5.645L + a - 3.012b)}$$

It is also useful to describe an overall difference (ΔE) in color between a sample and a control:

$$\Delta E^* = \sqrt{(\mathrm{L}_2^* - L_1^*)^2 + (\mathrm{a}_2^* - \mathrm{a}_1^*)^2 + (\mathrm{b}_2^* - \mathrm{b}_1^*)^2}$$

where the subscripts 1 and 2 refer to the reference and sample, respectively.

2.5.3 Yield/Cook Loss

During industrial processing of meat and poultry, one of the most important factors determining profitability and production control is processing yield (Sigurgisladottir et al. 1997). While yield can be considered at different points in the process, one critical place is the loss in yield due to cooking. That is, during cooking the product invariably loses some amount of juices, as moisture or fat exit the product. It occurs as the protein structure denatures and is less able to hold onto moisture. In addition, some of the solid fat may melt, making it easier to run out of the meat (Cadwallader 2007). The cook loss is determined by weighing samples before and after cooking (Ertbjerg et al. 1999). It is defined as:

$$\%\ \text{Cooking Loss} = \frac{\text{Cooked Weight} - \text{Raw Weight}}{\text{Raw Weight}} \times 100$$

2.5.4 Water-Holding Capacity

Water-holding capacity (WHC) is the ability of meat to retain its inherent moisture or added water even though external pressure (e.g. heat, pressing) is applied (Hamm 1960; Lawrie and Ledward 2006). In this context, we are mostly concerned with how well moisture is retained in the finished product. It is an important property as it influences several sensory properties, such as juiciness, tenderness, and flavor of meat during mastication. In may also be of consideration to the processing of the meat, as WHC may be related to other moisture properties such as how well meat retains moisture during handling or cooking.

Several methods exist for assessing water-holding capacity. Typically, these subject the sample to a force through pressing or centrifugation, then measure the water released. A common method involves pressing a sample by a known force (Kauffman et al. 1986). Nearly 1 g sample

is placed between two filter papers, and then pressed by a 40-kg weight (or equivalent force) for 2 min (Wang et al. 1993). The amount of water released from the sample is measured directly by weighing the filter papers.

2.5.5 Texture Attributes

There are several textural attributes important to meat and poultry products, including tenderness, chewiness and juiciness. Several instrumental methods have developed to try to measure these attributes. These have included the Warner-Bratzler shear force measurement, as well as indirect measures such nuclear magnetic resonance measures of moisture holding properties (Damez and Clerjon 2008). The usefulness of texture profile analyses (TPA) to assess several attributes of chicken meat has been described by Alfaig et al. (2013). This approach is imitative in nature, in that the sample is compressed 2 times in a chewing motion. From the force-distance response curve, various forces and areas are measured that correspond to hardness, cohesiveness, springiness and adhesiveness.

As mentioned, the Warner Bratzler shear force has been a common means of assessing tenderness in meat samples. In this approach, a cylindrical sample of the meat is prepared and cut laterally by a v-shaped blade. The maximum force is taken as a measure of tenderness. One complication of this approach is the need to prepare samples with precise geometry, and while attempting to maintain a regular orientation of muscle fibers. An alternative uses the “Meullenet-Owens razor shear blade” (A/MORS). The probe uses a small blade common to arts and crafts, with a flat leading edge 12 mm long. The analysis is performed on whole intact fillet halves with a height of at least 20 mm. The blade is brought directly into the sample and the maximum force recorded. Ten determinations, including shear force (N) and shear energy (N*mm), are made for

each treatment of the chicken samples (Lee et al. 2009). This method has been shown to be quicker and simpler than the Warner-Bratzler shear and Allo-Kramer shear methods, while providing as good or better correlation with sensory measures of tenderness (Cavitt et al. 2004, 2005).

2.6 Sensory Research

Smoked chicken is characterized by its unique smoky, sweet, woody and earthy flavors and juicy texture. In this study, we pursued both descriptive and consumer sensory methods. The former is an effective tool to quantify differences in specific flavor and texture attributes, in this case it applies to different woods and shells used to smoke chicken breast meat. The latter approach provides a better understanding of consumer preference or acceptance of products. When considered together, the two approaches may provide a better understanding of how differences in flavor or texture attributes influence consumer satisfaction with the smoked chicken products, and help to constitute an approach to consumer demands.

2.6.1 Descriptive Sensory Analysis

Descriptive sensory analysis is a sophisticated approach to obtain a complete description of the product, in a way that describes individual character attributes along with an intensity rating to each descriptor. It involves the detection, the qualitative description of the sensory attributes in a product, and development of an intensity scale of these attributes by a trained panelist of 5 to 20 experts, with usually about 10 panelists. These panelists are selected by a set of prescreening questions, tests and personal interviews to make sure they have strong interest, availability and sensorial capacities. Generally, more than 120 hours of intensive training with a

variety of food products is required. The descriptive analysis can take different forms such as the flavor/texture profile method, the quantitative descriptive analysis (QDA®), the Spectrum™ descriptive analysis, time-intensity analysis, and free-choice profiling. In this study, the Spectrum™ descriptive method based on a 15-point numerical scale with references (where 0= "not detected" and 15 = "extremely strong") was performed to detect the influence of the different woods (hickory, pecan shell, apple tree and mesquite woods) on the flavor and texture attributes of smoked chicken.

Usually, descriptive analyses methods follow a similar sequence of steps. The first step involves generation of terms, which includes literature searches, group discussion with trained experts about similar products or consumer opinions, or information from their knowledge of product characteristics (Lawless 1991). Next, there is the development of definitions and references for each attribute along with a scorecard. This includes designing an appropriate scale, presentation of references and protocol for assessing the attributes. This ensures that each assessor can accurately describe attributes and have a consistent understanding of the references and scales (Murray and Delahunty 2000). It is critical that all panelists are aligned about each descriptive term. After extensive group and individual training, performance tests are conducted to examine each individual panelist's ability and readiness as an evaluator, based on certain criteria, for example, sample interaction plots. Subsequently, several replicated sessions of actual descriptive analysis are held in individual booths once judges are proven to be ready. In this stage, the experiment design is carefully considered for balance and carryover effects. The final step is data collection and analysis.

2.6.2 Consumer Analyses

Often, it is also essential to assess the impact and acceptability of new products as perceived by consumers. In contrast to descriptive sensory analyses, assessors do not provide evaluation of the intensity of specific attributes, but rather provide valuable opinions about acceptance, preference or perception (Válková 2012). Contrast to descriptive sensory, the assessors in this study is limited or no training, however, they can still provide us valuable and irreplaceable opinions about their acceptance, preference and perception. Thus, panelists requite no or limited training.

Prior to initiating consumer studies, there are several points to be addressed in the experimental design and execution. First, it is vital to recruit a relatively large group of consumer participants (more than 80). Often, there are specific restrictions such as having no existing food allergies or conflicts of interest, and have experience with similar products, for example by consuming the product at least once a week. There is usually some substantial sample preparation and holding to be considered, such as the way in which it will be subdivided, cooked or held at a specific temperature. Finally, a questionnaire needs to be developed that includes the attributes to be evaluated and an appropriate scale for doing so. Consumers may be polled as to their liking or preference for products overall, or with respect to particular attributes such as appearance, flavor or texture. One common approach to assessing likeability is through hedonic ratings. For example, a nine-point hedonic scale may be presented that allows consumers to rate products on a continuous scale from 1 (dislike extremely) to 9 (like extremely) to indicate degree of liking.

2.7 Determination of Phenolic Compounds in Smoked Chicken

As discussed previously, smoking is a method that helps preserve foods by adsorption of aromatics from the wood smoke penetrating into the food. The composition of smoke is very complex. According to previous research (Hamm 1977; Daun 1979; Cliffod et al. 1980), there are only a few compounds, such as hydrocarbons, acids, ketones, aldehydes and phenols found in the smoked meat even though there are more than 300 substances existing in the smoke. It is generally believed that phenolic compounds produced by the pyrolysis of lignin give characteristic smoky flavor, bacteriostatic activity and antioxidant activity. Cardinal et al. (1997) found that the nature of the wood used for smoking, the method of smoke generation, pyrolysis temperature and the smoking process greatly affect the relative amount of phenolic compounds in smoked products.

Phenolic compounds contain a benzene ring bearing one or more –OH groups. These exist in various structures from simple phenolic molecules to high molecular-weight polymers (Balasundram et al. 2006). Studies indicate there are 15 phenolic compounds closely associated with smoky flavor in smoked products; Table 2.2 presents each of these 15 phenolic compounds and its corresponding flavor note.

Typically gas chromatography is used to separate and identify these key compounds. This includes some means of extracting or recovering the compounds from the food matrix, plus developing column conditions and temperatures that will best resolve the many compounds present. One popular process involves the use of solid phase microextraction (SPME) coupled with GC/MS (gas chromatography/mass spectrometry). SPME is a novel analytical technique, which was invented by Pawliszyn (1997) and used to analyze organic compounds in water or soil, especially phenolic compounds (Baciocchi et al. 2001; Bartak and Čáp 1997; Gonzalez-

Toledo et al. 2001; Ohlenbusch et al. 2000). Nowadays, SPME has been widely applied as an isolation approach in analyzing volatile compounds across various food categories, including cheese (Guillén et al. 2004), dry-cured ham (Lammers et al. 2011), and rice (Limpawattana et al. 2008). It constitutes of a short length of fused silica fiber coated with a thin polymer material. The fiber is held in place with a stainless steel plunger sheathed by a protective needle. In theory, the analyte may be adsorbed by immersion or static headspace extraction in liquid or solid samples. Most commonly, analytes are adsorbed onto the fiber as it sits within a headspace surrounding the sample. A more complete extraction of compounds requires distillation techniques. SPME offers several advantages over those methods in ease of use, requirements for smaller sample volumes and in limiting the need for chemical solvents (Harmon 1997; Kataoka et al. 2000; Pillonel et al. 2002; Reineccius 2002).

There are several parameters to be considered or optimized when designing an SPME procedure. This includes selection of a fiber coating with optimal adsorption characteristics, sample mass, extraction temperature and time. Guillén et al. (2002) determined that an SPME fiber coated with polyacrylate (85 μm film thickness) was a good choice for analyzing volatile components in raw and smoked black bream and rainbow trout. Similarly, Serot (2003) also used an 85-μm film thickness polyacrylate fiber for determining phenolic compounds in smoked herring. A schematic of the fiber and adsorption process is shown in Figure 2.4 (Lammers et al. 2011). In order to obtain reliable results, a water bath is commonly used for maintaining a constant temperature (Jurado et al. 2009).

Once adsorbed onto the fiber of choice, the fiber is placed within a gas chromatograph and subject to desorption onto the column. In the process, GC must separate the compounds so as to resolve the many peaks of interest. In addition, the peaks need to be identified. One approach

is to identify the compounds through the use of known standards from which matching retention times can be attained. Gas chromatography coupled with mass spectrometry (GC-MS) is another approach. GC-MS has been used to study the phenolic compounds in assorted foods, as it improves the accuracy and detection of volatile compounds. For instance, Liu et al. (2007) identified a total of 116 volatile compounds in traditional Chinese Nanjing marinated duck, using GC-MS coupled with SPME. Mass spectrometry work based on the principle that gas-phase ions are separated according to mass/charge ratio. These are sequentially detected then identified by their retention times and mass spectra compared with the known compounds in the instruments library. The semi-quantification can be calculated by several methods, such as introducing internal or external standard solutions, arbitrary units of the peak area counts divided by 10^5 (Guillén et al. 2002), or the ratio of the peak area to the total peak area (Lammers et al. 2011).

Table 2.1 Chemical Constituents of Several Types of Wood

Biological Material	Cellulose (%)	Hemicelluloses (%)	Lignin (%)
Hickory [a]	53.6	7.1	17.7
Mesquite [a]	8.0	8.1	64.0
Coarse Pecan Shell [b]	36.03	16.33	30.59
Almond Shell [c]	40.5	19.7	27.2
Hazelnut Shell [c]	27.8	14.8	19.2

[a] Mega (1986); [b] Littlefield (2010); [c] Wartelle and Marshall (2001).

Table 2.2 15 Phenolic Compounds Associated With Smoky Flavor

Phenolic Compounds	Flavor Response
Phenol [a]	Woody, sweet
2-Methylphenol [a]	Burnt, smoky
3-Methylphenol [a]	Smoky, spicy, woody
Guaiacol [a]	Sweet, smoky
2,6-Dimethylphenol [a]	Smoky, woody, spicy
3-Ethylphenol [a]	Smoky, woody, green
2,4-Dimethylphenol + 4-Ethylphenol [a]	Roasted, smoky, burnt
2,3-Dimethylphenol [a]	Roasted, woody, pungent
4-Methylguaiacol [a]	Burnt, smoky, woody
4-Ethyl-2-methylphenol [a]	Smoky, burnt
2-Ethyl-4-methylphenol [a]	Woody, smoky, salty
4-Ethylguaiacol [a]	Woody, spicy, smoky
2,6-Dimethoxyphenol [a]	Burnt, woody, smoky
2-Methoxy-4-propylphenol [a]	Sweet, spicy, woody
4-Methoxy-3-(methoxymethyl)phenol [a]	Caramel, woody, smoky

[a] Modified from Xie et al. (2008).

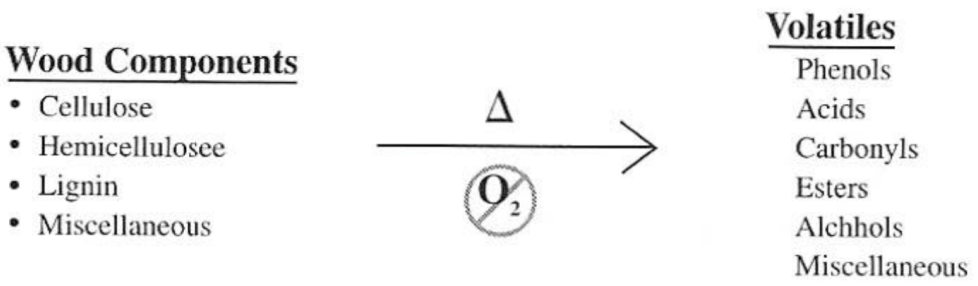

Figure 2.1 Controlled Pyrolysis of Wood Components (Cadwallader 2007).

Figure 2.2 ALKAR™ (Lodi, Wisconsin) Forced-ventilation Smokehouse

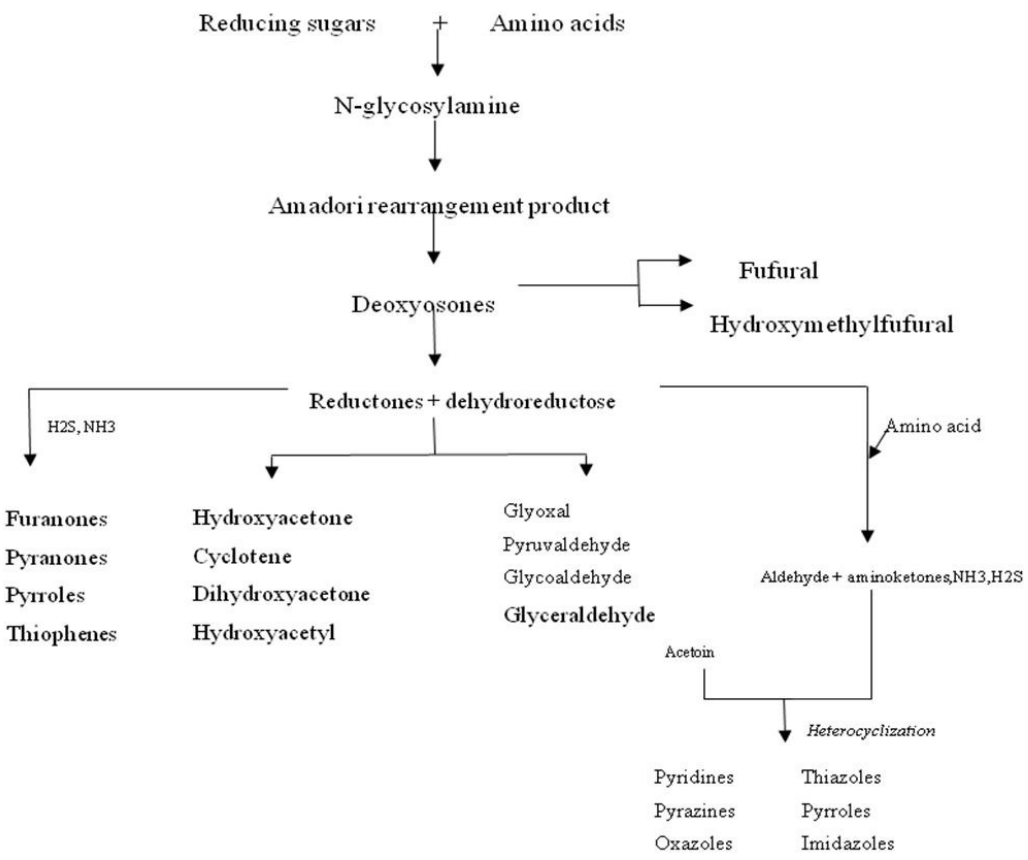

Figure 2.3 General Mechanism of Maillard Reaction Showing the Formations of Flavor Compounds (Van Boekel 2006)

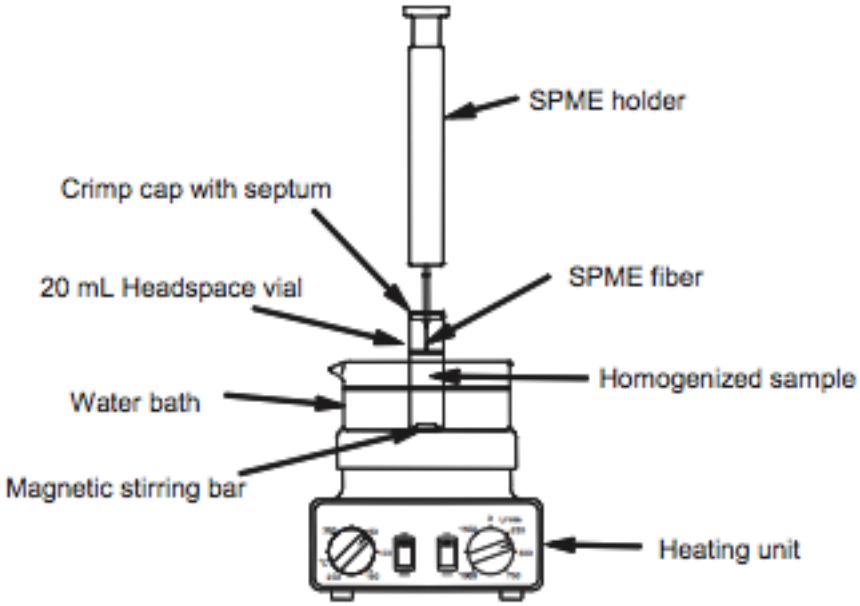

Figure 2.4 Extraction of Volatiles from The Headspace of Continuously Stirred Dry-Cured Ham Homogenate by the Solid Phase Microextraction (SPME) Device (Lammers et al. 2011)

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CHAPTER 3

Characterization of Pecan Shells and Their Effect on the Yield, Moisture Properties, Color and Texture of Smoked Chicken Breast

3.1 INTRODUCTION

Pecan [*Carya illinoinensis* (Wangenh.) K. Koch] is a tree nut crop cultivated in the southern and eastern areas of the Unites States, as well as Mexico, China, Israel and Australia. According to the International Nut & Dried Fruit Council (2015), the United States and Mexico lead the world's production of pecans, contributing to 93% of world production. The estimated production in those countries in 2014 was 60,180 and 40,800 metric tons, respectively. The next producers include South Africa (5,724 metric tons) and Australia (1,080 metric tons). Global production of pecans totaled more than 108,000 metric tons (kernel basis), with a 59 percent of increase in production over the last ten years. As a result of shelling operations, there are thousands of tons of pecan shells left over from the process, and this number is growing with the rapid increase in pecan production. The pecan shells are normally considered as waste materials and they are often discarded in landfills.

The efficiency of shelling is described by the shelling ratio (SR), which describes the weight of the meat compared to the total weight, and typically varies from 0.4-0.6 depending on cultivar (Xu 2000). Based on NASS (2015) data, it is estimated that 52,420 tons of pecan shells were generated in 2012 from shelling operations in the United States. Much of the pecan shell waste goes to landfill. Some pecan shells have been used commercially as mulch in ornamental landscapes, as they diminish soil moisture loss while providing an appealing color and texture to gardens and landscapes. They also increase soil acidity levels, which can be beneficial to acid-

loving vegetation. However, recurrent use of these byproducts can destroy less tolerant plants (Brison 1974). There have been a few studies examining other commercial uses for pecan shells, as in the production of biofuels (Mohammed-Khah and Ansari 2009). Babu et al. (2013) demonstrated that natural pecan shell extract is a potent alternative antimicrobial against food pathogens, such as *L. monocytogenes* and other *Listeria* spp.. In addition, pecan shells have been converted to activated carbon for water filtration (Bansode et al. 2003), sugar refining (Rao et al. 2000), and geosmin adsorption (Ng et al. 2002).

Pecan, and the related hickory, wood have been used for smoking and grilling meats for years. It is most commonly used in the southeastern United States, especially on pork and chicken, as it provides a sweet smoky flavor to the meat. As with the wood, pecan shells are high in cellulosic and ligneous materials that are combustible and produce smoke in the process. It is reasonable to think that the shells could produce smoky flavors in meats either similar to pecan wood, or having their own unique characteristics. However, there have been no specific scientific studies on the use of pecan shells in smoked meats, nor how they would compare to other common woods used for smoking.

Smoking has been used for thousands of years to impart a characteristic flavor, aroma, and color to meats. In addition, smoking has a preservative effect on foods, increasing the shelf life as a result of the combined effects of dehydration, antimicrobial and antioxidant activity of several of the chemical volatile compounds. These constituents include formaldehyde, carboxylic acids, and several phenolic compounds (Baltes et al. 1981; Pszczola 1995). Smoke can inhibit the growth of some microorganisms depending upon the particular profile of phenolic constituents in the smoked food (Ciecierska and Obiedziński 2007). Traditionally, smoking has

been applied to a variety of cured meats such as ham and bacon, but more recently has been used with a variety of products such as fish and cheese.

There are many factors that influence smoke production and the chemicals it delivers to foods. These include the type, age and density of the wood, as well as factors related to the smokehouse such as temperature, air velocity and relative humidity. Several researchers have shown that the type of wood and the wood moisture content determine the chemical composition of liquid smokes produced from the wood (Guillén and Ibargoitia 1999; Cadwallader 2007). The moisture content likely affects the pyrolysis temperature and duration of the smoke generation. Ash content is another important component in combustible biomaterials, as it represents inorganic matter that does not contribute to the combustion process. A key measure of the burning process is the calorific value, as it represents the heat energy that can be delivered per unit mass (Spinelli et al. 2011). Gruduls et al. (2013) showed that differing levels of ash could influence the relative moisture, bulk density and caloric value of wood chips used for fuels. The combustion efficiency, as well as the quantity of smoke produced, is also influenced by the size and shape of particles being heated (Vítěz and Trávníček 2010). Thus biofuels, as well as products designed for smoking, are produced in chips, sawdust or pellets of specific size to optimize the process.

The purpose of this study was to investigate the use of pecan shells as a source of smoke in the production of cooked chicken breast meat. Chicken breast was chosen as it has a relatively mild flavor profile compared to other meats, and would accentuate the contribution of smoky flavors in the final product. As part of the objective, key properties of the shells were determined including moisture content, ash content, and particle size distribution of the shells after processing them to a suitable size for use in a smokehouse. The pecan shells were also compared

to three woods commonly used in smoking including hickory, mesquite and apple tree wood. Important quality attributes of the finished product were determined including moisture content, water activity, pH, color and textural attributes. In addition, the comparative effects of smoking with pecan shells or one of the three woods on product yield were determined, as this is a key economic factor in processing of smoked meats.

3.2 MATERIALS AND METHODS

3.2.1 Preparation of Raw Materials

Pecan shells were obtained from South Georgia Pecan Company (Valdosta, GA). The shells were harvested in late 2014 and contained a mix of 'Elliot', 'Desirable' and 'Stuart' cultivars. The shells were further ground into small chips using a stone grinder (Westinghouse Electric Corporation, Pittsburgh, PA), then screened by shaking in a round separator (SWECO Model Vibro-Energy®, Macon, GA) for 20 min in order to remove loose soil from shells. The shells were kept in sealed in polyethylene bags and stored at room temperature.

Other sources of smoke were from commercial products developed specifically for smokehouse use. These included sawdust of hickory, mesquite and apple tree wood, and were purchased from Frantz Company, Inc. (Butler, WI). These were stored in similar conditions to the pecan shells before use.

3.2.2 Pecan Shells and Wood Properties

3.2.2.1 Moisture Content

The moisture content of the pecan shells and various woods was determined in by the Official Method 985.14 (AOAC 2005) with slight modification. Samples were weighted into

tared aluminum pans and dried at 103 °C and 17.5 in Hg in a vacuum oven (Model 1430 MS, VWR Scientific, Radnor, PA) until a constant mass was attained.

3.2.2.2 Ash Content

The ash content for the different sawdust products was measured by a method developed by the National Renewable Energy Laboratory for biomass (NREL 2005). Prior to analysis, all samples were pre-dried in a forced-air convection oven at 103 °C for at least 24 h. Subsequently, crucibles were heated to 700 °C to condition them. Samples were weighed into ceramic crucibles and thoroughly charred by a Bunsen burner flame, then transferred to a muffle furnace (Thermo Scientific Inc, Model Thermolyne 48000, Suwanee, GA). Ashing was performed in the muffle furnace at 525 °C for 18 h. The samples were removed after the oven temperature decreased below 250 °C, then crucibles were quickly transferred to a desiccator for cooling. The crucibles and ash were weighed after 24 h.

3.2.2.3 Particle Size Distribution

The particle size distribution of the shell or wood pieces was measured using a screening method (ASTM 2013). The following screens were used for the analyses: #8 (2.36 mm aperture size), #10 (2.00 mm), #14 (1.40 mm), #16 (1.18 mm), #20 (0.850 mm), #30 (0.600 mm), and #40 (0.425 mm). Approximately 100 g of wood sample was placed in the top sieve and shaken in the mechanical shaker for 15 min. After shaking, the mass of material residing beneath each screen size was determined. The values in grams, percentage by weight of each fraction, and cumulative weight (%) were determined.

3.2.3 Smoking Process

Frozen chicken breasts were obtained from Wayne Poultry (Pendergrass, GA). The pieces had been previously injected and tumbled with a marinade solution, brining the salt and phosphate level to in 0.2 and 0.4% in the finished product. The frozen pieces were stored at -20 °C, then thawed at 4 °C for 24 h prior to further processing. Once thawed, loose connective tissue was removed and the pieces rinsed with water and patted dry.

Cooking and smoking was conducted in an ALKAR^{TM} smokehouse (Model DEC, Lodi, WI) 1.4 m deep, 1.5 m in width and 1.9 m tall. The unit is designed to provide accurate control of air temperature and humidity during cooking, while providing high-volume flow of air. During cooking, smoke was produced by combustion of wood or shells in a smoke generator (ALKAR SmokeMaster^{TM} III, Lodi, WI). The pecan shell or wood pieces were placed in the hopper and fed to the burner plate at ~500 g/h. Pyrolysis of the material occurred at 365 °C.

For each treatment group (pecan shells, hickory, mesquite or apple wood), 40 chicken breasts were processed in the smokehouse. The pieces were distributed on 4 shelves of a trolley and wheeled into the smokehouse. The cooking/smoking protocol was as follows: an initial cook step at 60 °C for 20 min; a smoke/cook step at 66 °C, 40% RH, and air speed of 2 m/s; a final cook step at 77 °C and 88% RH until the inner temperature of the breast meat reached 74 °C and was maintained for 20 min. The smoked chicken breasts were allowed to cool to ~25 °C and immediately vacuum-packed using a Henkelman model 600 vacuum machine (Henkelman UK, Ltd., Ashford, Kent, UK). Samples were stored refrigerated at 2 °C until subsequent analysis. The smokehouse was thoroughly cleaned after each run by chemical detergent (Momar NOX Smoke II, Atlanta, GA) to eliminate carry-over of flavor volatiles to subsequent batches.

3.2.4 Properties of Smoked Chicken Breasts

3.2.4.1 Moisture Content Analysis

Moisture content of the raw and cooked chicken breast was determined by the oven-drying method (AOAC 2005). Samples were ground and ~5 g were dried in an aluminum dish at 103 °C in a forced-air convection oven (Model 1430 MS, VWR Scientific, Radnor, PA). Drying until a constant mass was attained. Samples were allowed to cool in a desiccator. The weight loss in drying was reported as moisture. All sample measurements were done in triplicate.

3.2.4.2 Water Activity

Water activity (a_w) was measured using an AquaLab Model Series 3 meter (Decagon Devices, Inc., Pullman, Washington). Approximately 1-2 g of ground smoked chicken sample was placed in an a_w sample cup and inserted into the meter at ~21 °C. All sample measurements were done in triplicate.

3.2.4.3 pH Measurement

For pH measurement, a ~10 g sample of the smoked chicken sample was ground and homogenized in 100 mL distilled water. The pH value was obtained by immersing the tip of the electrode into the suspension and allowing it to equilibrate at ambient temperature. The pH meter (Model AP5, Denver Instrument Co., Bohemia, NY) was calibrated prior to use using pH 4 and 7 buffer solutions. All analyses were performed in triplicate.

3.2.4.4 Color Measurement of Smoked Chicken Breasts

The color of smoked chicken breasts was assessed using a Minolta spectrophotometer (Model CM-700d, Konica Minolta Sensing, Inc., Ramsey, NJ). Before analysis, the instrument was calibrated using a white calibration tile (CM-A177). The color of the intact smoked chicken breasts was measured in the CIE L*a*b* system, where L*is the lightness (0-100 for black to white), a* ranging from -60 (green) to 60 (red), and b* ranging from -60 (blue) to 60 (yellow). The color was measured under a D65 illuminant system and there was no gap between the sample and the lens of the colorimeter. Color measurements were taken at three random locations across each product surface, and averages attained from three smoked chicken breast pieces per treatment.

3.2.4.5 Cook Loss

Cook loss was determined by weighing select samples before and after cooking in the smoke house. Changes in cooking occur due to moisture loss (or sometimes absorption), as well as from losses in fat content (Ertbjerg et al. 1999). Twenty replicates of each treatment were evaluated. Cooking loss was expressed as:

$$\%Cook\ Loss = \frac{Cooked\ Weight - Raw\ Weight}{Raw\ Weight} \times 100$$

3.2.4.6 Water-Holding Capacity (WHC)

Water holding capacity (WHC) was determined by the pressing method, which estimates the amount of water that can be squeezed from a sample under a given force (Kauffman et al. 1986). Approximately 1 g sample was placed between two filter papers (Whatman No.1), and

then pressed under 392 N for 2 min (Wang et al. 1993) using a TA-XT Plus Texture Analyzer (Stable Micro System, Surrey, UK) equipped with 10 cm parallel plates. The amount of water released from the sample was measured directly by weighing the sample and filter papers before and after pressing. Five repetitions of the analysis were performed for each treatment.

3.2.4.7 Texture Attributes

Texture attributes were determined using a TA-XT Plus Texture Analyzer (Stable Micro System, Surrey, UK) with the Texture Exponent TE32 version 5.0 software. Tenderness of cooked samples was assessed by a shear force method as described by Lee et al. (2009). The instrument crosshead was fitted with a probe holding an X-Acto #17 razor blade 24 mm long by 8 mm wide (Elmer Products, Westerville, OH). All samples were kept and tested at room temperature. The blade was lowered into the sample at a pretest speed of 2.0 mm/s, a test speed of 10.0 mm/s and a post-test speed of 10.0 mm/s. The analysis was performed on whole intact fillet halves with a height of at least 20 mm. Ten determinations of shear force (N) were made for each treatment of the smoked chicken samples.

3.2.5 Statistical Analysis

Data were analyzed using SAS statistical software (Version 9.3, SAS Institute, Cary, NC). All results were reported as means ± standard deviation. One-way analysis of variance (ANOVA) was performed followed by Tukey's test for significant differences at $p<0.05$.

3.3 RESULTS AND DISCUSSION

3.3.1 Moisture, Ash and Particle Size of Pecan Shells

The moisture content of the four kinds of sawdust ranged from 7.94 to 11.43 g/100g (Table 3.1). Pecan shell had the highest moisture content at 11.43 g/100g. For best smoke production and flavor development, the smoke source should be controlled to have less than 20g/100g moisture, and ideally be dried to between 8 to 15g/100g moisture. As previously noted (Guillén and Ibargoitia 1999; Cadwallader 2007), the type of wood and its moisture content are prime determinants of the chemical composition of liquid smokes. Moisture content in particular influences the pyrolysis temperature and the duration of smoke generation. With increasing moisture content of the wood, the combustion efficiency becomes lower and smoke formation increases, with less volatile flavor components being developed. However, excessively low moisture content causes the wood to burn faster, resulting in incomplete combustion and elevated formation of carcinogenic polycyclic aromatic hydrocarbons (PAHs) (Simon et al. 2005).

The ash content of the four materials is shown in Table 3.1. Values varied from 0.97 g/100g (hickory), 2.53 g/100g (pecan shells), 2.57 g/100g (apple tree) and 3.11 g/100g (mesquite). As noted previously, ash content is a measure of the minerals in the wood, and might be considered as an “impurity” as it is non-combustible matter. Calorific value is an essential property for any fuel (Spinelli et al. 2011) and ash will have some effect on the caloric value of the combusting material. Gruduls et al. (2013) observed that higher ash content was often correlated with lower calorific value, lower moisture and lower bulk density in several wood chip harvested from February to May. However, we observed no particular correlation between moisture content and ash in our study.

Figure 3.1 and Table 3.2 show the particle size cumulative distribution of the four kinds of wood or shells. Following the work of Littlefield et al. (2011), we classified the woods into three categories: those retained on the #10 screen were defined as “coarse”; those that passed through the #14 screen were define as “fine”; and those retained between the #10 and #14 screens were classified as “medium”. Thus, even though the pecan shells were further ground before using, they still retained a relatively large fraction of coarse particles (70.43%) as compared to the other wood sources (13.61 to 30.56%). In contrast, apple tree woods mainly consisted of fine particles (70.71%). The difference in particle size distribution of these wood fuels may have an influence on the combustion and pyrolysis process. For example, Suranani and Goli (2012) showed that the particle size of groundnut shells influenced burning performance in a fluidized bed combustor. They noted that maximum combustion efficiency was attained (89.5%) when pieces had an average size of 0.273 mm. It should be noted that the purpose of this study was to test the feasibility of pecan shells as a smoke source in smoked meats. Subsequent studies should examine the roles of optimal moisture content, particle size, porosity, and on the burning process.

3.3.2 Moisture Properties and pH of Smoked Chicken Breasts

The moisture, water activity and pH of chicken breasts smoked with the four different wood or shells are shown in Table 3.3. The moisture content of the differently treated pieces varied only modestly (70.13 -71.12 g/100g), although the differences were significant. The original moisture content of chicken breast meat was 80.56 g/100g. Chicken breast smoked with hickory had the greatest moisture and that with apple wood the lowest moisture content. There were no significant differences in a_w with values ranging from 0.952-0.954. The pH also varied

only slight, with values of 6.41-6.43 for the hickory and pecan shell samples, and 6.39 for the mesquite and apple samples. In comparison, the pH of unsmoked breast meat was 6.18. The pH of muscle foods varies with the bird age, conditions of processing, as well as the type and amount of salt and polyphosphates used for injection or marination. Research has shown that the generation of basic volatile compounds in the smoking processing, such as ammonia and trimethylamine, may contribute to higher pH, at least in hickory smoked meats (Hyytia et al. 1999; Reddy et al. 1997; Ruiz-Capillas and Moral 2001). Hence, it was suggested that the pH of the finished smoked product may depend on several properties of the wood, including species, moisture content, and structure, as well as the percentages of hemicellulose, cellulose and lignin.

It has been well established that, amongst other factors, the ability for microorganisms to grow on food products is dependent upon the water activity. In this study, all a_w values were greater than 0.85 and all pH values were above 4.5. Thus, the finished chicken breast samples would be deemed a perishable food with a high risk of supporting food poisoning microorganisms if not properly handled. Babu et al. (2013) demonstrated that extracts of pecan shell could inhibit *Listeria* spp. and *L. monocytogenes* serotypes when applied to poultry skin model. However, no studies have examined the efficacy of smoke components originating from pecan shell in inhibiting microbial growth in smoked meat products. Thus, this may an important area for future studies.

3.3.3 Color Assessment of Smoked Chicken Breasts

Smoking and cooking are simultaneously involved in color formation in smoked meat products. Lightness (L*) values ranged from 72.86 to 76.57, and chicken breasts smoked with pecan shells were the darkest (lowest L*) amongst the four samples (Table 3.4). The values of a*

ranged from 3.18 to 5.71, while those for b* ranged from 20.65 to 25.70. The a* and b* values were also greatest for the pecan-shell smoked chicken. While all samples were light brown in color, conversion to the L*c*h* system indicates the samples have modest color saturation with a hue more yellow than red. Amongst the samples, those smoked with pecan shells had somewhat greater chroma and a little redder hue. Thus, wood species was a significant factor in the final color of the smoked chicken ($p<0.05$).

Pearson and Gillett (1996) explained that the brown color on the surface of smoked meats emanates from Malliard reaction products. These involve interactions between protein amino groups (or some other nitrogeneous compounds) and carbonyl groups from reducing sugars and other carbohydrates. Wood smoke contains a variety of carbonyl compounds, such as formaldehyde, acetaldehyde, propanal, acetone, acrolein, isobutyraldehyde, and butanal, and these may be part of the browning reaction in smoked meats (Love and Bratzler 1966).

Meat products can also be subject to reactions that cause pinking. In cooked white poultry meat, consumers often consider pink color as an indicator of the unsafe and undercooked meat. Holownia et al. (2004) showed that high pH, muscle lightness and decreased oxidation-reduction potential contribute to pink defect. In this study, hickory and pecan shell smoked chicken had slightly higher pH, but likely not high enough to result in major color differences. In uncured meats, post cooking pink color can occur through contamination with nitrates. In smoked products “pink ring” can result from incomplete combustion or contaminates that produce nitrogen oxides (NO_x) (Cornforth et al. 1998). The latter react with meat surface to form nitrosomyoglobin. In rare occasions, carbon monoxide can be generated and react with myoglobin to form a reddish pigment. One of these mechanisms may have contributed to a slightly redder color in the surface of the pecan-smoked chicken breast.

Regardless of the cause of the darker, redder color in the pecan shell smoked product, the color change was localized to the surface of the meat. Figure 3.2 shows photographic images of the chicken breast samples, sliced in half to show a cross section of the sample. The dark color was present only in the first millimeter of the surface, and no signs of other coloration were noted within the products.

3.3.4 Cook Loss and Water-Holding Capacity of Smoked Chicken Breasts

The cook loss and water-holding capacity of the smoked chicken breasts are given in Table 3.5. The cook loss of pecan shell-smoked chicken breast was 26.22%, but there were no differences in cook loss amongst the differently treated samples. Khiari et al. (2013) reported cook losses in cooked chicken breasts of 19.35 to 21.90%, depending on the type of marinade ingredients used to treat the samples. These samples had been cooked in a convection oven at 177 °C until an internal temperature of 74 °C was reached.

Water-holding capacity (WHC) is the ability of meat to retain its inherent or added moisture even though external stress (e.g. heat, pressing) is applied (Hamm, 1960; Lawrie and Ledward 2006). It is an important property as it influences the sensory properties related to tenderness, juiciness, and flavor of meat during mastication. WHC also impacts processing, as meats with low WHC tend to be inferior products. As seen in Table 3.5, the type of wood or shells used for smoking did not result in a difference in WHC amongst the samples. As the WHC and final moisture contents of the different products were either the same, or had showed very little difference, one expects that the juiciness of the products would be very similar. Thus, juiciness might be defined for example as “the amount of juice released following seven chews” (Berry and Civille 1986). The material is chewed in the mouth and subject to mechanical, both

shear and compressive, and a sensory panelist would assess the quantity of free liquid that results. The WHC mimics this process to some extent. Subsequent work (Chapter 4) also indicates that there or no perceived differences in juiciness amongst the samples.

3.3.5 Instrumental Texture Analysis of Smoked Chicken Breasts

The effects of sawdust species on instrumental texture measurements of smoked chicken breasts are presented in Table 3.6. Values of 'tenderness' as measured by shear testing ranged from 7.70 to 8.20 N. However, there were no significant differences in shear values due to the different types of smoke source. Shear values in this study were measured by the Meullenet Owens razor blade method. Compared to other shear methods (Warner-Bratzler, Allo-Kramer) it was shown that this method best correlates with descriptive sensory measures of tenderness, while all shear methods correlate with consumer perception of tenderness in chicken breast meat (Cavitt et al. 2004, 2005). Subsequent chapters will address the issue of perceived hardness in the various smoked chicken breast samples.

3.4 CONCLUSIONS

In general, the physical properties of the chicken breast smoked with pecan shells, hickory, mesquite or apple tree wood did not vary much amongst the samples. There were no differences in water activity, cook loss, water-holding capacity and instrumental texture measurements. There were some small differences in moisture, pH and color. Of these, differences in color were most noteworthy. Chicken breast cooked and smoked with pecan shells were slightly darker and a bit redder as compared to the other samples. Thus, subsequent work

will focus on whether differences in appearance are noticeable by consumers, and whether it meets demand and expectations compared to existing products.

Subsequent chapters will study whether textural attributes such as tenderness and juiciness were measurably different when tested by trained panelists, and whether these findings were consistent with instrumental measures of shear values and WHC. In addition, the issue of whether these factors affected consumer acceptability of the products will be addressed. In addition, the impact of smoke source on flavor was not pursued in this study and will be addressed by sensory studies and GC-MS measures of volatile compounds in the finished product.

At this stage, the studies on smoked meat were designed to test the feasibility of using pecan shells as a source of smoke and whether products could be produced that had appropriate physical properties. As the products did not differ much in their properties, and subsequent chapters will show that the sensory properties were acceptable, it will make sense to optimize the conditions of smoking. Thus, future work will focus on the particle size of shells and moisture content that will result in the best products and provide the most efficient production of smoke.

Table 3.1 Moisture Content and Ash Content of Different Kinds of Sawdust

Sample	Moisture Content (g/100g)	Ash Content (g/100g)
Hickory	$8.89\pm0.63^{[b]}$	$0.97\pm0.01^{[c]}$
Pecan Shell	$11.43\pm0.27^{[a]}$	$2.53\pm0.14^{[b]}$
Mesquite	$8.58\pm0.10^{[b, c]}$	$3.11\pm0.02^{[a]}$
Apple Tree Wood	$7.94\pm0.20^{[c]}$	$2.57\pm0.01^{[b]}$

Values in each column not followed by the same letter are significantly different (Tukey's test, $p<0.05$); n=3

Table 3.2 Particle Size Cumulative Distribution of Different Kinds of Sawdust

Category	Hickory Sawdust (%)	Pecan Shells (%)	Mesquite Sawdust (%)	Apple Tree Sawdust (%)
Coarse	$13.61\pm0.77^{[c]}$	$70.43\pm4.24^{[a]}$	$30.56\pm2.57^{[b]}$	$10.02\pm1.11^{[d]}$
Medium	$32.55\pm1.05^{[a]}$	$10.81\pm1.57^{[d]}$	$23.64\pm2.30^{[b]}$	$19.26\pm1.68^{[c]}$
Fine	$53.84\pm1.83^{[b]}$	$18.77\pm4.70^{[d]}$	$45.79\pm4.34^{[c]}$	$70.71\pm2.76^{[a]}$

Values in each column not followed by the same letter are significantly different (Tukey's test, $p<0.05$); n=3

Table 3.3 Moisture Properties and pH of Cooked Chicken Breast Smoked with Different Wood or Shell Sources

Treatment	Moisture Content (g/100g)	Water Activity	pH
Hickory Smoked	71.72±0.25[a]	0.954±0.01[a]	6.43±0.00[a]
Pecan Shell Smoked	70.66±0.11[c]	0.954±0.00[a]	6.41±0.01[a, b]
Mesquite Smoked	71.17±0.07[b]	0.953±0.02[a]	6.39±0.01[b]
Apple Wood Smoked	70.13±0.19[d]	0.952±0.01[a]	6.39±0.01[b]

Values in each column not followed by the same letter are significantly different (Tukey's test, $p<0.05$); n=3

Table 3.4 CIE Color Values of Smoked Chicken Breast

Treatment	L*	a*	b*
Hickory Smoked	76.57±0.51[a]	3.18±0.62[c]	22.17±2.19[b]
Pecan Shell Smoked	72.86±0.55[c]	5.71±0.56[a]	25.70±0.78[a]
Mesquite Smoked	75.96±0.85[a, b]	3.94±0.22[b, c]	20.65±0.62[b]
Apple Wood Smoked	74.94±0.73[b]	4.19±0.83[b]	22.52±0.76[b]

Values in each column not followed by the same letter are significantly different (Tukey's test, $p<0.05$); n=3, color measurements were taken at 3 random locations across each product surface on 3 smoked chicken breasts pieces.

Table 3.5 Cook Loss and Water-holding Capacity of Smoked Chicken Breasts

Treatment	Cooking Loss (%)	WHC (%)
Hickory Smoked	26.91±1.23[a]	11.87±2.21[a]
Pecan Shell Smoked	26.22±1.13[a]	9.91±1.03[a]
Mesquite Smoked	26.13±0.93[a]	9.68±0.92[a]
Apple Wood Smoked	27.03±1.21[a]	10.21±0.40[a]

Values in each column not followed by the same letter are significantly different (Tukey's test, $p<0.05$); cooking loss (n=20, WHC (n=5).

Table 3.6 Shear Values for Smoked Chicken Breasts

Treatment	Instrumental Tenderness (N)
Hickory Smoked	7.72±0.40[a]
Pecan Shell Smoked	8.17±0.96[a]
Mesquite Smoked	8.20±0.55[a]
Apple Wood Smoked	8.16±1.06[a]

Values in each column not followed by the same letter are significantly different (Tukey's test, $p<0.05$); n=10.

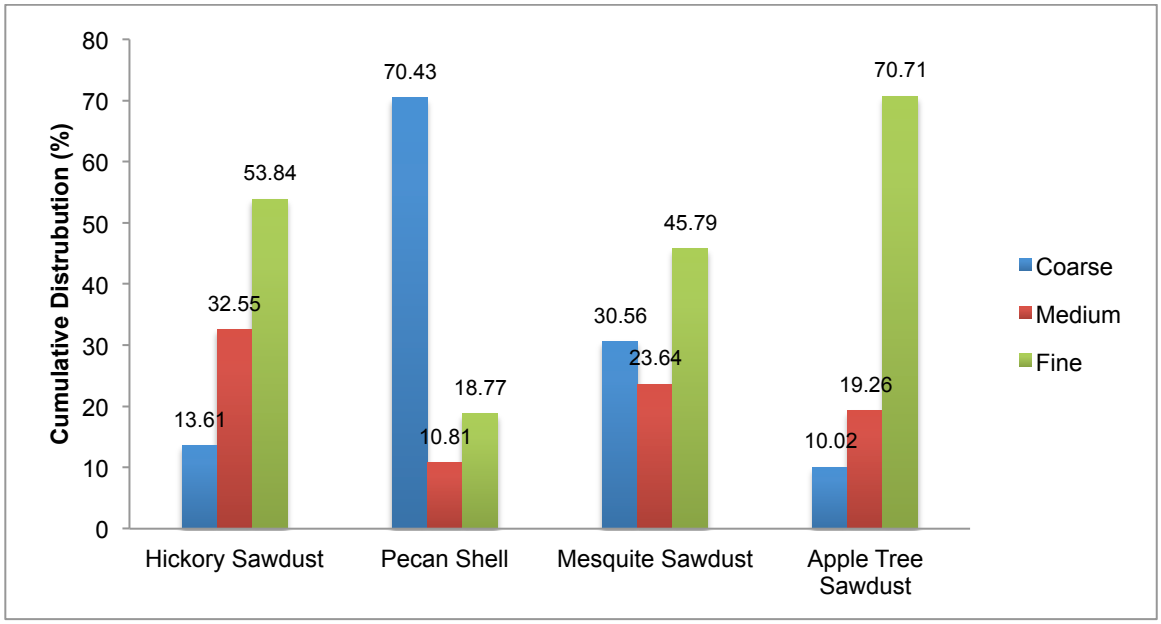
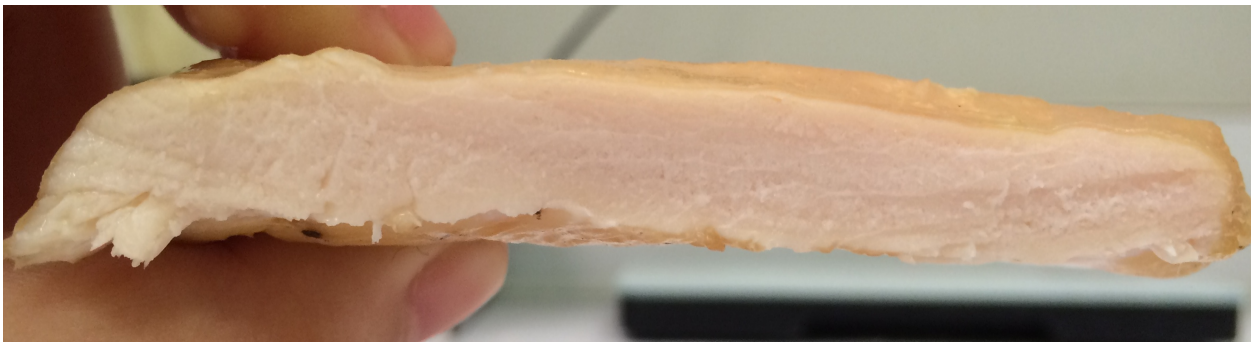


Figure 3.1 Cumulative Particle Size Distributions of Wood Sawdust and Pecan Shells

(a) Hickory Smoked Chicken Breast



(b) Pecan Shell Smoked Chicken Breast

(c) Apple Tree Smoked Chicken Breast

(d) Mesquite Smoked Chicken Breast

Figure 3.2 Cross-Section of Chicken Breasts Smoked with Four Different Wood Sources

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CHAPTER 4

Chicken Breast Prepared with Pecan Shells or Woods: Descriptive Sensory Analysis, Consumer Acceptance and Relationships with Phenolic Compounds Analyzed by Solid-Phase Microextraction in Combination with Gas Chromatography-Mass Spectrometry

4.1 INTRODUCTION

Pecan shells are a waste product resulting from the pecan shelling process. It is estimated that 52, 416 tons of pecan shells were generated in 2012 from shelling operations in the United States (NASS 2015). Generally, this material is of little value and often discarded. There has been some research in developing pecan shells as a source of biofuels, as activated carbon for water filtration (Bansode et al. 2003), for sugar refining processes (Rao et al. 2000), and for geosmin adsorption (Ng et al. 2002). For many years, people in the southeastern U.S. have used pecan wood for smoking and grilling meats. There have also been some anecdotal mentions of using pecan shells as a source of smoke, especially for grilled chicken and pork. It is said that the pecan shells can add a soft sweet-smoke flavor to meat, but so far there has been no scientific literature investigating the use of shells for smoked meat products. In addition, there has been no work showing how products made with shells as a smoke source would compare with those made using common wood sources.

Smoking has been used to preserve meats for thousands of years, while also imparting a characteristic flavor, aroma, and color to the product. While traditionally smoking was mostly used for meat products such as ham, bacon or fish, it has more recently been used for products such as cheese and sea salt. Preservation and increased shelf-life result from the effects of

cooking and dehydration, along with antimicrobial and antioxidant compounds created and delivered by the smoke. These mainly volatile molecules result from the breakdown of cellulose, hemicellulose and lignin, and consist of carbonyl compounds, carboxylic acids, and phenolic compounds (Baltes et al. 1981; Pszczola 1995). Smoke can inhibit the growth of several microorganisms due to compounds such as guaiacol, pyrocatachol, pyrogallol and formaldehyde (Ciecierska and Obiedziński 2007). To date, most studies have dealt with the generation of antimicrobial compounds from woods during smoking, but not from nut shells. Interestingly, there have been some studies to suggest that antimicrobial compounds can be extracted from pecan shells, but these are not the same antimicrobial materials generated by the combustion of tough biomaterials (Babu et al. 2013).

Most studies on smoked foods have emphasized the chemical constituents produced in the smoking process and how these carry over into the food. Little work has been done with descriptive analyses or consumer acceptability of smoked chicken products. Descriptive sensory analysis is a method to obtain detailed descriptions of the aroma, flavor and texture of a product. Using trained panelists, the important sensory attributes of a product are discovered, and a scale designed (along with references) so that panelists can assess the intensity of these attributes in select products. While it allows mapping product similarities and differences, it also enables researchers to discover relationships between specific ingredients or process variables with the sensory attributes of a product (Stone and Sidel 2004). In contrast, consumer studies use a larger number of untrained panelists to assess the degree of likeability, acceptability or preference of foods. In this study, we use both approaches to evaluate the comparative effects of pecan shells and select woods on the sensory properties of smoked chicken breast.

In the previous chapter, water-holding capacity (WHC) and instrumental texture analysis were used to determine factors related to the juiciness and tenderness of smoked chicken breast meat prepared in an equivalent manner. WHC relates to juiciness as the water released from the sample during mastication is determined by how well the meat holds onto water during the breakdown of the muscle structure under compressive forces. Similarly, instrumentally measured shear forces have been shown to provide good measure of tenderness in meat products. Several methods have been developed to measure shear forces such as the Warner-Bratzler shear (WBS) and Allo-Kramer shear (AKS) (Cavitt et al. 2004, 2005). An alternative method, using a narrow razor blade was proposed by Lee et al. (2009) and shown to provide good correlation with the sensory perception of tenderness in chicken breast.

With respect to flavor, it is generally believed that phenolic compounds produced by the pyrolysis of lignin give the characteristic flavor if smoked meats, while also providing bacteriostatic and antioxidant activity. Extensive studies on the volatile compounds of smoked foods have been done, but only a few focus on the contribution to the characteristic smoky flavor of smoked chicken (Guillén and Errecalde 2002; Guillén et al. 2004; Xie et al. 2008). Based on these studies, the key compounds responsible for smoky flavor are phenol, 2-methylphenol, 3-methylphenol, guaiacol, 2,6-dimethylphenol (syringol), 3-ethylphenol, 4-Ethylphenol, 2,3-Dimethylphenol, 4-Ethylphenol, 4-Methylguaiacol (cresol), 4-Vinylguaiacol, and 4-propylguaiacol (Xie et al. 2008; Hollenbeck 1994). The liquid-liquid extraction or steam distillation of compounds from a food matrix, followed by gas chromatography- mass spectrometry (GC-MS) is one of most the powerful methods to identify and quantify phenolic substances (Serot 2003).

An alternative to exhaustive extraction of compounds is through the solid-phase microextraction (SPME). Here, the analytes are adsorbed onto a fiber coated with a polymer that can adsorb volatile compounds from the sample headspace. The SPME method has the advantage of being a simple and solvent-free system that can be used with small sample volumes. In addition, many samples can be prepared in a given time, and the method can provide great sensitivity (Harmon 1997; Kataoka et al. 2000; Pillonel et al. 2002; Reineccius 2002). It assumes, however, that an equilibrium is reached between the analyte in the sample and in the fiber coating.

A few studies have used SPME-GC techniques to analyze compounds in smoked products. Most researchers have found that SPME fibers coated with polyacrylate (85 μm film thickness) have been best suited for analyzing volatile compounds in smoked foods such including black bream, rainbow trout and smoked herring (Serot 2003; Guillén and Errecalde 2002; Guillén et al. 2004). Amongst different fibers tested, the polyacrylate coating gave a broader volatility range and high retention ability of smoke components. The conditions for extracting key phenolic compounds varied somewhat, but usually involved extraction on to the fiber at temperatures of 50-60 °C, for 50-60 min. Thus, in this study a range of conditions were tested to optimize the extraction temperature, extraction time, and the amount of sample.

In summary, the aim of this work was to investigate the use of pecan shells as a smoke source on the flavor and acceptability of smoked chicken breast, and in comparison with common smoking woods such as hickory, mesquite and apple tree wood. Thus, samples of smoked chicken breast were assessed using Spectrum ^{TM} descriptive sensory analysis to describe the flavor and texture attributes of samples. This involved developing a complete terminology for flavor and texture attributes, along with definitions and intensity references. Next, the sensory

acceptability of pecan-shell smoked chicken was determined along with that for hickory, mesquite or apple tree wood smoked chicken breast. Finally, SPME coupled with GC-MS was used to analyze the key phenolic compounds elated to smoky flavor. Where possible, the results are examined to determine how chemical or physical properties determine the descriptive or acceptability attributes.

4.2 MATERIALS AND METHODS

4.2.1 Preparation of Raw Materials

The pecan shells used in this study were obtained from South Georgia Pecan Company (Valdosta, GA). The pecan shells were harvested in 2014 and contained assorted pecan varieties, most notably 'Elliot', 'Desirable' and 'Stuart' cultivars. Once obtained, the shells were further grounded to small pieces by a Mill-Rite grinder (Westinghouse Electric Corporation, Pittsburgh, PA) and screened by shaking in a round separator (SWECO Model Vibro-Energy®, Macon, GA) for twenty minutes in order to remove soil and debris from the shells. The shells were kept in sealed bags at room temperature prior to use. Other types of sawdust used in this study, including hickory, mesquite and apple tree, were purchased from Frantz Company (Butler, WI) and stored in the same conditions as the pecan shells.

Frozen marinated chicken breasts were supplied from Wayne Poultry Company (Pendergras, GA) and stored at -20 °C until furthering processing. Before the day of smoking, the chicken breasts were thawed overnight in a refrigerator at 4 °C. Excess connective tissue was removed, and the pieces rinsed with tap water and patted dry.

4.2.2 Smokehouse Processing

Cooking and smoking of the chicken pieces was conducted in an ALKAR smokehouse (Model DEC, Lodi, WI), with dimensions of 1.4 m deep, 1.5 m in width and 1.9 m tall. Smoke was produced from an automatic smoke generator (ALKAR SmokeMaster^{TM} III, Lodi, WI) by pyrolysis of each sawdust at a temperature of 365 °C, with a feed rate of ~500 g/h maintained for each treatment. Smoking was done with either pecan shells or sawdust of hickory, mesquite or apple tree wood.

For each trial 40 pieces of chicken breasts were processed. Pieces were layered onto 4 shelves of a trolley, and the trolley rolled into the smokehouse. Thermocouples were placed into the center of meat in order to monitor temperatures during processing. The cooking/smoking protocol was as follows: an initial cook step at 60 °C for 20 min; a smoke/cook step at 66 °C, 40 % RH, and air speed of 2m/s; a final cook step at 77 °C and 88% RH until the inner temperature of the breast meat reached 74 °C and was maintained for 20 min. The smoked chicken breasts were cooled to room temperature (~25 °C) and immediately vacuum-packed (Henkelman Vacuum Machine 600, UK). Samples were stored refrigerated at 2 °C until subsequent analysis. The smokehouse was thoroughly cleaned by chemical detergent (Momar NOX Smoke II, Atlanta, GA) running under cleaning program between each batch.

4.2.3 Descriptive Sensory Analysis

Panelists Training

For descriptive sensory analyses, 8 panelists (2 male, 6 female, aged between 40 and 65 years) trained on sensory descriptors for more than 120 h with a variety of food products in USDA-ARS sensory lab. For current study, the panelists received further orientation on smoked

poultry products.

The method used for descriptive sensory analyses was an adaption of the Spectrum™ method (Munoz and Civille 1992), and used 15-point numerical scales with references (where 0= "not detected" and 15 = "extremely strong"). The panelists received sensory training in fifteen sessions, with 2 h total per session. Descriptive terms were generated during the first orientation session, while the definitions and references for the attributes were developed in the next four sessions. A scorecard was developed which included the sequence of attributes, evaluation protocols and intensity scale design. The references were developed based on those found in the literature for similar attributes, standing USDA lab protocols, or developed directly by the trained panelists. For references derived from the literature, panelists were polled to see if they agreed the reference fit with the specific attribute in the smoked chicken breast. Where necessary, panelists also helped determine the appropriate intensity to be assigned to the reference sample. For references incorporated from the Spectrum™ or USDA protocols, intensity levels were used as described by the method.

During training, intensity scores were discussed amongst panelists in order to reach a consensus. The descriptors chosen related to both flavor and texture of smoked chicken breasts. For flavor, the attributes were sweet, salty, smoky, earthy and woody; for texture, cohesiveness, hardness, juiciness and chewiness. Next, eight specific training sessions were implemented in both group and individual forms for practice. Test samples included commercially available meat products. Finally, two sessions of performance tests were conducted to ensure assessors were ready to perform the final descriptive analysis.

Sample Serving and Evaluation

Once training was complete, panelists were tasked to evaluate the smoked chicken breast samples. Smoked samples that had been stored frozen were allowed to thaw overnight at 4 °C. All samples were cut into 1.3 cm cubes and placed into odor-free 90 mL covered plastic cups on the morning of evaluation. All samples and references were served at room temperature. Each panelist received 7 pieces of the product for evaluation, with additional samples available as needed.

Four 1.5 h sessions were organized to test all the samples. Sessions were performed in individual partitioned booths under white, using a computerized scoring system.

Panelists rated each attribute on a continuous scale presented on the computer screen. Intensities were scored on a 15-point numerical scale divided into half-point increments, with 0 meaning absence of the attribute and 15 being extremely strong. Purified water, unsalted crackers, apples and lemon sorbet were provided to cleanse the palate between samples. Panelists were asked to rinse their mouth with water between each sample.

All of the samples were coded with three-digit random numbers, and a completely randomized design was used to determine the serving order of different samples. The form containing definitions about the reference materials (Table 4.1) as well as the actual reference material was shown to the panelist during the session.

Data Analysis

Compusense Five software (Version 4.6.702; Compusense, Guelph, Canada) was used to collect data. The experiment design was completely randomized with four repetitions. The mean, standard deviation, and one-way analysis of variance (ANOVA) was determined for each

sensory attribute using SAS statistic software (Version 9.3, SAS Institute, Cary, NC). Any statistical differences amongst the means was determined by Tukey's tests at a level of $p<0.05$.

4.2.4 Consumer Evaluation

Sample Preparation

Pre- frozen smoked samples were thawed overnight at 4 °C. Samples were heated on stainless perforated platforms by steam generated underneath. Samples were immediately cut into 1.3 cm cubes and placed in 90 ml plastic cups for serving. Each consumer panelist received one piece of product for evaluation, although additional samples were available as needed. Samples were served at 68±2 °C.

Panelists

Panelists were recruited from amongst students, staff and faculty at the University of Georgia. The panelists were required to meet all eligibility requirements, particularly that they had no tree nut (or other) allergies, and eat smoked foods at least once a week. In total, 106 panelists (42 males, 64 females with ages ranging from 18 to 64 years old) participated in the test. The consumer evaluation took place in the sensory labs of the Food Processing Research and Development Laboratory at the University of Georgia over a 1-day period.

Test Design and Sample Evaluation

Samples were assessed using a 9-point hedonic scale (1=dislike extremely and 9= like extremely) and each consumer evaluated each sample for flavor, texture, appearance and overall acceptability. Consumers evaluated samples in individual booths illuminated with white lighting.

Each sample was labeled with a randomized three-digit code and served in a random and nearly balanced order. Purified water and unsalted crackers were provided to cleanse the palate between samples.

Data Collection and Analysis

A printed questionnaire was used to collect the data. Analysis of Variance (ANOVA) and Tukey's test ($p<0.05$) was used to test if significant differences existed among the smoked samples with respect to acceptability scores. All statistical analyses were performed using SAS statistic software (Version 9.3, SAS Institute, Cary, NC).

4.2.5 Analysis of Phenolic Flavor Compounds

Solid Phase Microextraction Procedures

For SPME extraction of volatile compounds, a Supelco (Bellefonte, PA) 85 μm polyacrylate fiber was chosen based on previous research (Serot 2003; Guillén and Errecalde 2002; Guillén et al. 2004). Based on these studies, extraction performance was first evaluated at 45, 50 or 60 °C, for 50, 55 or 60 min, and using either 1 or 3.5 g sample. The best match (>800) with the mass spectral library (NIST MS Search 2.0 Version, Gaithersburg, MD) for the target compounds was found for extractions at 60 °C for 50 min, using a 3.5 g sample. Attempts to shorten the extraction time to 45 min resulted in a poorer match for targeted phenolic compounds (<750). Consequently, an aliquot of 3.5 g ground chicken breast was weighed into 40 mL screw top vial (Supelco), and 1.2 g sodium chloride (Fisher Scientific) and 10 mL of deionized water was added into vial in order to increase the ratio of volatiles in the headspace. A 0.2 μL portion of 2-chlorophenol (Sigma-Aldrich, St. Louis, MO) in methanol (Fisher Scientific) (2 mg/mL)

was added as an internal standard, and then the vial sealed with a crimp cap with a silicone septum (Thermo Scientific, Suwanee, GA). After the contents were fully mixed, the vial was placed in a water bath (Fisher Scientific) at 60 °C; the contents were stirred with a magnetic stir bar and the sample allowed to equilibrate for 15 min.

Prior to initial use, the SPME fiber was conditioned in the injection port of the gas chromatograph at 280 °C for 1 hour. Subsequently, the fiber was inserted into a manual holder and inserted into the heated vial, so as to expose the fiber to the vial headspace. The fiber and sample were allowed to equilibrate over 50 min at 60 °C. A diagram of SPME assembly for extraction is shown in Figure. 4.1.

Gas Chromatography-Mass Spectrometry

GC-MS analysis was performed on a Clarus® 680 gas chromatograph coupled with a Clarus® SQ 8T mass spectrometry (Perkin Elmer, Waltham, MA). Samples were run on a fused silica capillary column Elite-5MS (30 m length, 0.25 mm i.d., and 0.25µm film thickness; Perkin Elmer, Waltham, MA). The SPME fiber was desorbed for 5 min at 280 °C in the injection port in the splitless mode. The temperature of both the injector and detector was kept at 280 °C. The carrier gas was helium and the flow rate was 1 mL/ min at a head pressure of 80 kPa. The GC oven was programmed as follows. An initial temperature of 50 °C for 1 min was followed by a 4 °C/min temperature ramp to 80 °C. After a 1 min hold at 80 °C, the temperature was increased at 2 °C/min to 150 °C followed by a 1 min hold. Finally, the temperature was increased 10 °C/min to 280 °C, followed by a 10 min hold.

The mass spectromery operated in electron impact (EI) mode with an electron energy of 70 eV. The chromatographs were recorded by monitoring the total ion current in 50-620 mass

ranges. After desorption, the fiber was maintained in the injection port an additional 10 min to assure desorption was completed.

Identification and Semi-quantification

Select phenolic compounds were tentatively identified by comparing their retention times with those published in the literature and by their mass spectra compared with those in a commercial library (NIST MS Search 2.0 Version, Gaithersburg, MD). Based on previous research, the dominant phenolic substances related to smoky flavors were phenol, 2-methylphenol, 3-methylphenol, guaiacol, 2,6-dimethylphenol (syringol), 3-ethylphenol, 4-ethylphenol, 2,3-dimethylphenol, 4-ethylphenol, 4-methylguaiacol (cresol), 4-vinylguaiacol, and 4-propylguaiacol (Xie et al. 2008; Hollenbeck 1994). A match score of greater than 780 with the mass spectra library was used as a criteria for successful identification.

Semi-quantitative determinations were obtained using 2-chlorophenol as an internal standard. The quantity of each phenolic substance was calculated by its GC-peak area relative to the GC-peak area of the internal standard. Each treatment was carried out in triplicate.

Statistic Analysis

All samples were carried out in triplicate, and results were reported as means ± standard deviation. Analysis of Variance (ANOVA) and Tukey's test ($p<0.05$) was used to test if significant differences existed between each compounds. All analyses were performed using SAS statistic software (Version 9.3, SAS Institute, Cary, NC).

4.3 RESULTS AND DISCUSSION

4.3.1 Descriptive Sensory Analysis

The descriptive flavor and texture attributes for smoked chicken breast and their intensity scores (n=32) are listed in Table 4.2. Sweet, salty, earthy, woody, cohesiveness, juiciness and chewiness were not significantly different amongst the four samples. Smoky flavor had the highest intensity scores amongst other flavor attributes, followed by woody (3.28-3.95), salty (2.89-3.27) and earthy (2.38-2.85). While giving some sense of their importance, the values cannot be strictly compared across attributes as they are based upon different reference samples and scales. The hickory-smoked chicken breasts had the strongest smoky flavor (5.28). Smoky flavor intensity for pecan-shell (4.29) smoked chicken was similar to that from apple tree wood (4.18). The mesquite-smoked chicken had the lightest smoky flavor amongst all samples.

Amongst the textural attributes, hardness had the highest scores (5.62-6.07) followed by cohesiveness (5.14-5.90), chewiness (3.97-4.39) and juiciness (2.04-2.85). Only hardness showed any significant differences amongst the treatments. The hickory-smoked chicken was rated the hardest intensity and apple wood smoked chicken the least. It has been shown that hardness is inversely related to tenderness (Cross et al. 1986). However, previous results (Chapter 3) showed that there was no difference in meat tenderness, as measured by shear values, for the 4 differently treated chicken breast samples. Interestingly, hardness was correlated with moisture content ($r^2 = 0.84$), even though there was not a big difference in moisture contents (70.1-71.2 g/100g) amongst the samples. Juiciness was not particularly correlated with previous measured values of water-holding capacity ($r^2 = 0.23$).

As compared with others, pecan shell smoked chicken exhibited moderate scores in both flavor and texture attributes (Figure 4.2 and 4.3). It is worth mentioning that pecan shell did not

provide any extra sweet flavor to the smoked chicken as compared to samples smoked with other woods.

4.3.2 Consumer Evaluation

A summary of the consumer liking scores for chicken breasts smoked with four kinds of sawdust is shown in Table 4.3. In general, all scores fell in the acceptable range of the scale, with overall likeability ranging from 5.82 to 6.43. Samples smoked with mesquite had the lowest overall liking, with significant differences amongst the other samples. There were no significant differences in likeability for texture or appearance amongst the four samples. On average, pecan shell smoked chicken received a moderate score (6.20), associated near a "like moderately" assessment.

With respect to flavor, the highest scores were attained for hickory, apple tree and pecan shell smoked samples (6.21-6.51), with mesquite-smoked samples rated lower in flavor likeability. In summary, the performance of pecan shell smoked chicken was acceptable in overall liking, flavor, texture and appearance, and performed similarly to other existing and popular woods for smoking.

4.3.3 Phenolic Compounds Related to Flavor

A typical gas chromatogram from each smoked chicken type is shown in Figure 4.4 (a-d). In total, five key phenolic substances were identified among the headspace volatiles from the smoked chicken samples: guaiacol, 4-methylguaiacol (creosol), 4-ethylguaiacol, 2,6-dimethoxyphenol (syringol), and iso-eugenol were found in all samples. According to previous studies, guaiacol is associated with a sweet smoky flavor; creosol is responsible for a burnt,

smoky and woody flavor; 4-ethylguaiacol has been related to a woody, spicy and smoky aroma; and syringol is associated with a burnt, woody and smoky aroma (Xie et al. 2008). Nevertheless, iso-eugenol is a key phenolic compound but there is no literature to prove it is responsible to smoky flavor. Iso-eugenol was not listed as a component of smoky aroma in those studies, but some sources indicate it imparts a spicy or floral note characteristic of clove oil.

Concentrations of the phenolic compounds recovered from the chicken breasts are summarized in Table 4.4. The total concentration of the target phenolic compounds in the four samples (hickory, pecan shells, mesquite, and apple tree wood) was 1.19, 0.59, 0.16 and 1.11 ppm of meat, respectively. The total concentration of phenolic substances (excluding iso-eugenol) relating to smoky flavor (guaiacol, 4-methylguaiacol, 4-ethylguaiacol, and 2,6-dimethoxyphenol) was 0.96, 0.44, 0.14 and 0.88 ppm of meat, respectively. In general, hickory smoked samples had higher, or the same, levels of the key phenolic compounds than the other samples. Samples smoked with mesquite generally had lower levels of the compounds than the other samples. The proportions of the compounds were not the same in all samples. Thus, levels of 2,6-dimethoxyphenol were relatively high in apple wood smoked samples, but had lesser levels of guaiacol, 4-methylguaiacol and 4-ethylguaiacol as compared to hickory smoked samples. Samples smoked with pecan shells had lower overall levels of the phenolic compounds than hickory or apple wood smoked samples, but more than for mesquite-smoked samples.

The total concentration of phenolic substances relating to smoky flavor was 2-6 times higher in the hickory-smoked chicken than in the mesquite or pecan-shell smoked breasts. These results may help to explain the sensory scores. Thus, hickory and apple wood smoked chicken had higher flavor likeability than mesquite smoked samples, and this may be related to a higher sensation of smoky flavor. Moreover, the hickory and apple wood smoked samples had higher

smoky flavor intensity as evaluated by descriptive panelists, although only mesquite had significantly lower smoky flavor than the other samples.

In addition, there was a relatively high proportion of 2,6-dimethoxyphenol in all 4 samples, with ~43-74% of total phenolic substances related to smoky flavor. Kjallstrand and Petersson (2001) showed that high levels of 2,6-dimethoxyphenol are associated with the burning of hardwoods. Again, hickory and apple wood smoked samples had the highest levels of 2,6-dimethoxyphenol.

As noted, the hickory smoked chicken samples had the greatest overall levels of the phenolic compounds. In addition to contributing to smoky flavor, these may also provide bacteriostatic and antioxidant activity to the meat. The pecan shell smoked chicken had moderate levels (0.59±0.04 ppm of sample), less than hickory or apple wood but greater than for mesquite.

While hickory had the highest levels of the identified compounds, it does not mean that other compounds are not present that contribute to smoky flavor, or to a more unique flavor profile associated with a particular wood. As Figure 4.4 shows, other volatile compounds were present in the chicken breasts smoked with the four different woods. In addition to the noted phenolic substances, a few additional volatile compounds were detected in all four samples, but only few had good match with a commercial library. The compound 1, 2, 4-trimethoxybenzene was found in all four samples with good match, and which has been characterized to have a "musty dry" odor (Vázquez-Araújo et al. 2011). The molecule 1,2,3- trimethoxy-5-methylbenzene was also detected, which was usually put into a miscellaneous or ethers category in other research (Montazeri et al. 2013; Serot 2003). However, it has been identified as an important compound in fermented teas (Lv et al. 2014).

4.4 CONCLUSION

The species of wood (or shells) did manifest some differences in the descriptive sensory analyses, consumer likeability and the amount and type of phenolic compounds in the differently smoked samples. From descriptive sensory analysis, only smoky and hardness attributes were different amongst the samples. Amongst the samples, mesquite smoked chicken had lower overall likeability than the others, and this was likely related to a lower smoky flavor. Analyses of the phenolic compounds showed that hickory smoked samples generally had higher levels, while mesquite had the lowest, and again this may be responsible for the lower sensation of smoky flavor. In general, pecan shell smoked chicken breast had comparably good likeability, smoky flavor, and moderate levels of smoke-related phenolic compounds expected in smoked meats.

For future studies, it would be recommended to investigate the relationships between woody and earthy flavors and their corresponding chemical compounds. It may also prove useful to develop mathematical models to relate sensory characteristic of smoke chicken products with the volatile aroma components. Also, sensory studies dealing with pecan shells and other woods may be useful to determine its optimal use in blends of these products.

Table 4.1 Descriptive Sensory Attributes, Definitions and Reference Materials:

(a) Flavor Attributes

Attribute	Definition	Reference Materials
Sweet	Taste simulated by sugar and other sweet substances.	Sucrose solution (20 g/L) = 2 Sucrose solution (50 g/L) = 5 Sucrose solution (100 g/L) = 10 Sucrose solution (160 g/L) = 15
Salt	Taste simulated by sodium salts.	Salt Solution (2 g/L) = 2 Salt Solution (5 g/L) = 5 Salt Solution (10 g/L) = 10 Salt Solution (15 g/L) = 15
Smoky	Aromatic associated with any type of smoke flavor[a]	Wright's Liquid Smoke (0.125 ml/L) = 1 Oscar Mayer Hot Dog (0.5 inch slice) [b] = 5.3 Wright's Liquid Smoke Solution (5 mL/L) = 7.7 (7.7 mL/L) = 10.5 (10 mL/L) = 14
Earthy	Aromatic associated with damp, wet soil[c] (Chew references to 10 times to score your perception).	Lima Beans[c] = 3 Red Beet (Peeled 0.5 inch cube) = 6.5 Raw Potato (Peeled 0.5 inch cube) = 7.4 Button Mushroom (0.5 inch cube) = 8
Woody	Brown, musty aromatics associated with very fibrous plants and bark[c].	Skippy Creamy Peanut Butter[b] = 2.0 Fresh Asparagus Stem (0.5 inch slice) [c] = 6.0 Walnuts = 9.0 Almond Butter, Natural = 11.2

(b) Texture Attributes

Attribute	Definition	Reference Materials
Cohesiveness	The distance to bite into a sample before it breaks, cracks, or crumbles (the first bite)	Cornbread (0.5 inch cube) = 1 Soft Sugar Cookies (6 pieces/cookie) = 3.2 American Cheese (0.5 inch cube) = 5.0 Soft Pretzel (Heat at 204°C for 3.5 min, cool 4 min, 0.5 inch slice) = 8.0 Raisins = 10.0 Freedent Gum = 15.0
Hardness	The force to compress the sample with the molars during the first few bites (the first few bites).	Cream Cheese (0.5 inch cube) = 1 American Cheese (0.5 inch cube) = 4.5 Olive = 6.0 Bordeux Cookies = 8.0 Roasted Peanuts = 9.5 Almonds =11 Life Savers = 14.5
Juiciness	The amount of moisture coming from the sample during the first 5 chews (the first few bites).	Banana (0.5 inch cube) = 1.0 Button Mushroom (0.5 inch cube) = 4.0 Cucumber (0.5 inch cube) = 8.0 Watermelon (0.5 inch cube) = 15.0
Chewiness	The cumulative attribute from the first chew through the last chew. The amount of work to chew the sample to get it to the point of swallowing (Evaluated the time of swallow).	Rye Bread = 1.8 Gum Drops = 5.8 Tootsie Roll = 12.7

[a] Adapted from Limpawattana and Shewfelt (2010); [b] adapted from Meilgaard et al. (2007); [c] adapted from Park et al.(2009).

Table 4.2 Descriptive Sensory Scores for Chicken Breasts Prepared with Four Different Smoke Sources (n=32)

Sensory Attribute	Hickory Smoked Chicken	Pecan Shell Smoked Chicken	Mesquite Smoked Chicken	Apple Wood Smoked Chicken
Flavor				
Sweetness	1.79±0.75[a]	1.48±0.95[a]	1.59±0.93[a]	1.46±0.63[a]
Salty	3.27±1.06[a]	2.89±1.24[a]	2.94±0.91[a]	3.09±0.94[a]
Smoky	5.28±1.70[a]	4.29±1.58[a,b]	4.18±1.54[b]	4.52±1.36[a,b]
Woody	3.95±1.44[a]	3.50±1.62[a]	3.37±1.40[a]	3.28±1.29[a]
Earth	2.85±1.07[a]	2.73±1.13[a]	2.53±1.24[a]	2.38±0.82[a]
Texture				
Cohesiveness	5.90±1.23[a]	5.43±1.32[a]	5.41±1.25[a]	5.14±1.27[a]
Hardness	6.07±0.56[a]	5.92±0.73[a,b]	5.90±0.66[a,b]	5.62±0.74[b]
Juiciness	2.07±1.17[a]	2.04±1.36[a]	2.67±1.26[a]	2.85±1.37[a]
Chewiness	4.39±1.06[a]	4.18±1.22[a]	4.14±0.91[a]	3.97±0.90[a]

Means in row with different superscripts (a and b) represent significant difference ($p \leq 0.05$). Means in same row followed by the same letter are not significantly different at $p \leq 0.05$ (Tukey's test).

Table 4.3 Summary of Mean Likeability Scores for Chicken Breasts Prepared with Four Smoke Sources

	Smoke Source			
	Hickory	Pecan Shell	Mesquite	Apple Wood
Overall	6.35±1.43[a]	6.20±1.37[a,b]	5.82±1.61[b]	6.43±1.53[a]
Flavor	6.46±1.35[a]	6.21±1.48[a,b]	5.84±1.65[b]	6.51±1.59[a]
Texture	6.15±1.80[a]	5.85±1.76[a]	5.75±1.86[a]	6.26±1.97[a]
Appearance	6.26±1.48[a]	6.08±1.55[a]	5.76±1.66[a]	5.95±1.67[a]

The table presents a mean of 106 panelists. Results are presented as mean ± standard deviation.
Means with the same letter within each column are not significantly different at 5% by Tukey test.
Hedonic scale was based on a 9-point scale (1= dislike extremely; 5= neither like nor dislike; 9= like extremely).
A total of 106 consumers participated in the test. The consumers group included 42 males and 64 females with ages ranging from 18 to 64 years old.

Table 4.4 Volatile Phenolic Compounds from Chicken Breast Meat Prepared with Different Smoke Sources

RT (min) [a]	Phenolic Compounds	CAS#	Concentration (ppm)			
			Hickory	Pecan Shell	Mesquite	Apple Wood
12.50	Guaiacol*	90-05-1	0.16±0.06[a]	0.06±0.01[b,c]	0.04±0.01[c]	0.13±0.03[a,b]
17.71	4-methylguaiacol*	93-51-6	0.11±0.04[a]	0.06±0.01[a,b]	0.02±0.01[b]	0.05±0.01[b]
22.50	4-ethylguaiacol*	2785-89-9	0.06±0.02[a]	0.03±0.01[b,c]	0.02±0.00[c]	0.05±0.01[a,b]
26.80	2,6-dimethoxyphenol*	91-10-1	0.62±0.08[a]	0.29±0.02[b]	0.06±0.02[c]	0.65±0.01[a]
32.72	iso-eugenol	97-54-1	0.24±0.04[a]	0.14±0.01[b]	0.03±0.00[c]	0.23±0.04[a]
	Subtotal [b]		0.96±0.17[a]	0.44±0.03[b]	0.14±0.01[c]	0.88±0.03[a]
	Total [c]		1.19±0.21[a]	0.59±0.04[b]	0.16±0.01[c]	1.11±0.07[a]

Results are presented as mean ± standard deviation, which were derived from three replicates of calculated quantities by relating peak areas to that of 2-chlorophenol internal standard.

Means in row with different superscripts (a, b and c) represent significant difference ($p \leq 0.05$). Means in same row followed by the same letter are not significantly different at $p \leq 0.05$ (Tukey's t test).

[a] RT, retention time; [b] Subtotal, the sum concentration of smoky phenolic compounds; [c] total, the total concentration of phenolic compounds detected in samples.

Asterisked compounds were contributed to smoky flavor according to previous research (Xie et al. 2008)

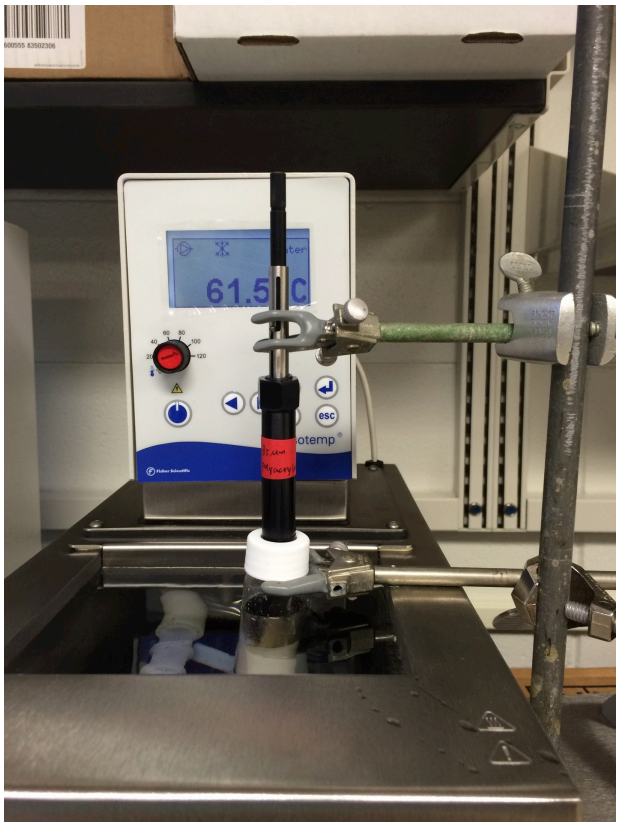


Figure 4.1. Assembly for Extracting Wood Smoke Volatiles onto SPME Fibers

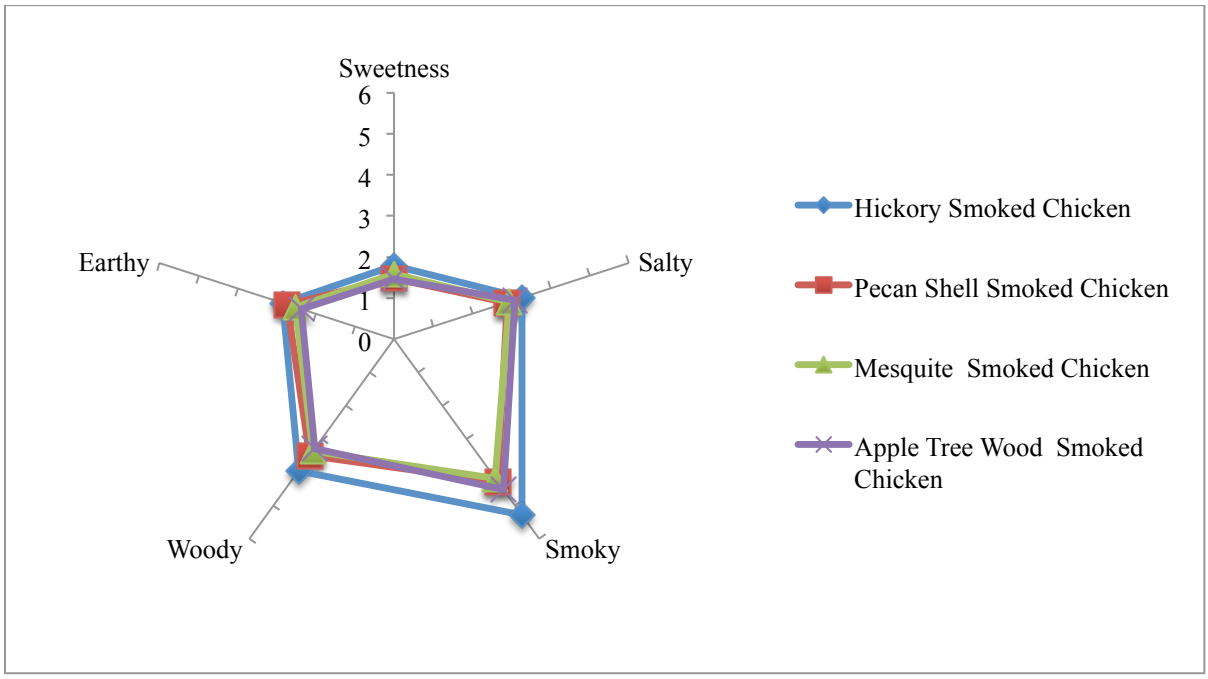


Figure 4.2 Spider Plot of the Mean Intensity of Flavor Descriptors Found in Chicken Breasts Smoked with Four Different Sawdust Species

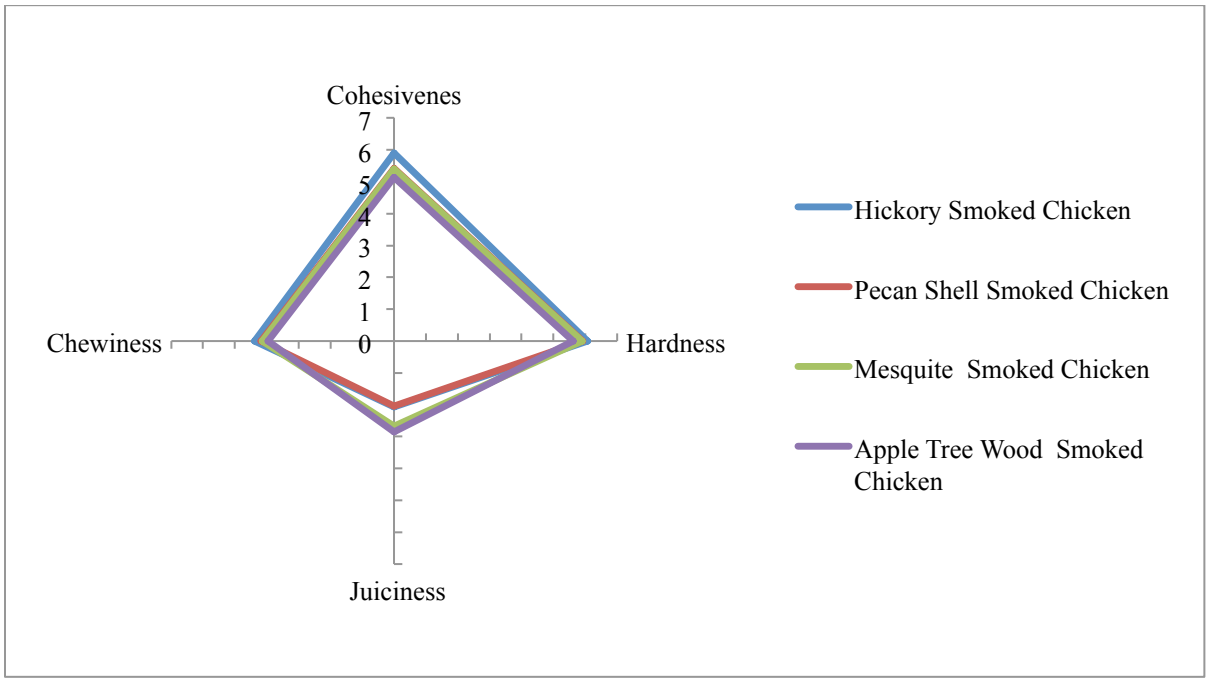
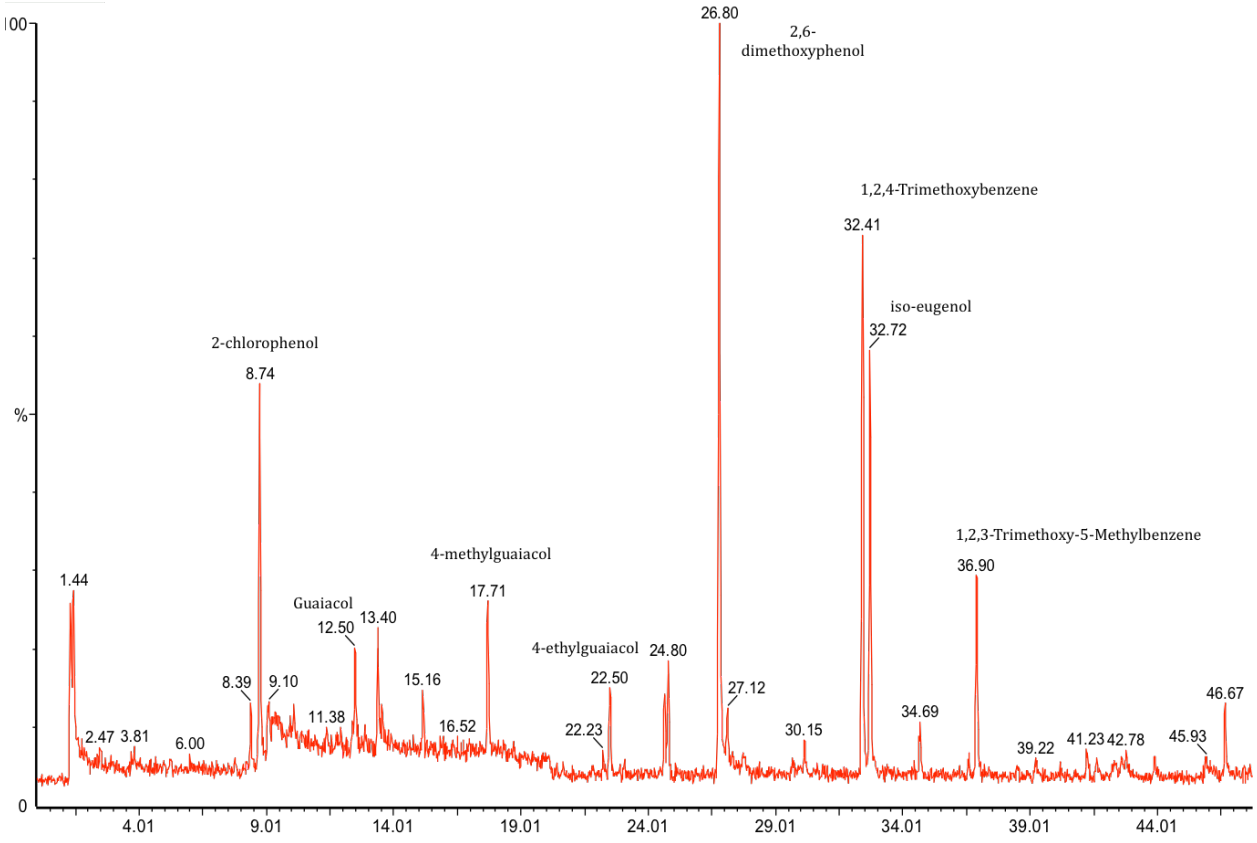
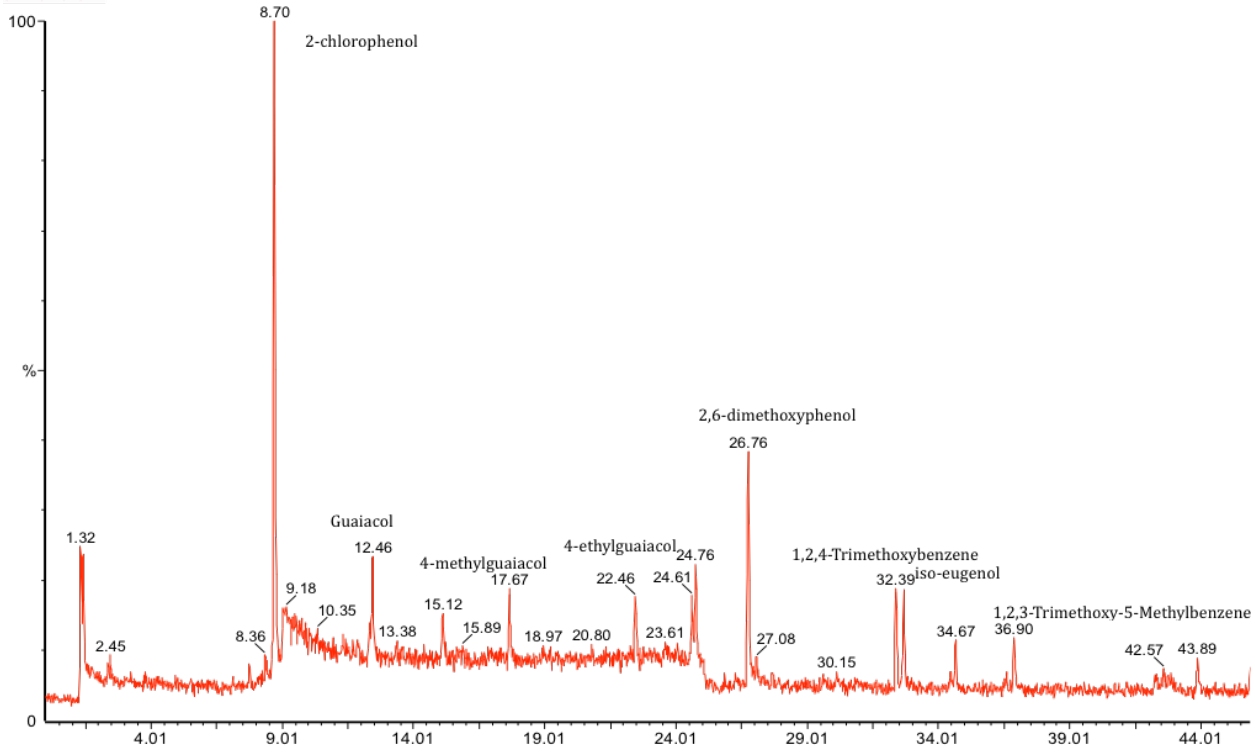


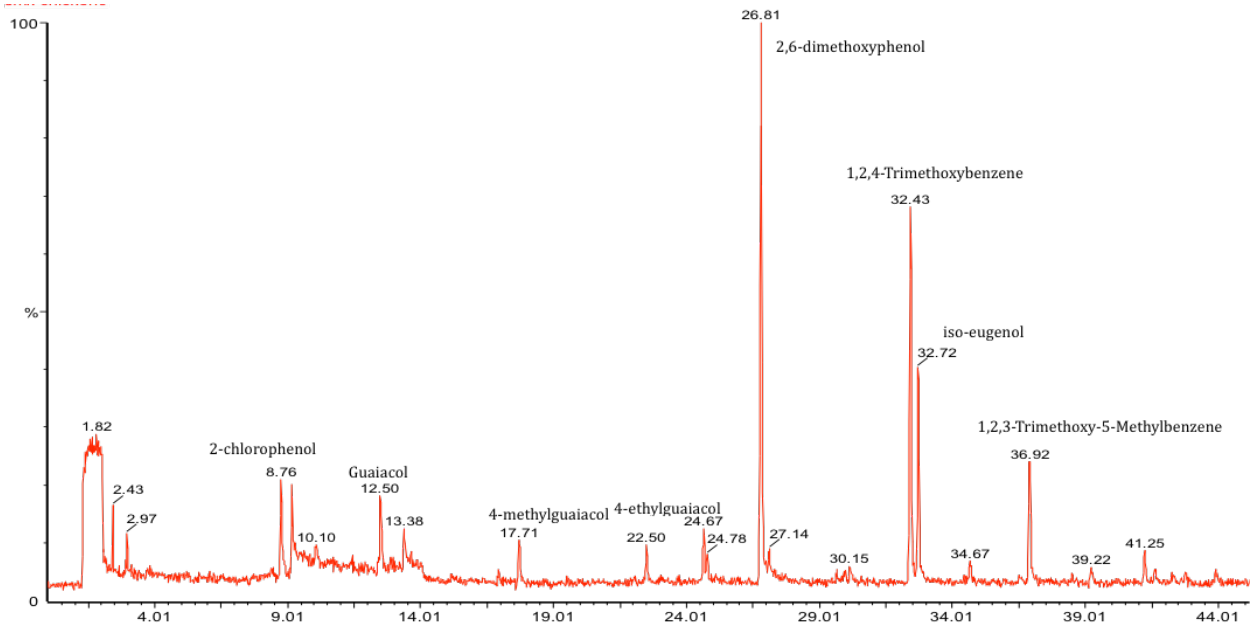
Figure 4.3 Spider Plot of the Mean Intensity of Texture Descriptors Found in Chicken Breasts Smoked with Four Different Sawdust Species



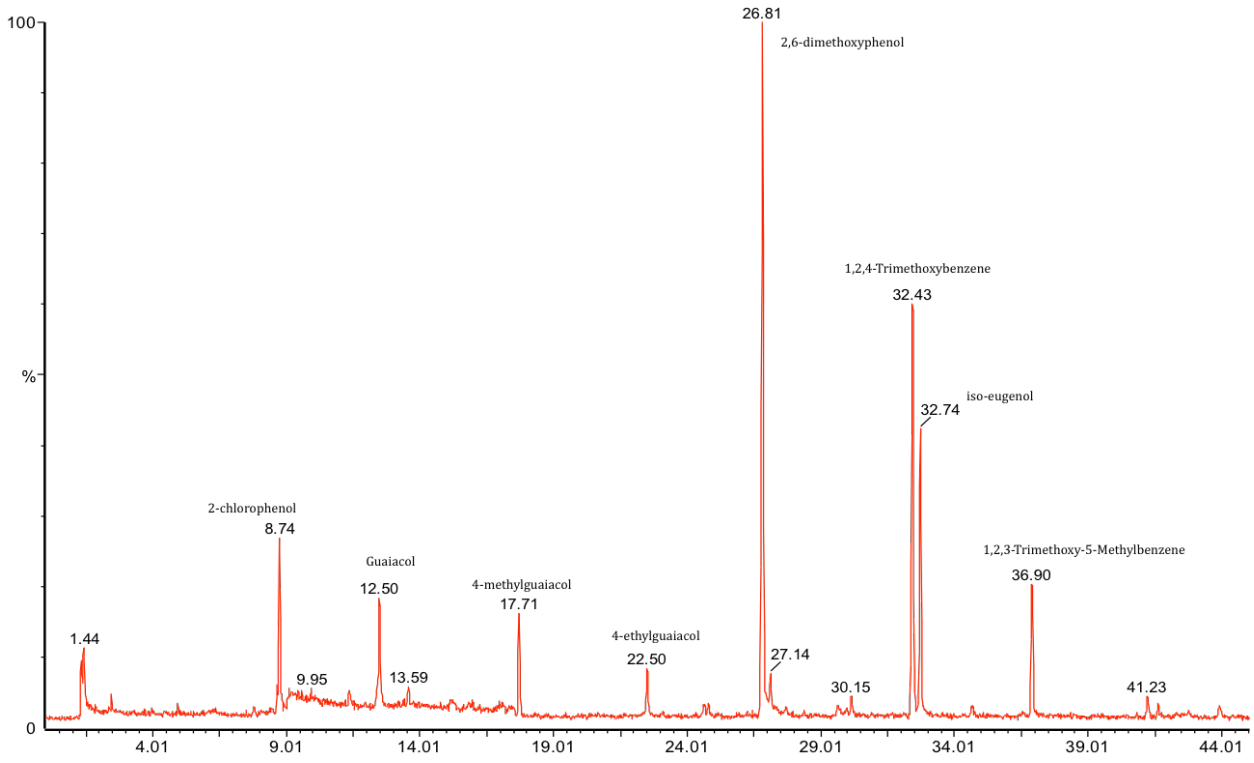
(a) Pecan Shells Smoked Chicken Breast



(b) Mesquite Smoked Chicken Breast



(c) Apple Tree Wood Smoked Chicken Breast



(d) Hickory Smoked Chicken Breast

Figure 4.4 Gas Chromatograms of Chicken Breasts Smoked with Four Different Woods

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CHAPTER 5

CONCLUSION

The goal of this study was to evaluate the use of pecan shells as a relatively cheap source of combustible material for smoked foods. Chicken breast meat was chosen as a model as it has a relatively low intensity flavor profile. The pecan shells did prove to be a reasonable choice for this application. The already broken shells could be easily transformed to pieces that could be fed to an industrial smoke generator. Chicken breast made with pecan shells had similar physical properties compared to product made with other common wood sources (hickory, mesquite and apple tree). In addition, samples prepared with pecan shells compared well with other samples in sensory tests.

In chapter three, quality parameters including moisture content, water activity, color, cook loss, water-holding capacity and instrumental texture analysis were investigated in chicken products smoked with four different varieties of sawdust (pecan shell, hickory, mesquite and apple tree wood). Also, some basic analysis of the smoke sources were measured, including moisture content, ash content and particle size distribution of these four varieties of smoke sources. Results indicated that only the moisture content, pH and color were significantly affected by wood species, and only in relatively small amounts. Pecan shell smoked chicken breasts did have a darker and redder color than other smoked samples.

In chapter four, we examined the comprehensive sensory properties of the smoked chicken breasts prepared from the four smoke sources, including both descriptive sensory and consumer evaluation study. In addition, this study also established a headspace sampling procedure, using SPME fibers and GC-MS for analysis of phenolic compounds related to smoky flavor, including guaiacol, 4-methylguaiacol, 4-ethylguaiacol and 2,6-dimethoxyphenol. In this

work, we developed references and intensity specifications for smoked chicken products (sweetness, salty, smoky, woody and earthy flavor; cohesiveness, hardness, juiciness and chewiness texture profiles). Of these, only the 'smoky' and 'hardness' attributes were different amongst the samples. Hickory smoked chicken breasts were characterized the strongest smoky flavor, while the smoky flavor in both pecan shell and apple tree wood smoked chicken breasts were similar in intensity. From the consumer evaluations, all scores generally fell in the acceptable range of the scale. There was no significant difference in texture and appearance between the four samples. On average, pecan shell smoked chicken received scores indicative of "like moderately". The hickory-smoked chicken received the highest score of overall liking, but the differences were not great between hickory and pecan-shell. A higher level of smoky flavors was perceived as favorable in terms of consumer satisfaction with the smoked products. With respect to headspace analyses, there were five phenolic substances detected of which four were most responsible for smoky flavor based on previous research. These included guaiacol, 4-methylguaiacol (cresol), 4-ethylguaiacol, 2,6-dimethoxyphenol (syringol). It was shown that hickory smoked chicken samples had the highest concentration of phenolic compounds relating to smoky flavor (0.96 ppm of meat) while pecan shell smoked chicken breasts had 0.44 ng phenolic substances per mg. Overall levels of smoke related phenolics correlated with perceived 'smoky' flavor evaluated by the sensory panel.

Overall, pecan shells can be used as a value-added smoke wood in smoked food products. Future studies might focus on means to improve performance and efficiency, as by investigating the influence of particle size or moisture content on physical and sensorial properties. It may also be helpful to gather more fundamental information of the properties of the shells, such as porosity, flow properties or major constituents including cellulose, hemicellulose and lignin.

Moreover, it would be useful to investigate the relationships between woody and earthy flavor and their corresponding chemical compounds, and to establish models that provide a quantitative link between sensory characteristics of smoked chicken product and the aroma volatile components. Also, it may be worthwhile to investigate blends of pecan shells with other woods, to determine if these may provide high quality products at lower cost.