EVALUATING THE EFFECT OF OIL CONTENT ON THE QUALITY AND PHYSICAL PROPERTIES OF PECAN-BASED NUT BUTTER [Carya illinoinensis (Wangenh.) K. Koch]

by

EMILY ALICE WAGENER

(Under the Direction of William Kerr)

ABSTRACT

Pecan-based nut butters were made using ground sugar, flour salt, partially defatted pecan flour at two different roast levels (unroasted and roasted at 163°C for 8 minutes), and varied levels of pecan oil (50%, 55%, 60%, 65%, and 70%). The purpose of this research was to determine which oil concentration range was the most consumer acceptable and physically similar to commercial peanut butters (JIF Creamy, Skippy Natural Creamy, and Smucker’s Natural Creamy). The full-fat 70% was the least acceptable in consumer sensory testing and 60% was the most acceptable. Textural and rheological testing showed that the most significant increase in firmness and decrease in spreadability occurred between samples with 55%-60% oil. The 60% roasted and unroasted and 55% roasted samples most closely mimicked with the commercial butters. Overall, the optimum oil range was found to be between 55%-60% in pecan-based nut butter.

INDEX WORDS: pecan butter, nut butter, pecans, rheology, texture analysis, hedonic consumer panel, pecan oil
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by

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Bachelor of Science, Clemson University, 2012

A Thesis Submitted to the Graduate Faculty of The University of Georgia in Partial Fulfillment of the Requirements for the Degree

MASTER OF SCIENCE

ATHENS, GEORGIA

2015
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August 2015
DEDICATION

I dedicate this thesis to my parents, who have always supported me in everything that I have accomplished and reminded me to keep things in perspective. No matter what, “Life is Good.” I wouldn’t have gone nearly as far as I have at this stage of my life without your love.
ACKNOWLEDGEMENTS

This research and thesis would not be possible without the help and assistance of numerous folks.Thank you to my major professor, Dr. Kerr, for being patient and guiding me as I stumbled through the design and execution of the many moving parts of this project. Thank you to my committee members, Dr. Reyes and Dr. Swanson, for their expert input and encouragement. Thank you to the support personnel at UGA (Carl Ruiz, Lisa Cash, Lisa Porterfield, Marian Wendinger, Daniel Morris, Eve Mayes and Karen Simmons) for their assistance with various elements of both this project and navigating graduate school. Thank you to Dr. Hurst and Dr. Mohan for the unique opportunities and challenges that I was able to take on as an Extension Outreach graduate assistant. Thank you to Ross Barghi of Just Born Skincare for taking so much time to press the dozens of pounds of pecan midgets that I brought to Atlanta. Thank you to Jeff Worn and Clint Willis of the South Georgia Pecan Company for shipping me at least a hundred pounds of pecan midgets without hesitation. Thank you to my hard working labmates for supporting me throughout this project and helping serve samples at my sensory panel: Juzhong Tan, Andrea Jackson, Cathy Micali, and Wu. Thank you to my former labmates Audrey Varner, Meg Liu, and Kat Kitchen for their guidance during my first year of graduate school. Thank you to Dr. Swanson’s lab for allowing me to use various pieces of equipment for my sensory panel. Thank you to my fantastic Extension coworker Laurie Leveille for taking on extra work to ensure I could finish graduate school at a reasonable time and for helping me label dozens of scorecareds and sample cups. Finally, thank you to all the wonderful graduate students in the UGA food science department for helping me maintain a few shreds of sanity during the tough times of
graduate school, especially my fellow pecan scholars Elizabeth Carr and MaryBeth Kellett.
You all are some of the smartest and most creative guys and gals I have ever been to a dinner party with. I will never just bring chips and salsa to a social shindig again.
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CHAPTER 1
INTRODUCTION

Overview

The pecan \( Carya illinoinensis \) (Wangenh.) K. Koch] is a nut that is a member of the hickory Juglandaceae family and native to North America. It is suggested that the word “pecan” originated from the Algonquin language and means “requiring a stone to crack,” because Native Americans were among the first recorded groups to consume the nuts before they were discovered by European settlers and traded throughout the world [1]. These deciduous trees can grow up to 70 feet tall and reach maturity after seven years if grafted and longer if grown from seedlings [2]. Modern farming practices have allowed for the development of different cultivars, which determine if the trees will bear fruit annually or biennially [1]. Over a hundred cultivars have been developed by farmers and registered [3]. Pecan halves, pieces, or a combination of both are graded as “U.S. No. 1” for the highest quality products or “Commercial” for lower quality products by the United States Department of Agriculture (USDA) [4]. A notable Georgia cultivar is the ‘Desirable,’ named for both its meaty seed for the nut itself and thin shell for ease of removal during processing. The first harvest of this cultivar was reportedly developed around 1915 [5].

Pecans in particular have an excellent nutritional value of macronutrients such as protein and dietary fiber and micronutrients such as Vitamin E (a natural antioxidant) and manganese. Their caloric content comes from a high lipid content of 65%-75% depending on the cultivar and harvesting conditions [6]. The ‘Desirable’ cultivar is reported to contain approximately 70-72% lipids [7]. Furthermore, the FDA has recognized them as “heart healthy”, thus boosting their
image in the public eye [6]. In light of these benefits, pecan markets have grown significantly both domestically and globally [8].

Nut butters are an easy way for consumers to enjoy the aforementioned health benefits. As of 2011, the USDA Commercial Item Description mandates that nut butters comprise at least 90% nuts, whereas spreads are 40% nut products. Smooth butters must have a fine consistency with few noticeable particles in the product, and chunky/crunchy nut butters should have a fine background texture with a moderate portion of nut pieces larger than 1/16” dispersed throughout. There are several types of nut butters and spreads currently on the market, including peanut, almond, hazelnut, and cashew [9]. These market segments are active and are expected to grow in the coming years [10].

Nut butters can be characterized using both sensory and instrumental methods. Untrained consumer sensory panels are used to determine a sample population’s consensus on acceptability of a product, thus predicting the perceptions of the general population. Simplified surveys use hedonic ratings that indicate a panelist’s degree of like or dislike of certain aspects of a food sample. These results can then be quantified for further statistical analysis [11]. Spreadability, firmness, and adhesiveness attributes of a food product can be measured using a texture analyzer equipped with a cone spreadability rig that measures forces of penetration, work of shear, and adhesiveness as the male cone is lowered into a female cone that contains the sample [12]. Rheological analysis using various geometries can show the flow behavior of a food product by subjecting a sample to various types of shear and measuring the resulting forces of deformation and stress. This information is useful for predicting how a product will physically behave during commercial processing [13].
Rationale

There has been an increasing demand for nut-based butters and spreads in the marketplace. Pecans are not currently present, however, due to their high oil content that results in a runny product when ground raw. This research sought to create a novel process for crafting pecan butter by separating the raw nuts into partially defatted flour and oil, then recombining them at varying oil contents. Ultimately, a target oil level range was determined for future commercialization work using sensory and instrumental methods.

Objectives

The purpose of this research was:

- To evaluate the pecan butter formulations with varied oil content for physical characteristics such as color, particle size, water activity, firmness, adhesiveness, and spreadability as compared to commercial peanut butters.
- To evaluate the pecan butter formulations with varied oil content for consumer acceptability characteristics such as texture, consistency, spreadability, flavor, and overall acceptability.
- To evaluate the pecan butter formulations with varied oil content for rheological characteristics of shear stress as a function of shear rate to determine apparent viscosity and viscoelastic properties when subjected to increasing shear stresses.
References


4. United States Department of Agriculture Agricultural Marketing Service United States Standards for Grades of Shelled Pecans 01/19917.


CHAPTER 2
REVIEW OF LITERATURE

History of Pecans

Pecans [Carya illinoensis (Wangenh.) K. Koch.] were originally of interest to the to Native Americans settled along the central region in the United States in Mississippi, Missouri, central Texas, and northeastern Mexico. These tribes valued pecans for their ease of shelling, nutritional value, and superior taste when compared to other nuts of the Carya genus. As a result, tribes focused on cultivating pecan trees for use as a food source. European colonists also discovered the value of the nut and began planting pecan trees from New York down to the Gulf of Mexico during the 1700s and early 1800s. Early growers were limited by unsuccessful farming techniques until commercial tree grafting procedures were developed by farmers in the Southeastern United States. As demand for the crops grew, so did the number of farms, which pushed out towards the Western region of the United States. Pecans later became one of the few native North American crops to spread across the world [1].
**Pecan Grading**

Shelled pecans are graded based on quality and classified based on size by the USDA. Other factors that determine the final grade include color, visible defects, and development of the kernel. Inspectors assign grades to lots based on random samples drawn from locations spread throughout storage containers in a processing facility as long as each container clearly contains nuts of approximately the same size [2]. Table 2.1 outlines the grading specifications for pecan halves ranging from “Mammoth” for the largest nuts to “Midget” for the smallest nuts and how many of each size classification are sold in a pound.

Table 2.1 USDA Size Classifications for Pecan Halves$^a$

<table>
<thead>
<tr>
<th>Size Classifications for Halves</th>
<th>Number of Halves per Pound</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mammoth</td>
<td>250 or less</td>
</tr>
<tr>
<td>Junior Mammoth</td>
<td>251 - 300</td>
</tr>
<tr>
<td>Jumbo</td>
<td>301 - 350</td>
</tr>
<tr>
<td>Extra Large</td>
<td>351 - 450</td>
</tr>
<tr>
<td>Large</td>
<td>451 - 550</td>
</tr>
<tr>
<td>Medium</td>
<td>551 - 650</td>
</tr>
<tr>
<td>Small (topper)</td>
<td>651 - 750</td>
</tr>
<tr>
<td>Midget</td>
<td>751 - or more</td>
</tr>
</tbody>
</table>

$^a$Data from the USDA Standards for Grades of Shelled Pecans [2]
Table 2.2 outlines grading specifications for pecan pieces based on size diameters. The “Mammoth” pieces are the largest and “Granules” are the smallest sizes measured as maximum to minimum allowable diameters of pecan pieces to pass through a screen during the shelling process [2]. Sampled lots of pecans are also assessed based on an application of standards for development of the kernel as outlined in Table 2.3.

Table 2.2 USDA size classifications for pecan pieces

<table>
<thead>
<tr>
<th>Size Classifications</th>
<th>Maximum Diameter</th>
<th>Minimum Diameter</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mammoth Pieces</td>
<td>No Limitation</td>
<td>8/16”</td>
</tr>
<tr>
<td>Extra Large Pieces</td>
<td>9/16”</td>
<td>7/16”</td>
</tr>
<tr>
<td>Halves and Pieces</td>
<td>No Limitation</td>
<td>5/16”</td>
</tr>
<tr>
<td>Large Pieces</td>
<td>8/16”</td>
<td>5/16”</td>
</tr>
<tr>
<td>Medium Pieces</td>
<td>6/16”</td>
<td>3/16”</td>
</tr>
<tr>
<td>Small Pieces</td>
<td>4/16”</td>
<td>2/16”</td>
</tr>
<tr>
<td>Midget Pieces</td>
<td>3/16”</td>
<td>1/16”</td>
</tr>
<tr>
<td>Granules</td>
<td>2/16”</td>
<td>1/16”</td>
</tr>
</tbody>
</table>

aData from the USDA Standards for Grades of Shelled Pecans [2]
Table 2.3 USDA application definitions for standards of pecans\textsuperscript{a}

<table>
<thead>
<tr>
<th>Definition</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>Half-Kernel</td>
<td>One of the separated halves of an entire pecan kernel with not more than one-eighth of its original volume missing, exclusive of the portion which formerly connected the two halves of the kernel.</td>
</tr>
<tr>
<td>Piece</td>
<td>A portion of a kernel which is less than seven-eighths of a half-kernel, but which will not pass through a round opening two-sixteenths inch in diameter.</td>
</tr>
<tr>
<td>Particles and Dust</td>
<td>For all size designations except &quot;midget pieces&quot; and &quot;granules,&quot; fragments of kernels which will pass through a round opening two-sixteenths inch in diameter.</td>
</tr>
<tr>
<td>Well Dried</td>
<td>The portion of kernel is firm and crisp, not pliable or leathery.</td>
</tr>
<tr>
<td>Fairly Well Developed</td>
<td>The kernel has at least a moderate amount of meat in proportion to its width and length.</td>
</tr>
<tr>
<td>Poorly Developed</td>
<td>The kernel has a small amount of meat in proportion to its width and length.</td>
</tr>
<tr>
<td>Fairly Uniform in Color</td>
<td>Ninety percent or more of the kernels in the lot have skin color within the range of one or two color classifications.</td>
</tr>
<tr>
<td>Fairly Uniform in Size</td>
<td>In a representative sample of 100 halves, the 10 smallest halves weigh not less than one-half as much as the 10 largest halves.</td>
</tr>
</tbody>
</table>

\textsuperscript{a}Data from the USDA Standards for Grades of Shelled Pecans [2]

*Nutritional Properties of Pecans*

Like most edible nuts, pecans are considered to be high in a variety of nutrients. Both roasted and unroasted pecan nutritional content is summarized in Table 2.4 below as reported by the USDA Nutrient Database for Standard Reference, Release 27. The only significant change between raw and roasted pecans is in the moisture content, which is 3.52\% for raw and is
reduced to 1.12% in the roasted nuts, and all other macronutrients are approximately the same between the two types. Carbohydrates are reported to be 13.70g on average, which is low overall, but the fiber content at an average of 9.5g is almost 40% of the daily value based on a 2,000 calorie diet. The calorie count for 100g of pecans is between 691 and 710 kcal. This is not surprising considering pecans are predominantly comprised of lipids at over 70% for both raw and dry roasted types (Table 2.4) [3].

Table 2.4 proximate composition per 100g of pecans

<table>
<thead>
<tr>
<th>Proximate Composition</th>
<th>Raw&lt;sup&gt;a&lt;/sup&gt;</th>
<th>Dry Roasted&lt;sup&gt;a&lt;/sup&gt;</th>
</tr>
</thead>
<tbody>
<tr>
<td>Water</td>
<td>3.52g</td>
<td>1.12g</td>
</tr>
<tr>
<td>Energy (kcal)</td>
<td>691 kcal</td>
<td>710 kcal</td>
</tr>
<tr>
<td>Total Lipid</td>
<td>71.97g</td>
<td>74.27g</td>
</tr>
<tr>
<td>Carbohydrate</td>
<td>13.86g</td>
<td>13.55g</td>
</tr>
<tr>
<td>Fiber</td>
<td>9.6g</td>
<td>9.4g</td>
</tr>
<tr>
<td>Sugars</td>
<td>3.97g</td>
<td>4.06g</td>
</tr>
</tbody>
</table>

<sup>a</sup>Data from the USDA National Nutrient Database for Standard Reference, Release 27 [3]

Pecans, like all tree nuts, are high in fat. Total lipid content can vary between 65-75% depending on the pecan cultivar and harvest conditions of the orchard as shown by Santerre’s study [1]. As shown in Table 2.5, the crude lipid content is primarily comprised of monounsaturated fats at over 50%, followed by polyunsaturated fats at approximately 40% and saturated fats at under 10% [3]. A study conducted by Ryan, Gavin, and others in Ireland found that the pecans had the highest ratio of saturated to unsaturated fats at 13.54 when compared to cashews, pistachios, and Brazil nuts [4].
Table 2.5 Lipid compositions per 100g of pecans

<table>
<thead>
<tr>
<th>Lipid Type</th>
<th>Raw[^a]</th>
<th>Roasted[^a]</th>
</tr>
</thead>
<tbody>
<tr>
<td>Total Saturated</td>
<td>6.180g</td>
<td>6.283g</td>
</tr>
<tr>
<td>Total Monounsaturated</td>
<td>40.801g</td>
<td>43.957g</td>
</tr>
<tr>
<td>Total Polyunsaturated</td>
<td>21.604g</td>
<td>20.572g</td>
</tr>
</tbody>
</table>

[^a]Data from the USDA National Nutrient Database for Standard Reference, Release 27 [3]

**Health Benefits of Pecans**

Tree nuts harbor materials required to generate a new tree, thus they are rich with nutrients. Regular consumption of tree nuts over fives times per week has been linked to a variety of health benefits, including lowering a person’s risk of coronary heart disease and lowering low-density lipoprotein cholesterol levels (LDL) [5]. This significant benefit is in addition to reducing the likelihood of developing hypertension, type 2 diabetes, and other diseases due to a high content of vitamins E and K, minerals such as folate, magnesium, potassium, and selenium, and anti-inflammatory phytonutrients as reported by NHANES [6]. Pecans (along with chestnuts) have been reported to have one of the highest concentrations of antioxidants, including vitamins. This is of particular interest to researchers studying chronic inflammatory diseases related to oxidative stress [7].

**Pecan Market**

Today, pecans are one of the most popular nuts in the United States. They are grown commercially in 14 U.S. states, which accounted for 80% of the world’s total pecan production in 2012. During that same year, the state of Georgia led the production at 100 million pounds, followed by Texas at 55 million, Oklahoma at 25 million, and Arizona at 20 million pounds. The demand for pecans overseas has grown significantly in recent years, which is positive for the
market outlook. In 2012, the United States’ pecan exports were valued at 30% more than the previous year at $486.9 million. China remains the primary buyer of U.S. pecans. Hong Kong purchased $165.4 million worth of the nuts (a 69% increase from 2011 to 2012), and Vietnam bought $69.7 million’s worth (a 115% increase) [8].

*Nut Butters and Spreads*

Nut butters are a popular form of a nut-based product produced by grinding tree nuts (such as cashews, almonds, or hazelnuts) and/or peanuts into a paste, with the addition of other ingredients such as sugar, salt, and stabilizers intended to prevent oil separation [9]. These nuts are often roasted prior to grinding to add flavor as well. Nut butters can be used in a variety of ways, including dipping, spreading on bread or crackers, and as an ingredient in baked goods. A commercial product should be soft yet spreadable [10]. According to the USDA commercial item descriptions, nut butter formulas must contain 90% of the original nut product, whereas spreads require 40% of the nut to be included. There are three ways to classify the overall texture and consistency of nut butter. Smooth nut butters must be comprised of “very fine” particles that are not visible, medium nut butters may have particles that do not exceed 1/16 of an inch, and crunchy nut butters must have the consistency of a smooth butter with dispersed nut pieces larger than 1/16 of an inch throughout [9].

As of March 2015, nut butters were a $3.9 billion dollar market in the United States. This is projected to grow to $4.2 billion in 2019 according to Mintel. Millennials are the most likely to buy nut-based spreads, and they are increasingly interested in those with natural ingredients and nutritional health benefit. 75% of the adult population in the United States had reported purchasing a spreadable nut product within the last 6 months, and peanut butter was the most
often selected at 67% [11]. JIF brand peanut butter by the J.M. Smucker Company topped out the market share in 2010-2011 at $322 million followed by Skippy at $82 million [12].

Nut Butter Processing

The basic production flow of a creamy peanut butter is a process of roasting, milling, and grinding nuts into a paste with the addition of extra ingredients as outlined in the flow diagram in Figure 2.1 [13]. Roasting is often necessary for the development of flavor in nuts and occurs in peanuts at 160°C for 45-50 minutes [10]. Colloid or attrition mills are used for the primary grinding process. Medium or coarse nut butters move on after a single grinding step, but a roller mill can be used to further reduce particle size in the secondary step to produce a smooth, creamy product. Other ingredients are then added and include salt, sugar, a stabilizer, and oils that are completely hydrogenated (such as palm or soybean oil) to avoid using trans fats but are solid at room temperature, thus allowing the product to be more spreadable. Peanut butter is pumped through a processing facility at 43°C to promote flow without causing additional browning effects. Filling is done under nitrogen to prevent oxidation, and the product is allowed to solidify in the packaging for at least 24 hours prior to shipping [12].
Figure 2.1: Process flow diagram for the commercial production of nut butter\(^a\)

- Cleaning/Sorting
- Roasting/Blanching
- 1\(^{st}\) Stage Grinding
- 2\(^{nd}\) Stage Grinding
- Addition of Other Ingredients
- Filling/Packing
- Cooling

\(^a\)As depicted during private communication with Dr. Leopold Strecker, former R&D director of Unilever [12]
**Nut Butter Safety**

Despite a low water activity, nut butters still pose a threat for aflatoxin, *Salmonella*, *E. coli*, yeast, and mold contamination. Nuts used in commercial production of nut butters and spreads are usually cleaned and roasted prior to grinding to remove pathogens in addition to facilitating flavor development. Once ground, overheating nut butter will produce an undesirable burnt flavor [14]. *Salmonella* outbreaks in nut butter have occurred 5 times between 1996-2014. The largest of these was an outbreak in 2007 that affected 425 people across 44 states in the US who consumed tainted Peter Pan peanut butter that originated from a facility in Georgia [15-17]. The most recent one in 2014 occurred in both almond butter and peanut butter manufactured by nSpired Natural Foods [16]. Manufacturers of nut butter are required to test their products for tolerances of the adulterants outlined in Table 2.6 [8].

<table>
<thead>
<tr>
<th>Requirement</th>
<th>Tolerance</th>
</tr>
</thead>
<tbody>
<tr>
<td>Aflatoxin</td>
<td>≤ 15 ppb</td>
</tr>
<tr>
<td>Salt</td>
<td>≤ 1.6%</td>
</tr>
<tr>
<td>Standard Plate Count</td>
<td>≤ 10,000 per g</td>
</tr>
<tr>
<td>Yeast and Mold</td>
<td>≤ 100 per g</td>
</tr>
<tr>
<td><em>Salmonella</em></td>
<td>Negative</td>
</tr>
<tr>
<td><em>E. coli</em></td>
<td>Negative</td>
</tr>
</tbody>
</table>

*Adapted from the 2011 USDA Commercial Item Description for Nut Butters and Nut Spreads [8]*
Sensory Attributes

Sensory testing allows for non-instrumental analysis of how well human consumers will interact with a product. Trained or untrained panelists may be used to evaluate characteristics such as flavor, texture, spreadability, aroma, and purchasing decisions [10]. The 9-point hedonic scale allows for a consumer to report their acceptability of a product based on how much they like or dislike a certain aspect. The Food Research Division of the Quartermaster Food and Container Institute in Chicago first used this method of equal interval attribute scoring in the 1940s when serving products to military panelists. The lowest end of the scale is “dislike extremely” and the highest point is “like extremely”, with an option for a neutral “neither like nor dislike” in the middle. A 9-point range of choices allow for consumers to simply yet accurately record their feelings towards the attribute in question. A scale with fewer options (such as 5 or 7) may result in skewing the data as consumers usually avoid extreme responses when scoring samples [18].

A study conducted by McNeill et al (2000) sought to determine the most important attributes and descriptors to consumers when evaluating peanut butters using a focus group of 20 participants so that accurate surveys could be subsequently created. They found that desirable flavor components were saltiness, sweetness, nuttiness, and a lack of rancid or stale notes [19]. Though peanut butter is the most common nut butter in the marketplace, other types of nut butters have been evaluated as well. Researchers at the Michael Okpara University of Agriculture in Nigeria developed an almond butter that scored similarly to peanut butter in terms of flavor and overall acceptability [20]. The University of Rwanda found that soy butter also scored similarly to peanut butter, though the soy-based product is subject to oxidation [21].
Spreadability and texture are also important attributes for consumer attitudes towards nut butters and spreads [22]. The focus groups surveyed McNiell et al (2000) reported that the optimal peanut butter texture was creamy with a smooth consistency. Anything grainy, dry, stiff, or runny was considered unacceptable. Shakerardekani et al (2013) evaluated 17 formulations of pistachio spread with varying degrees of soy protein isolate and red palm oil and found that higher overall acceptability scores were negatively correlated with higher spreadability scores. A survey of 100 consumers evaluating 15 hazelnut spreads conducted by Monaco et al (2008) showed again that spreadability and overall liking were the most discriminatory variables when evaluating the nut-based products.

Textural Attributes

Instrumental assessment of texture in semisolid food products allows for the determination of a number of physical properties of a food product, including firmness, adhesiveness, and spreadability. One of the ways that these can be measured is by using a conical attachment to a texture analyzer with male and female components. The sample is loaded completely into the female component and the male component is lowered at a constant speed [23]. Spreadability is measured as the work of shear of the product as it flows out of the female cone, firmness is the force of penetration of the male cone, and adhesiveness is the force of the product acting on the male cone as it is subsequently raised [24] [23].

Multiple parameters can have an effect on the instrumental texture of a semisolid food product. Ahmed and Ali (1986) were among the first researchers to publish spreadability data using a cone rig. They found that the spreadability of peanut butters was correlated with the oil content in the formulation [25]. Shakerardekani et al (2013) performed tests using a similar
method on 17 different formulations of pistachio spreads. Those with red palm oil were
discovered to be more spreadable than those without [26].

*Rheological Testing*

Rheological evaluation of food products is a way to determine their flow behavior when
subjected to different types of stresses such as a constant shear or oscillation. Nut butters,
toothpaste, and dairy spreads are classified as “soft solids.” These types of products have a yield
stress and have elastic responses to small deformations, but larger stresses will result in complex
responses depending on the composition of the sample. Rheometers can be used to
instrumentally measure these forces and simulate processing environments that involve mixing
and pumping the food product in a commercial plant [27].

Apparent viscosity is the measure of how the shear stress of a product responds to
increasing shear rates. Newtonian fluids have a fixed proportionality between shear stress and
shear rate, whereas Non-Newtonian materials (most food products) respond differently to the
applied stresses. Bingham plastics show constant slopes, but a yield stress is required to create
flow. Pseudoplastic curves show a decreasing slope as the shear stress increases (shear
thickening behavior), and dilatent fluids show the opposite (shear thinning) [28]. These are
shown in Figure 2.2 [29].

Research performed by Monaco *et al* (2008) on hazelnut spreads found that all 15
samples exhibited shear thickening (pseudoplastic) behavior [30].
Another method of examining the rheological properties of the material is by subjecting it to increasing oscillatory small deformation stresses and measuring the elasticity as $G'$ (storage modulus) and viscosity $G''$ (loss modulus) response, also known as a stress sweep. Though semisolid foods exhibit both behaviors one generally predominates over the other, thus predicting flow of a food product in a processing environment. From this data, the linear viscoelastic region (LVR) or a product can also be predicted. This is the crucial region in which the shear stress applied will not affect the viscosity or elasticity of a product up to a certain strain, beyond which it gives way and both $G'$ and $G''$ significantly decrease [31].

Research performed by Monaco et al (2008) on hazelnut spreads found that all 15 samples exhibited shear thinning (pseudoplastic) behavior [30]. A different study by Taghizadeh
and Ravazi on showed that peanut butter performs the same way [32]. Shakerardekani’s paper (2013) showed that among 17 different formulations of pistachio spread, those with 16.7% or less red palm oil showed that G’ was the predominant element, whereas those with more red palm oil exhibited higher viscous behavior. A different set of experiments was performed by Emadzadeh et al (2012) on pistachio butter subjecting the same formulation to a stress sweeps at increasing temperatures. It was shown that the higher the temperature, the more viscous the samples behaved [33]. Citerne (2001) evaluated peanut butters subjected to varying grinding speeds and showed that G’ was positively correlated with a larger particle size [34].

References

2. United States Department of Agriculture Agricultural Marketing Service United States Standards for Grades of Shelled Pecans 01/19917.
7. Geisler, M. Pecans http://www.agmrc.org/commodities__products/nuts/pecans/#. 


26. TA Instruments Understanding Rheology of Structured Fluids.


CHAPTER 3
MATERIALS AND METHODS

Overview

Nine different formulations were evaluated for sensory, physical, and rheological attributes for this study using pecan butter that was processed using pecans pressed into partially defatted pecan flour and pecan oil components, ground sugar, and flour salt. Three additional commercial peanut butters were also evaluated for the physical and rheological portions for comparison: Smucker’s Natural Creamy and JIF Creamy both from The J.M. Smucker Company (Orrville, Ohio) and Skippy Natural Creamy from The Hormel Foods Corporation (Austin, MN).

To meet the USDA standard of identity for nut butter [1], each formulation contained over 90% pecan flour and pecan oil in combination, 6% ground sugar, and 0.25% flour salt. ‘Desirable’ pecan midget pieces were supplied by South Georgia Pecan Co (Valdosta, GA). Domino Sugar Corporation (New York, NY) supplied the sugar that was then ground into a fine powder using a hammer mill equipped with a 0.020 RD 28 screen (The Fitzpatrick Mill, Elmhurst, IL). Cargill Salt (Minneapolis, MN) supplied the flour salt. Nine pecan butter formulations were created as shown in Table 3.1 using five different oil levels (50%, 55%, 60%, 65%, and 70%) and two heat treatments (unroasted flour and flour roasted at 163°C for 8 minutes) applied to the partially defatted pecan flours at all oil levels except for the 70% to yield nine total experimental samples. The 70% oil level was chosen because the ‘Desirable’ pecan cultivar has been reported to have approximately 70-72% lipid composition, and this sample’s physical, rheological, and sensory properties reflect that of full-fat pecan butter [2].
Table 3.1: Formulas used for creating pecan-based butters with specific oil levels using partially defatted pecan flour and pecan oil (%)

<table>
<thead>
<tr>
<th></th>
<th>50% total oil</th>
<th>55% total oil</th>
<th>60% total oil</th>
<th>65% total oil</th>
<th>70% total oil</th>
</tr>
</thead>
<tbody>
<tr>
<td>Pecan Flour (45% oil)</td>
<td>79.6</td>
<td>70.5</td>
<td>61.4</td>
<td>52.3</td>
<td>43.2</td>
</tr>
<tr>
<td>Pecan Oil</td>
<td>14.2</td>
<td>23.3</td>
<td>32.4</td>
<td>41.5</td>
<td>50.6</td>
</tr>
<tr>
<td>Ground Sugar</td>
<td>6</td>
<td>6</td>
<td>6</td>
<td>6</td>
<td>6</td>
</tr>
<tr>
<td>Flour Salt</td>
<td>0.25</td>
<td>0.25</td>
<td>0.25</td>
<td>0.25</td>
<td>0.25</td>
</tr>
</tbody>
</table>

The University of Georgia Feed and Environmental Water Lab (Athens, GA) conducted a proximate analysis on both the roasted and unroasted pecan flours for basic nutritional content (Table 3.2). Both roasted and unroasted flours contained approximately 45% oil, which was factored into the final oil calculations when processing the pecan butters outlined in Table 3.1.
Table 3.2: Proximate analysis$^a$ of defatted pecan-based flours unroasted and roasted at 163°C for 8 minutes

<table>
<thead>
<tr>
<th></th>
<th>Roasted (%)</th>
<th>Unroasted (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Crude Protein</td>
<td>25.60</td>
<td>25.2</td>
</tr>
<tr>
<td>Crude Fiber</td>
<td>9.60</td>
<td>11.10</td>
</tr>
<tr>
<td>Moisture</td>
<td>4.20</td>
<td>5.00</td>
</tr>
<tr>
<td>Total Fat</td>
<td>43.98</td>
<td>44.15</td>
</tr>
<tr>
<td>Ash</td>
<td>2.99</td>
<td>2.91</td>
</tr>
<tr>
<td>Carbohydrate</td>
<td>13.63</td>
<td>11.64</td>
</tr>
</tbody>
</table>

$^a$As-sampled weight was used for calculating the composition of each partially defatted pecan flour.

Process Design for Crafting Pecan-Based Butter

The ‘Desirable’ pecan midget pieces were pressed into flour by Just Born Skincare (Atlanta, GA) using an IBG Monforts 24 mm single-screw oil press press DD85 G model running at 121°C and 2.5 speed (Nordrhein-Westfalen, Germany). The pecan oil was reserved for later use and stored in brown glass jugs under refrigerated conditions (4.4°C) to prevent oxidation. The partially defatted pecan flour was stored in -40°C conditions in 13 gallon plastic bags for the same purpose until ready for use.

On the day of processing, the partially defatted pecan flour was ground to reduce particle size using a SuperMasscolloider MKCA6-5 stone mill (Masuko Sangyo Co Ltd, Saitama-pref, Japan) with mKE stones equipped. Flour designated for roasting was spread out on aluminum sheet trays and roasted in a conventional oven at 163°C for 8 minutes and set aside to cool. A commercial food processor (Robot-Coupe R 10 V.V., Robot-Coupe, Jackson, Mississippi)
equipped with an “S” cutter blade and bowl hooked up to a Neslab RTE-140 water bath heated to 38°C (ThermoFisher Scientific, Waltham, MA) was used to blend all ingredients for each formulation weighed out as outlined in Table 3.1. The food processor was allowed to run for 2 minutes per sample at speed 3. All samples were stored in plastic buckets with snap tops in a walk-in refrigerator unit at 4.4°C until needed for further use. This process is summarized in the Figure 3.1 flowchart.

Figure 3.1: Process flow diagram for crafting pecan-based nut butter
Sensory Evaluation

Consumer Panel

The University of Georgia Institutional Review Boards on Human Subjects approved all procedures for conduction of the sensory panel. A preliminary sensory evaluation was done by a small committee to determine which pecan-based butters would be presented to the panel: 50% roasted, 50% unroasted, 60% roasted, 60% unroasted and 70% unroasted, which served as the control sample. A group of consumers was recruited (n=95, 29 male 66 female) to evaluate four samples using a 3x3 incomplete block design augmented by the control. Pecan butters were dispensed into small plastic cups (~11g per cup) labeled with a 3-digit random code and stored covered in a walk-in refrigerator overnight. All samples were allowed to come up to room temperature (21-23°C) prior to evaluation.

Sensory booths walled on three sides were lit with orange sodium lights for the duration of testing. The ninety-five consumer panelists were first given a consent form upon arrival to the testing area prior to booth assignment. Once seated in a booth, they were given a questionnaire asking basic demographic questions such as age, gender, and frequency of consuming pecans or pecan-based products (Appendix A). They then evaluated each pecan-based butter sample one at a time in a randomized order using a 9-point Hedonic scale (1=dislike extremely, 9=like extremely) for texture, flavor, spreadability, overall acceptability, and willingness to buy (Appendix B). A plastic knife and unsalted Saltine crackers were available to each panelist to spread nut butter samples on. Sparkling water and baby carrots were served as palate cleansers between samples.
Physical Evaluation

Texture Attributes

Nine pecan butter samples and three commercial peanut butters were evaluated in triplicate for firmness, spreadability (work of shear) and adhesiveness with a TA-XT2i texture analyzer (Texture Technologies Corp., Scarsdale, NY) calibrated using a 5kg load cell and a 45° cone spreadability rig. Data was analyzed from the texture analyzer using the software Texture Expert Exceed version 2.6 (Texture Technologies Corp., Scarsdale, NY). The female cone was filled to the top with each nut butter sample at room temperature (21-23°C) and leveled using a straightedge. The crossarm of the TA-XT2i was programmed to move the male cone at both a pre- and post-test speed of 5 mm/s with a button response to generate a time-force curve like the one shown in Figure 3.3 below [3]. Firmness (F1) was measured as the gram force it took to penetrate the surface of the nut butter at the peak of the curve; spreadability (work of shear, A1) was measured as the area under the curve in grams per second, and adhesiveness (A2) was measured as the absolute value of the area of the curve below the x-axis in grams per second.
Color Determination

Color of the nine pecan butter samples and three commercial butters was analyzed on the HSL (L* c* h*) scale using a chroma meter (Model CR-410 Minolta Co Ltd., Tokyo, Japan). After calibration using the white calibration plate (No. 13333105) (Konica Minolta Sensing, Inc., Tokyo, Japan), each of the twelve nut butter samples were spread out on a petri dish on top of white paper and measured in triplicate.

Water Activity

Water activity was measured for the nine pecan butter samples and three commercial butters in triplicate using an AquaLab Model Series 3 water activity meter (Decagon Devices, Inc., Pullman, WA). Nut butter samples at room temperature were spooned into a small plastic cup fit for the device and spread using a knife prior to measuring.
Particle Size Analysis

The particle size for each of the nine pecan-based butter formulations and three commercial peanut butters was measured using a LSI3 320 Laser Diffraction Particle Size Analyzer (Beckman Coulter Inc., Indianapolis, IN). Samples were dispersed in hexane and measured in duplicate. Data were analyzed using LS13320 Software version 6.01 (Beckman Coulter Inc., Indianapolis, IN) and plotted on a percent volume per channel diameter basis.

Rheological Evaluation

Stress Sweep

Oscillatory amplitude tests were performed using a Discovery HR-2 hybrid rheometer (TA Instruments, New Castle, DE) equipped with a 40mm at a 1.977° angle cone-in-plate geometry. All measurements were performed on all unroasted pecan butter samples (5 total) at 25°C with a 90s soak time at 1 Hz and measured at 10 points per decade using a logarithmic sweep mode. Data was analyzed from the hybrid rheometer using Trios software version 3.1 (TA Instruments, New Castle, DE). Table 3.3 lists the stress ranges for each sample tested based on preliminary measurements used to determine the most appropriate region for each sample. Data points were plotted using Microsoft Excel 2011 software (Microsoft, Redmond, WA).
Table 3.3: Oscillation amplitude logarithmic sweep test parameters

<table>
<thead>
<tr>
<th>Sample</th>
<th>Stress Range (Pa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>50% oil</td>
<td>1-1000</td>
</tr>
<tr>
<td>55% oil</td>
<td>0.1-1000</td>
</tr>
<tr>
<td>60% oil</td>
<td>0.001-1</td>
</tr>
<tr>
<td>65% oil</td>
<td>0.001-0.1</td>
</tr>
<tr>
<td>70% oil</td>
<td>0.001-0.1</td>
</tr>
</tbody>
</table>

Apparent Viscosity

The apparent viscosity for the nine pecan butter samples and three commercial peanut butters was measured using a Merlin VR rheometer (Rheosys LLC, Hamilton, NJ) equipped with a 4-blade vane geometry. Each test was ran at 25°C in linear mode using 50 steps of measurement in triplicate. Data was analyzed from the rheometer using the Rheosys μMicra software version 3.1 (Rheosys LLC, Hamilton, NJ) and rheological models were fit to the data using MATLAB version 2013b (The MathWorks, Natick, MA). The testing parameters for all nut butter samples are summarized in table 3.4 below. Each range was determined based on preliminary measurements on each sample.
Table 3.4: Testing parameters for apparent viscosity on pecan-based butters and commercial peanut butters

<table>
<thead>
<tr>
<th>Sample</th>
<th>Integration Time (s)</th>
<th>Shear Stress Range (s⁻¹)</th>
</tr>
</thead>
<tbody>
<tr>
<td>70% Unroasted</td>
<td>5</td>
<td>0.5-50</td>
</tr>
<tr>
<td>65% Roasted</td>
<td>5</td>
<td>0.5-50</td>
</tr>
<tr>
<td>65% Unroasted</td>
<td>5</td>
<td>0.5-50</td>
</tr>
<tr>
<td>60% Roasted</td>
<td>5</td>
<td>0.5-50</td>
</tr>
<tr>
<td>60% Unroasted</td>
<td>5</td>
<td>0.5-50</td>
</tr>
<tr>
<td>55% Roasted</td>
<td>5</td>
<td>0.5-25</td>
</tr>
<tr>
<td>55% Unroasted</td>
<td>2</td>
<td>0.5-5</td>
</tr>
<tr>
<td>50% Roasted</td>
<td>2</td>
<td>0.5-5</td>
</tr>
<tr>
<td>50% Unroasted</td>
<td>2</td>
<td>0.5-5</td>
</tr>
<tr>
<td>JIF Creamy</td>
<td>2</td>
<td>0.5-45</td>
</tr>
<tr>
<td>Smucker’s Natural Creamy</td>
<td>2</td>
<td>0.5-45</td>
</tr>
<tr>
<td>Skippy Natural Creamy</td>
<td>2</td>
<td>0.5-45</td>
</tr>
</tbody>
</table>

Statistics

Data from all sections were analyzed using JMP Pro 11 (SAS Institute Inc., Cary, NC). The Fit Y by X function on the program was used to perform a One-Way ANOVA test on all independent variables and generate a means and standard deviations table. Tukey’s HSD Test was used to determine significant differences between samples. Microsoft Excel 2011
(Microsoft, Redmond, WA) was used to organize all data tables, plot graphs, and fit models to rheological data.

References

CHAPTER 4
RESULTS AND DISCUSSION

Sensory Evaluation Results

Out of 95 panelists, 26 were male and 66 were female. The general demographic was comprised of young adults, as 84% of panelists were between the ages of 18-27. 45% reported that they buy pecans or pecan-based products “several times per month”, approximately the same amount as the group who reported purchasing them “several times per year.” This is unsurprising considering Georgia is one of the most prominent producers of pecans in the United States [1]. The acceptability scores on the Hedonic scale from the sensory panel are summarized in Table 4.1 below. A rating of “1” means that the panelist “disliked [the attribute] extremely”, whereas a 9 indicates “like extremely.”

Overall, the panelists reported the 70% oil unroasted pecan butter was between “dislike slightly” and “neither like nor dislike” with a mean overall acceptability score of 4.6 (Table 4.1). This was significantly lower than all of the other pecan butters evaluated with mean values that ranged from 5.9 for the 50% oil level butters to 6.3-6.4 for the 60% oil level butters. These samples fell in the range of “like slightly” on the scorecard. The 70% oil was clearly the thinnest sample and did not resemble typical nut butters in the marketplace.

Panelists had more diverse opinions regarding the spreadability of the pecan-based butter onto unsalted Saltine crackers (Table 4.1). The 70% unroasted sample was again significantly lower than the others, scoring between “dislike moderately” and “dislike slightly” with a mean score of 3.7. The next lowest value was the 50% roasted at 4.9 (“neither like nor dislike”). The unroasted versions of the other butters scored at a similar significance level of 5.5 and 5.9 for the 50% unroasted and 60% unroasted, respectively. The statistically highest-scoring butter for
acceptability of spreadability was 60% roasted at 6.2, “like slightly.” The 50% oil butters were the firmest and most difficult to spread, and the 60% oil samples were still runny but more spreadable than those with 70% oil.

Panelists rated the acceptability of the texture of the 70% oil pecan butter sample significantly lower than all of the other butters with an average of 4.5 in between “dislike slightly” and “neither like nor dislike” (Table 4.1). All other samples with lower oil contents had a range of means between 5.8-6.1, closest to “like slightly.” This means that the highest oil content had a least pleasant mouthfeel in the opinions of the consumer panel.

Flavor was the highest scoring attribute for acceptability ratings (Table 4.1). Both roasted and unroasted versions of the 60% oil pecan butters were the highest scoring with statistically similar means of 6.7 for the roasted version and 7.0 for the unroasted (“like moderately”). The others were also above a neutral rating. Unroasted and roasted versions of the 50% oil were scored approximately the same at 6.5 and 6.7 respectively between “like slightly” and “like moderately.” The 70% oil pecan butter was again the lowest scoring sample with a mean score of 6.0, “like slightly.”

The consumers surveyed for this panel would not regularly buy any of these pecan butters if they were on the market as-is (Table 4.1). The 60% oil and 50% oil versions were had statistically similar responses between 3.0 and 3.5, indicating “I don’t like this but would buy it occasionally” and “I like this and would buy it every now and then,” which more of an emphasis on the former. Again, the 70% oil sample scored significantly lower than the others with an average of 2.3, closest to “I would hardly ever buy this.”

These results indicate that formulations still need to be improved in order for a pecan-based butter to be more attractive to consumers. However, sensory results across the board
demonstrated that the 70% oil unroasted pecan-based butter sample scored statistically lower on all attributes, thus using partially defatted flour improves the appeal of the product (Table 4.1).
Table 4.1: Consumer acceptability of pecan butters with varied roast levels and oil contents means ± std error (n=95, 66 female 29 male)\textsuperscript{ab}

<table>
<thead>
<tr>
<th>Sample</th>
<th>Spreadability</th>
<th>Texture</th>
<th>Flavor</th>
<th>Overall Acceptability</th>
<th>Willingness to Buy</th>
</tr>
</thead>
<tbody>
<tr>
<td>70% Oil Unroasted</td>
<td>3.7 ± 0.2\textsuperscript{a}</td>
<td>4.5 ± 0.2\textsuperscript{b}</td>
<td>6.0 ± 0.2\textsuperscript{b}</td>
<td>4.6 ± 0.2\textsuperscript{b}</td>
<td>2.3 ± 0.1\textsuperscript{b}</td>
</tr>
<tr>
<td>60% Oil Roasted</td>
<td>6.2 ± 0.3\textsuperscript{a}</td>
<td>6.1 ± 0.2\textsuperscript{a}</td>
<td>6.7 ± 0.2\textsuperscript{a}</td>
<td>6.4 ± 0.2\textsuperscript{a}</td>
<td>3.5 ± 0.2\textsuperscript{a}</td>
</tr>
<tr>
<td>60% Oil Unroasted</td>
<td>5.9 ± 0.3\textsuperscript{ab}</td>
<td>5.9 ± 0.3\textsuperscript{a}</td>
<td>7.0 ± 0.2\textsuperscript{a}</td>
<td>6.3 ± 0.2\textsuperscript{a}</td>
<td>3.5 ± 0.1\textsuperscript{a}</td>
</tr>
<tr>
<td>50% Oil Roasted</td>
<td>4.9 ± 0.3\textsuperscript{b}</td>
<td>5.9 ± 0.2\textsuperscript{a}</td>
<td>6.7 ± 0.2\textsuperscript{ab}</td>
<td>5.9 ± 0.2\textsuperscript{a}</td>
<td>3.0 ± 0.1\textsuperscript{a}</td>
</tr>
<tr>
<td>50% Oil Unroasted</td>
<td>5.5 ± 0.3\textsuperscript{ab}</td>
<td>5.8 ± 0.2\textsuperscript{a}</td>
<td>6.5 ± 0.2\textsuperscript{ab}</td>
<td>5.9 ± 0.2\textsuperscript{a}</td>
<td>3.0 ± 0.2\textsuperscript{a}</td>
</tr>
</tbody>
</table>

\textsuperscript{a}Means with different letters down columns are significantly different according to Tukey’s HSD Test (p≤0.01, \(\alpha=0.05\))

\textsuperscript{b}Acceptability was measured using a 9-point Hedonic scale (1=dislike extremely, 9=like extremely)
Physical Evaluation Results

Texture Assessment

Generally, the time-force curves generated by the TAX-T2i texture analyzer (Texture Technologies Corp, Scarsdale, NY) equipped with a 45° cone spreadability rig showed that the firmness, spreadability (work of shear), and adhesiveness of 9 pecan-based butters as compared to 3 commercial peanut butters were significantly impacted by the oil content as shown in Table 4.2 below.

The firmness data indicated the amount of force that it takes for the male cone of the spreadability rig to penetrate the sample in the female cone and is the peak of the time-force curve. Nut butter firmness increased with a decrease in oil content in the formulation. The Smucker’s Natural Creamy commercial peanut butter and 70% oil pecan-based butters was the least firm ranging from 28 g to 51.8 gram force of penetration by the male cone, respectively. JIF and Skippy peanut butters were comparable in firmness to both the roasted and unroasted 60% oil pecan butters, though they were still statistically comparable to samples with higher oil content. The decline in oil content from 60% oil to 55% was the point of significant increase in firmness. The 60% oil samples were significantly softer 203.8g (roasted) to 287.7g (unroasted), but the 55% oil samples were 1647g (roasted) and 1086g (unroasted). The 50% oil samples were the firmest at 4880g for the roasted samples and 2949g for the unroasted. Both values are more than double than the 55% oil samples, which is significant (p<0.05).

The spreadability (work of shear) data corresponds with the amount of force in gram-seconds it would take to spread the nut butter with a knife and is the area under the time-force penetration curve (Table 4.2). The 70%, 65%, and 60% oil samples had a relatively
small range of work of shear from 19.1g•s (70% oil unroasted) to 93.3g•s (60% oil unroasted). The Smucker’s Natural Creamy peanut butter was in that range at 51.1g•s. Though the JIF and Skippy Natural Creamy samples were statistically similar to the aforementioned nut butters, they still measured higher spreadability readings at 359.2 g•s and 435.0g•s, respectively. Like the firmness data, the drop in oil content from 60% to 55% was also the point of significant increase in work of shear. The 55% oil unroasted samples averaged at 724.4g•s, but the unroasted ones of the same oil content were 2,616g•s. The 50% oil samples required the greatest force to spread at 7748g•s (unroasted) to 13,613g•s (roasted).

Adhesiveness was measured as the area underneath the x-axis in the lower portion of the time-force curve in gram-seconds (Table 4.2). Data for this portion of Table 4.2 showed that oil content again had an impact on the adhesiveness of the nut butters to the male cone as it lifted out of the female cone. Smucker’s Natural Creamy peanut butter, 70%, 65%, and 60% oil pecan butters had similar readings and had a looser texture than the other samples. The 70% oil unroasted sample was the least adhesive nut butter (9.8g•s). JIF and Skippy Natural Creamy commercial peanut butters were comparable to the 55% oil unroasted pecan butter. 50% oil samples and the 55% oil unroasted sample were the most adhesive, ranging from 343.2g•s for the 55% oil unroasted and 383.5g•s for the 50% oil unroasted.
Table 4.2: Texture analysis of pecan-based butters as affected by oil concentration compared to commercial butters means ± std error$^a$

<table>
<thead>
<tr>
<th>Sample</th>
<th>Firmness (g)</th>
<th>Spreadability (g•s)</th>
<th>Adhesiveness (g•s)</th>
</tr>
</thead>
<tbody>
<tr>
<td>70% Oil Unroasted</td>
<td>51.8 ± 1.2$^d$</td>
<td>19.1 ± 0.5$^d$</td>
<td>9.8 ± 0.3$^c$</td>
</tr>
<tr>
<td>65% Oil Roasted</td>
<td>100.4 ± 1.2$^d$</td>
<td>33.9 ± 0.6$^d$</td>
<td>14.4 ± 0.2$^c$</td>
</tr>
<tr>
<td>65% Oil Unroasted</td>
<td>97.8 ± 3.5$^d$</td>
<td>34.7 ± 1.1$^d$</td>
<td>16.2 ± 0.5$^c$</td>
</tr>
<tr>
<td>60% Oil Roasted</td>
<td>203.8 ± 6.8$^d$</td>
<td>68.7 ± 2.1$^d$</td>
<td>31.5 ± 1.2$^c$</td>
</tr>
<tr>
<td>60% Oil Unroasted</td>
<td>287.7 ± 11.1$^d$</td>
<td>93.3 ± 1.8$^d$</td>
<td>45.8 ± 1.3$^c$</td>
</tr>
<tr>
<td>55% Oil Roasted</td>
<td>1647.7 ± 56.9$^c$</td>
<td>724± 19.8$^{cd}$</td>
<td>343.2 ± 10.8$^a$</td>
</tr>
<tr>
<td>55% Oil Unroasted</td>
<td>1086.0 ± 59.7$^c$</td>
<td>2616.0 ± 187$^c$</td>
<td>163.5 ± 28.0$^b$</td>
</tr>
<tr>
<td>50% Oil Roasted</td>
<td>4880.0 ± 474.7$^a$</td>
<td>13613 ± 1371.0$^a$</td>
<td>383.5 ± 15.3$^a$</td>
</tr>
<tr>
<td>JIF Creamy</td>
<td>158.78 ± 4.4$^b$</td>
<td>359.2 ± 8.2$^d$</td>
<td>110.1 ± 5.7$^b$</td>
</tr>
<tr>
<td>Smuckers Natural Creamy</td>
<td>28.0 ± 2.2$^d$</td>
<td>51.1 ± 6.4$^d$</td>
<td>33.2 ± 4.3$^c$</td>
</tr>
<tr>
<td>Skippy Natural Creamy</td>
<td>209.8 ± 18.73$^d$</td>
<td>435.0 ± 47.8$^d$</td>
<td>154.4 ± 12.0$^b$</td>
</tr>
</tbody>
</table>

$^a$Means with different letters down columns are significantly different according to Tukey’s HSD Test (p≤0.01, α=0.05)
**Color Determination**

The L* c* h* colorspace was used to determine lightness (L*), saturation (c*) and hue angle (h*) of the products measured with a chroma meter. Nut butters on the market generally are orange-brown to brown, which was reflected in the results shown in Table 4.3 below. For L*, lightness did not appear to be directly affected by oil content and there was no trend in either direction within the pecan butter sample groups (40.8-44.5). However, the commercial peanut butters were significantly lighter than the others (55.6-59.0). All pecan samples exhibited similar c* values (12.8-13.8) and h* values (60.5-63.5), but again the commercial peanut butters were significantly different with c* values ranging from 29.0-30.7 (indicating a higher degree of saturation) and h* values at 71.3-72.7, which is more towards the orange-yellow portion of the L* c* h* color wheel than the red-purple area. These differences can be attributed to the fact that the commercial nut butters are made from peanuts instead of pecans, a visibly darker nut. It is noted that roasting did not significantly affect any color attributes for any of the pecan-based nut butters.
Table 4.3: L* c* h* values of pecan-based nut butters as affected by oil concentration compared to commercial butters

<table>
<thead>
<tr>
<th>Sample</th>
<th>L*</th>
<th>c*</th>
<th>h*</th>
</tr>
</thead>
<tbody>
<tr>
<td>70% Oil Unroasted</td>
<td>43.6 ± 0.3^c^d_</td>
<td>13.8 ± 0.2^b_</td>
<td>60.9 ± 0.6^b_</td>
</tr>
<tr>
<td>65% Oil Roasted</td>
<td>43.9 ± 0.5^c_</td>
<td>13.3 ± 0.2^b_</td>
<td>63.5 ± 0.9^b_</td>
</tr>
<tr>
<td>65% Oil Unroasted</td>
<td>44.1 ± 0.4^c_</td>
<td>13.5 ± 0.3^b_</td>
<td>62.6 ± 0.8^b_</td>
</tr>
<tr>
<td>60% Oil Roasted</td>
<td>44.0 ± 0.4^c_</td>
<td>13.3 ± 0.2^b_</td>
<td>63.3 ± 0.5^b_</td>
</tr>
<tr>
<td>60% Oil Unroasted</td>
<td>44.5 ± 0.4^c_</td>
<td>13.6 ± 0.2^b_</td>
<td>63.0 ± 0.6^b_</td>
</tr>
<tr>
<td>55% Oil Roasted</td>
<td>41.3 ± 0.6^d^e_</td>
<td>12.9 ± 0.2^b_</td>
<td>60.5 ± 0.5^b_</td>
</tr>
<tr>
<td>55% Oil Unroasted</td>
<td>43.1 ± 0.8^c^d^e_</td>
<td>13.5 ± 0.6^b_</td>
<td>62.5 ± 0.6^b_</td>
</tr>
<tr>
<td>50% Oil Roasted</td>
<td>42.7 ± 0.3^d^e_</td>
<td>13.1 ± 0.2^b_</td>
<td>62.6 ± 0.4^b_</td>
</tr>
<tr>
<td>50% Oil Unroasted</td>
<td>40.8 ± 0.3^e_</td>
<td>12.8 ± 0.4^b_</td>
<td>61.7 ± 0.4^b_</td>
</tr>
<tr>
<td>JIF Creamy</td>
<td>59.0 ± 0.1^a_</td>
<td>29.0 ± 0.4^a_</td>
<td>72.2 ± 0.8^a_</td>
</tr>
<tr>
<td>Smuckers Natural Creamy</td>
<td>55.6 ± 0.5^b_</td>
<td>30.1 ± 0.5^a_</td>
<td>71.2 ± 0.2^a_</td>
</tr>
<tr>
<td>Skippy Natural Creamy</td>
<td>57.7 ± 0.8^ab_</td>
<td>30.7 ± 0.7^a_</td>
<td>71.3 ± 0.5^a_</td>
</tr>
</tbody>
</table>

*Means with different letters down columns are significantly different according to Tukey’s HSD Test (p≤0.01, α=0.05)

Water Activity

The water activity is a reflection of the unbound water available to the 9 pecan-based nut butters and commercial peanut butters in Table 4.4 below. Oil content did not directly impact the water activity of neither the pecan butter samples nor the commercial peanut butters. The highest water activity sample was the 55% oil unroasted pecan butter sample, which was caused by the product taking on moisture during storage. All samples are well below the safety threshold for microbial spoilage.
Table 4.4: Water Activity ($A_w$) of pecan-based nut based butters as compared to commercial peanut butters means ± std error$^a$

<table>
<thead>
<tr>
<th>Sample</th>
<th>Water Activity ($A_w$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>70% Oil Unroasted</td>
<td>0.419 ± 0.001$^b$</td>
</tr>
<tr>
<td>65% Oil Roasted</td>
<td>0.418 ± 0.003$^b$</td>
</tr>
<tr>
<td>65% Oil Unroasted</td>
<td>0.417 ± 0.002$^{bc}$</td>
</tr>
<tr>
<td>60% Oil Roasted</td>
<td>0.387 ± 0.001$^c$</td>
</tr>
<tr>
<td>60% Oil Unroasted</td>
<td>0.411 ± 0.006$^{bc}$</td>
</tr>
<tr>
<td>55% Oil Roasted</td>
<td>0.435 ± 0.005$^b$</td>
</tr>
<tr>
<td>55% Oil Unroasted</td>
<td>0.472 ± 0.000$^a$</td>
</tr>
<tr>
<td>50% Oil Roasted</td>
<td>0.406 ± 0.006$^{bc}$</td>
</tr>
<tr>
<td>50% Oil Unroasted</td>
<td>0.467 ± 0.006$^a$</td>
</tr>
<tr>
<td>JIF Creamy</td>
<td>0.328 ± 0.006$^d$</td>
</tr>
<tr>
<td>Smucker’s Natural Creamy</td>
<td>0.279 ± 0.006$^e$</td>
</tr>
<tr>
<td>Skippy Natural Creamy</td>
<td>0.262 ± 0.004$^e$</td>
</tr>
</tbody>
</table>

$^a$Means with different letters down columns are significantly different according to Tukey’s HSD Test (p≤0.01, α=0.05)

Particle Size Analysis

The particle size analysis by percent volume is displayed in Figure 4.4 below. The majority of the pecan-based butters were approximately the same size, thus only two are included in the figure with the three commercial peanut butters. JIF and Skippy peanut butters measured the smallest particle size range, and Smucker’s Natural Peanut butter was the largest. The pecan-based butter particle size range was in between the commercial butters but closer in size to the Smucker’s Natural commercial butter. From these results it can be inferred that the grinding procedures and processing time affect particle size more so than oil content of a product.
Figure 4.1: Particle Size Analysis of Pecan-Based Butters and Commercial Peanut Butters

- 50% Oil Unroasted JIF
- SMUCKERS
- SKIPPY
- 60% Roasted

Percent Volume

Channel Size (μm)

0.1 1 10 100 1000 10000
Rheological Assessments

Apparent Viscosity

The data for 9 pecan-based butters at varying oil contents and roasts was compared to 3 commercial peanut butters for viscous rheological behavior using the following two mathematical models for plots of shear rate versus shear stress:

\[
\tau = \tau_o + \eta \dot{\gamma} \quad \text{Bingham Plastic}
\]

\[
\tau = \tau_o + K \dot{\gamma}^n \quad \text{Herschel-Bulkley}
\]

In the Bingham Plastic model, \( \tau \) is the resulting shear stress (Pa), \( \tau_o \) is the yield stress (Pa), \( \eta \) is the viscosity, and \( \dot{\gamma} \) is the shear rate (s\(^{-1}\)). In the Herschel-Bulkley model, \( \tau_o \) is the yield stress, \( K \) is the consistency index, and \( n \) is the power law index of the material [3]. A graph of the apparent viscosity as measured by the rheometer of all materials assessed is shown in Figure 4.2. Both of the aforementioned models were fit to all data and presented in Tables 4.5 (Herschel-Bulkley) and 4.6 (Newtonian). Samples not shown include the 55% unroasted and both 50% oil samples because they experienced major slippage at low shear rates and produced unreadable data.

According to the \( r^2 \) values presented in Tables 4.5 and 4.6, the only two samples that fit the linear Bingham Plastic model better than the non-linear Herschel-Bulkley were the JIF Creamy and unroasted 70% oil samples. The 65% unroasted oil fit both models equally with an \( r^2 \) of 0.999. Thus, it can be concluded that the majority of the pecan-based nut butter samples and commercial peanut butters used for comparison exhibit shear thinning behavior with a yield stress.
Table. 4.5: Herschel-Bulkley model fit of pecan-based butters as compared to commercial peanut butters means ± std error

<table>
<thead>
<tr>
<th>Sample</th>
<th>K (mPa-s)\textsuperscript{1-n}</th>
<th>n</th>
<th>(\eta) (Pa)</th>
<th>(r^2)</th>
</tr>
</thead>
<tbody>
<tr>
<td>70% Oil Unroasted</td>
<td>0.417 ± 0.047\textsuperscript{b}</td>
<td>1.01 ± 0.022\textsuperscript{a}</td>
<td>0.994 ± 0.065\textsuperscript{a}</td>
<td>0.981</td>
</tr>
<tr>
<td>65% Oil Roasted</td>
<td>0.724 ± 0.039\textsuperscript{b}</td>
<td>0.967 ± 0.006\textsuperscript{a}</td>
<td>1.16 ± 0.168\textsuperscript{a}</td>
<td>0.999</td>
</tr>
<tr>
<td>65% Oil Unroasted</td>
<td>1.31 ± 0.074\textsuperscript{b}</td>
<td>0.906 ± 0.010\textsuperscript{a}</td>
<td>0.685 ± 0.415\textsuperscript{a}</td>
<td>0.999</td>
</tr>
<tr>
<td>60% Oil Roasted</td>
<td>4.02 ± 0.039\textsuperscript{b}</td>
<td>0.894 ± 0.006\textsuperscript{a}</td>
<td>6.25 ± 0.598\textsuperscript{a}</td>
<td>0.999</td>
</tr>
<tr>
<td>60% Oil Unroasted</td>
<td>4.37 ± 0.47\textsuperscript{b}</td>
<td>0.846 ± 0.022\textsuperscript{a}</td>
<td>4.33 ± 1.05\textsuperscript{a}</td>
<td>0.999</td>
</tr>
<tr>
<td>55% Oil Roasted</td>
<td>75.8 ± 7.93\textsuperscript{a,b}</td>
<td>0.619 ± 0.021\textsuperscript{a}</td>
<td>2.42 ± 10.6\textsuperscript{a}</td>
<td>0.999</td>
</tr>
<tr>
<td>JIF Creamy</td>
<td>20.1 ± 14.4\textsuperscript{a,b}</td>
<td>0.655 ± 0.220\textsuperscript{a}</td>
<td>25.2 ± 18.4\textsuperscript{a}</td>
<td>0.934</td>
</tr>
<tr>
<td>Smuckers Natural Creamy</td>
<td>36.7 ± 5.82\textsuperscript{a,b}</td>
<td>0.655 ± 0.027\textsuperscript{a}</td>
<td>26.1 ± 22.5\textsuperscript{a}</td>
<td>0.966</td>
</tr>
<tr>
<td>Skippy Natural Creamy</td>
<td>718 ± 427\textsuperscript{a}</td>
<td>0.172 ± 0.067\textsuperscript{b}</td>
<td>32.34 ± 26.5\textsuperscript{a}</td>
<td>0.998</td>
</tr>
</tbody>
</table>

\(a\) Means with different letters down columns are significantly different according to Tukey’s HSD Test (p≤0.01, \(\alpha=0.05\))

Table. 4.6: Newtonian model fit of pecan-based butters as compared to commercial peanut butters means ± std error

<table>
<thead>
<tr>
<th>Sample</th>
<th>(\eta) (mPa-s)</th>
<th>(\eta) (Pa)</th>
<th>(r^2)</th>
</tr>
</thead>
<tbody>
<tr>
<td>70% Oil Unroasted</td>
<td>403 ± 1.8\textsuperscript{e}</td>
<td>1.1 ± 0.05\textsuperscript{d}</td>
<td>0.999\textsuperscript{a}</td>
</tr>
<tr>
<td>65% Oil Roasted</td>
<td>615 ± 3.14\textsuperscript{c}</td>
<td>2.1 ± 0.2\textsuperscript{d}</td>
<td>0.979\textsuperscript{a}</td>
</tr>
<tr>
<td>65% Oil Unroasted</td>
<td>833 ± 9.01\textsuperscript{c}</td>
<td>3.9 ± 0.02\textsuperscript{d}</td>
<td>0.999\textsuperscript{a}</td>
</tr>
<tr>
<td>60% Oil Roasted</td>
<td>2434 ± 13.2\textsuperscript{d}</td>
<td>18.2 ± 1.6\textsuperscript{d}</td>
<td>0.998\textsuperscript{a}</td>
</tr>
<tr>
<td>60% Oil Unroasted</td>
<td>2035 ± 3.77\textsuperscript{d}</td>
<td>17.7 ± 1.2\textsuperscript{d}</td>
<td>0.997\textsuperscript{a}</td>
</tr>
<tr>
<td>55% Oil Roasted</td>
<td>19490 ± 267\textsuperscript{a}</td>
<td>106 ± 2.7\textsuperscript{b}</td>
<td>0.983\textsuperscript{ab}</td>
</tr>
</tbody>
</table>

\(a\) Means with different letters down columns are significantly different according to Tukey’s HSD Test (p≤0.01, \(\alpha=0.05\))
Figure 4.2: Apparent viscosity of pecan-based nut butters as compared to commercial peanut butters.

- 70% Oil
- 65% Roast
- 65% No roast
- 60% No roast
- 60% Roast
- 55% Roast
- 55% No roast
- 50% Roast
- 50% No roast
- JIF
- SMUCKERS
- SKIPPY
Stress Sweep

Figure 4.3 below shows the data plotted of stress sweep viscoelastic curves for unroasted versions of 70%, 65%, 60%, 55%, and 50% oil unroasted versions of pecan-based butter when subjected to increasing stresses at 1 Hz using a 40 mm cone-in-plate geometry with a ~2° angle at 25°C. Each plotted dataset demonstrates a linear viscoelastic region (LVR) and the approximate stress at which the pecan butter begins to slip. As the oil level increased, the storage modulus (G’) and the length of the linear viscoelastic region (LVR) decreased. The firmest sample (50% oil) had a LVR from 10-1,000 Pa at approximately 250,000 Pa, and both the 65% and 70% oil pecan butters showed an LVR from 0.001 to 0.01 Pa.

Another view of these results is shown figure 4.4, a plot of oil contents versus the LVR for the storage modulus (G’) and the loss modulus (G”) for the aforementioned pecan butter samples [4]. Here, the 50% is again the firmest and the 65%-70% samples are approximately of the same firmness. It is also shown that G’ is above G” for most except for the 65%-70%, which indicates that the samples are more elastic than viscous. This gap is the widest at 50% and decreases with increasing oil content, thus showing that the samples become more viscous with the addition of more pecan oil in the formulation.
Figure 4.3: Oscillation amplitude stress sweeps for unroasted pecan-based nut butters.

- 50% Oil
- 55% Oil
- 60% Oil
- 65% Oil
- 70% Oil

Storage Modulus $G'$ (Pa)

Oscillatory Stress (Pa)
Figure 4.4: Linear viscoelastic regions of pecan-based nut butters as a function of oil content
References

1. Geisler, M. Pecans http://www.agmrc.org/commodities__products/nuts/pecans/#.


CHAPTER 5

CONCLUSION AND SUMMARY

This purpose of this research was to create a novel pecan-based nut butter. Due to the high oil content of pecans, the process was designed to create this product comprised of partially defatted pecan flour, sugar, salt, and varying degrees of pecan oil and roast levels to determine the influence of oil content and roasting conditions on sensory, physical, and rheological attributes.

Sensory testing proved that the full-fat 70% oil pecan-based butters were the least acceptable among consumers in all attributes, and panelists rated it as the product that they would be the least willing to buy as well. The most acceptable butters were the 60% oil roasted and unroasted samples, but they still only had an average rating between “like slightly” and “like moderately” and consumers indicated that they would at best purchase the sample occasionally. Formulations of pecan-based butter should thus be improved prior to a commercial launch.

Physical testing showed that the pecan-based samples were significantly darker than the commercial peanut butter samples, but had a particle size in between that of JIF Creamy/Skippy Natural and Smucker’s Natural. Water activity was not affected by the oil content. Texture analysis showed that firmness, adhesiveness, and spreadability (work of shear) was not statistically different for 70%-60% oil level samples and the commercial peanut butters. The 55% and 50% oil ones were significantly firmer, more adhesive and more spreadable.

Fitting rheological models to the apparent viscosity curves showed that the nut butters demonstrated predominantly shear thinning behavior, thus indicating that the viscosity
decreased with an increase in shear stress. The Bingham Plastic model best fit for the 70% oil and JIF creamy samples, whereas the others better fit the Herschel-Bulkley model best because they required a yield stress to begin flow. Other low-oil samples were not surveyed because they were too firm to be measured correctly by the vane geometry.

All unroasted pecan-based butter samples were measured using stress sweeps at varying stresses to determine the viscoelasticity for each oil level. The 70% and 65% oil samples were effectively the same regarding G’ and G” values. Decreasing the oil content increased both moduli for all other samples and the stress ranges for their linear viscoelastic regions. The elastic behavior (G’) was higher than viscous behavior (G”) as well.

Overall results showed that oil content of the various pecan-based butter samples has a significant impact in all areas, but roasting the pecan flour prior to processing did not. The most substantial changes occurred between the 55% and 60% oil content. Three commercial creamy peanut butters that were compared alongside the pecan-based products for physical and rheological testing tested in between of the 55% and 60% pecan-based butters. Thus, it can be concluded that the optimum oil content for spreadable and acceptable pecan butter lies within that range.

Future studies regarding this product should focus on a narrow range of oil levels between 55% and 60% for formulations to pinpoint a specific percentage that is the most acceptable physically and with consumers. A stabilizer such as red palm oil may also be introduced to formulations to help accomplish a firm, yet spreadable texture with a smooth consistency so that the product can be optimized for a commercial launch. Other important aspects of developing a product should also be considered in future studies, such as product safety, shelf life, and oxidation.
APPENDIX A – Panelist Demographic Questionnaire

1. Your gender: ______ Male ______ Female

2. Your age:
   ______ 18-22
   ______ 22-26
   ______ 26-30
   ______ 31-45
   ______ 46-50
   ______ 50 and older

3. How often do you eat pecans or pecan-products? (Check the best response)
   ______ Several times a week
   ______ Several times a month
   ______ Several times a year
   ______ Never
APPENDIX B – Panelist Hedonic Scorecard

Product: Pecan-based product

Please indicate how much you like or dislike each sample on the parameters listed by columns based on your feeling toward the sample. Drink some water and eat a baby carrot after each sample to cleanse your palate.

<table>
<thead>
<tr>
<th>SAMPLE</th>
<th>Appearance</th>
<th>Texture</th>
<th>Flavor</th>
<th>Spreadability</th>
<th>Overall</th>
</tr>
</thead>
<tbody>
<tr>
<td>Like extremely</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Like very much</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Like moderately</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Like slightly</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Neither like or dislike</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Dislike slightly</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Dislike moderately</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Dislike very much</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Dislike extremely</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Please check the appropriate comment for pecan-based sample ________.

I would buy this very often
I would frequently buy this
I like this and would but it now and then
I don't like this but would buy it occasionally
I would hardly ever buy this
I would never buy this