

PARTIAL OIL EXTRACTION OF OKLAHOMA PECANS

By

MARILYN BAUGH WATERS

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Thesis Approved:

Gene B Knight
Thesis Adviser

Esther Nesterfeld

P. Larry Claypool

Norman N. Durham
Dean of the Graduate College

1251841

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CHAPTER I

INTRODUCTION

Pecans (Carya Illinoensis) are the number one horticulture crop in Oklahoma but optimum quality is difficult to maintain. Because of poor keeping quality, pecans have become a seasonal food product.

Geographically, the native habitat of the pecan tree lies in the southern portion of the United States. These trees grow along the Mississippi Valley and its tributaries, along the valleys and overflow land of smaller rivers and creeks. Oklahoma and Texas possess the greatest number of native pecan trees of all of the pecan-producing states (Atwood, 1949; Peterson and Johnson, 1978). Oklahoma also possesses a small portion of improved variety pecans, but growers consider the native crop of greater importance because of the better flavor and texture.

Climate is a paramount factor affecting the success and failure of pecan production. Pecan trees are deciduous and grow within a wide range of temperature climates; however, the optimum conditions for pecan growth are in regions with a long growing season, a hot summer, and cool to cold winters. These climatic factors are prevalent within a belted strip stretching diagonally from the northeast corner of the state of Oklahoma, to the state of Texas and along the Red River Valley (Atwood, 1949). This part of Oklahoma also receives about 40 inches of seasonably favorable annual rainfall which pecans require.

A favorable seasonal rainfall distribution provides greater amounts of moisture during the filling of the nut, which is of prime importance, with a smaller amount of moisture needed during the remainder of the year. During dry periods, available soil moisture must be conserved as much as possible. Lower rainfall and conditions that allow loss of soil moisture result in sparse pecan growth in the western section of the state (Atwood, 1949).

Soil adaptation is next to climate in importance for productive pecan growth. The ideal soil for pecans is a combination of fertile loam as topsoil to provide nourishment for feeder roots and a good clay mixture as subsoil for proper anchorage for extensive root systems. The soil must be fertile, have good drainage, and be of sufficient depth for prosperous tree growth. The top three to four feet of soil is of greatest significance, for it supplies the food necessary for growth (Atwood, 1949). Availability of these ideal conditions leads to pecans being an important horticulture crop in the state of Oklahoma.

In the last 50 years there has been a phenomenal increase in the number of pecan trees and the quality of pecans produced. Development of improved machinery, fertilizers, pest control methods, and grafting techniques have been instrumental in increasing the quantity and quality of nuts reaching the market. The pecans fall from September to December, when the pecan market is most active. Also, the size of the pecan crops tends to alternate from year to year (Sparks, 1975; Stein, 1980). Thus, a large amount of nuts come to market during a short period of time, and unless properly stored, will lose their fresh flavor, color, and aroma.

Pecan nuts, which contain high levels of unsaturated oil, show high levels of flavor instability, since the oil is susceptible to rancidity. Pecans held at 75-80°F (24-26.5°C) will retain their fresh flavor and texture for only about one month. Rancidity is accelerated as the storage temperature increases and humidity remains low. At the present time, the most effective way of retarding rancidity is by refrigeration (Woodroof, 1967; Senter, Horvat, and Forbus, 1980). For instance, several million kg of shelled and in-shell pecans are usually cold stored to meet market demands throughout the spring, summer, and early fall. The quantity of cold-stored pecans in May, 1979, was in excess of 45 million kg (Senter, Horvat, and Forbus, 1980). Cold storage is very costly and has aided in pricing pecans out of the common market. In April of 1980, the National Pecan Marketing Council (NPMC) conducted a survey and found that the pecan was the preferred nut, although the study also showed that it was often substituted because of price (Anonymous, 1980).

Godkin, Beattie, and Cathcart (1951) reported that rancidity is the major form of deterioration in pecans and is of major interest and concern to industry. Researchers have attempted to improve the post-harvest quality of pecans and to make recommendations as to the processing, handling, and storage in order to maintain optimum quality and retard rancidity (Heaton and Woodroof, 1970; Heaton, Worthington, and Shewfelt, 1975; Forbus and Senter, 1976; Forbus, Tyson, and Ayres, 1979; Senter, Horvat, and Forbus, 1980). Although vast improvements have been made in kernel quality, it is still estimated that more than 50% of the pecan kernels in the market are of substandard quality (Williams, LaPlante, and Heaton, 1973).

Pecan oil is the source of rancidity in pecans, and if the pecan oil could be removed or the amount reduced, perhaps pecans would be more stable. Pecans are also high in calories, largely due to the fat content. Watt and Merrill (1963) reported a caloric value of 687 calories/100 g for shelled pecan kernels, and an average of 71% fat. It would be advantageous if part or all of the fat could be removed from pecans in such a manner that the pecan flavor would remain, with no unpleasant flavors or odors added.

Purposes and Objectives

The purpose of this study was to determine if an acceptable fat-reduced pecan could be developed through partial extraction of the pecan oil.

The objectives of the study were as follows:

1. To determine the amount of fat available in the pecans used in this study
2. To develop a fat extraction process for pecans which will produce no off taste or off odor
3. To determine the effect of fat extraction on the quality of pecans through sensory evaluation
4. To determine the amount of fat extracted from three different-sized, chopped pecans
5. To make recommendations for further studies in this area

Hypotheses

The following hypotheses were postulated for this research:

H₁: For each of the following sensory qualities, there is no

significant difference between mean response levels of interaction between particle size and treatment for treated and untreated pecan pieces of fine, medium, and coarse particle size. The sensory evaluation will include the following attributes: nutty aroma, off-odors, texture, sweetness, full nut flavor, off-flavors, unpleasant after-taste, and overall acceptability.

H₂: For each of the preceding sensory qualities, there is no significant difference between mean response levels of extracted pecan pieces and untreated pecan pieces.

H₃: For each of the preceding sensory qualities there is no significant difference between mean response levels for pecan pieces of fine, medium, and coarse particle sizes.

Assumptions and Limitations

The following assumptions were made for this study:

1. A trained taste panel will evaluate the bread products as instructed
2. The experiments will be conducted under controlled environmental conditions

Limitations for this study were identified as follows:

1. Only six products will be investigated:
 - a. Finely chopped, untreated pecan pieces
 - b. Medium chopped, untreated pecan pieces
 - c. Coarsely chopped, untreated pecan pieces
 - d. Finely chopped, treated pecan pieces
 - e. Medium chopped, treated pecan pieces
 - f. Coarsely chopped, treated pecan pieces

2. Only 1984 native Oklahoma-grown pecans obtained from the Oklahoma Pecan Commission will be used

Definition of Terms

The following are the definition of terms used in this study:

Distillation. A process of vaporizing and condensing a substance to purify a substance (Saunders, 1981).

Cotyledons. Two halves of the pecan kernel (Woodroof and Woodroof, 1927).

Hexane. (n-Hexane) $\text{CH}_3(\text{CH}_2)_4\text{CH}_3$; mol. st. 86.17; C_6H_{14} (C, 83.62%); H, 16.38%). Chief constituent of petroleum ether or ligroin. Colorless, very volatile liquid; faint, peculiar odor.

b.p. 69°
Solidify; -95° to -100°
Insoluble in water
Miscible with alcohol, chloroform, ether

Toxicity: May be irritating to respiratory tract and in high concentration, narcotic. Use: Determining refractive index of materials; filling for thermometers with blue or red dye (Stecher, 1968).

Saturated. Compounds which do not contain double bonds (Jones, Netterville, Johnson, and Wood, 1980).

Septum. Corky middle separating the two cotyledons (Woodroof and Woodroof, 1927).

Triglycerides. A compound consisting of three molecules of fatty acids esterified to glycerol (Dorland's Illustrated Medical Dictionary, 1981).

Unsaturated. Compounds which contain double or triple bonds (Jones et al., 1980).

Format of Thesis

The experiment described in Chapter III was organized and prepared as an individual manuscript for publication in the most acceptable journal. The experiment was written according to the Style Guide for Research Papers, Institute of Food Technologists and the Journal of Food Science.

CHAPTER II

REVIEW OF LITERATURE

General Studies of Pecans

The pecan can be traced back to the Cretaceous period, according to paleontological evidence, but only in the last four centuries has this plant become a cultivated crop (Stuckey and Kyle, 1925). The pecan tree is classified as a hickory (Carya) and is a member of the walnut family (Juglandaceae) (Peterson and Johnson, 1978). It is native to the southeast portion of North America, and the trees are common along waterways (Woodroof, 1967).

George Washington planted pecans in 1775 which are still standing at Mount Vernon, and Thomas Jefferson planted trees in 1789. Antoine, a slave gardener on a plantation in Louisiana, was the first to successfully graft these trees; by the end of the Civil War, there were 126 bearing varieties of pecans (Woodroof, 1967).

Composition

Tree nuts have long been an important part of man's diet (Woodroof, 1978), and like other nuts, pecans are a nutritious food source (Considine and Considine, 1982). As summarized in Table I, pecans are high in unsaturated fat, the type of fat that many health authorities recommend be included in the daily diet (Watt and Merrill, 1963). The

of oil in pecans varies more than any other component, from less than 60% to slightly more than 70% (Brinson, 1974). Pecans are approximately 9% protein and 14.6% carbohydrate. They contain substantial amounts of the minerals phosphorus and potassium; moderate levels of magnesium; and a limited supply of iron, sodium, and calcium (Woodroof, 1967).

TABLE I
COMPOSITION OF PECAN MEATS

Proximate Analysis - g/100g	
Fat	71.2
Saturated - 3.5%	
Polyunsaturated - 93-95%	
Carbohydrate	14.6
Protein	9.2
Moisture	3.4
Fiber	2.3
Ash	1.6
Mineral Content - mg/100g	
Potassium	603.0
Phosphorus	289.0
Calcium	73.0
Iron	trace
Sodium	trace
Vitamin Content in 100g	
Vitamin A	130 IU
Ascorbic Acid	2.00 mg
Thiamin (B)	0.86 mg
Riboflavin (B)	0.13 mg
Niacin	0.90 mg
Vitamin E	15-50 mg
Calories in 100g (approximately one cup)	689

Source: B. K. Watt and A. L. Merrill, Composition of Foods (1963).

Growing Pecans

The nineteenth century witnessed the transformation of the wild pecan tree into an orchard product of great commercial value (Woodroof, 1967). Oklahoma, a major pecan-producing state, possesses ideal physical factors necessary for large yields. It has 34 to 42 inches of annual rainfall, 200 to 230 frost-free days, and highly fertile flood plain soil (Atwood, 1949). These conditions are most common in the central and southeastern sections of Oklahoma (Atwood, 1949).

Quality Determinants

Certain characteristics have been established by the United States Department of Agriculture (USDA) as being determinants of quality in fresh pecans. These characteristics are: light color, crispness, and the absence of rancidity and stale taste (Forbus, Senter, Lyons, and Dupuy (1979). Pecan kernels maintain their optimum flavor qualities for only a short time after harvest and then begin to gradually deteriorate unless properly stored.

Since pecan kernels are semiperishable, they should be refrigerated to maintain optimum quality (Senter, Horvat, and Forbus, 1980). As the cost of energy increases, proper pecan storage is also more costly. As a result, pecans sold in the retail market may be sold in substandard conditions. Williams, LaPlante, and Heaton (1973) estimated that 50% of the pecans sold in retail outlets are sold below the USDA standards.

Marketing Concerns

Entire crops are often destroyed by late frost, but even in

frost-free springs there is a tendency for a heavy crop to be followed the next year by a light yield. This alternate year-bearing pattern in pecan production has created marketing as well as managerial problems for pecan growers (Blake and Clevenger, 1980). Horticulturally, no single factor will alleviate alternate bearing (Sparks, 1975). However, Blake and Clevenger recommended that growers and processors try to predict these changes in production so that they can plan their financial and marketing strategies accordingly.

Pecan production is slowly increasing (Peterson and Johnson, 1978), but there is evidence that per capita consumption is actually decreasing (Stebbins, 1980). Thus, we see an increase in supply with no growth in demand; this leads to an imbalance in the market.

Stebbins (1980) indicated that one reason for low consumption of pecans is that poor quality pecans are allowed into the market. A variety of reasons have been given for this. If marketing does not keep up with pecan production, older pecans will remain unsold and in stock, even after a new crop has been harvested (Williams, 1977; Blake and Clevenger, 1980). Poorly dried pecans and those stored in too moist conditions will develop bad flavors and may even develop mycotoxins as a result of mold growth (Woodroof and Heaton, 1958; Lillard, Hanlin, and Lillard, 1970; Doupnik and Bell, 1971; Heaton, 1972; Taylor and Worley, 1972; Chhinnan, 1980). Storage conditions where the temperatures are too high or where there is too much exposure to light lead to poor quality, due largely to oxidation of the unsaturated fats (Heaton and Woodroof, 1955; Brinson, 1974; Heaton, 1974; Wagner, 1980; Peterson and Johnson, 1978).

Pecan Oil and Rancidity

Little research was done on pecans before the twentieth century. In 1910, Deiler and Frads reported that there was 70.4% oil in pecan kernels. In 1926, George and Gertler (cited in Pyriadi, 1967) analyzed the oil but found only oleic and linoleic fatty acids.

Since the development of gas chromatography and ultraviolet spectrophometry, more is known about the composition of pecans and pecan oil. More fatty acids were identified in pecan oil in the 1960's (French, 1962; Senter and Horvat, 1976; Woodroof, 1967), and the fatty acid content was further updated in the 1970's, as seen in Tables II and III.

TABLE II
FATTY ACID COMPOSITION OF PECAN OIL

Total Oil (%)	70.3
Fatty Acid (%)	
16:0	5.7
16:1	0.1
17:0	trace
17:1	trace
18:0	2.2
18:1	66.9
18:2	22.1
18:3	1.1
20:0	0.2
20:1	0.4
Trace and Unidentified	1.3
Unsaturated: Saturated Ratio	11:2

Source: L. R. Beuchat and R. E. Worthington, "Fatty Acid Composition of Tree Nut Oil," J. Food Tech. (1978).

TABLE III
FATTY ACIDS IN PECAN OIL

10:0	Decanoic	17:0	Heptadecanoic
12:0	Dodecanoic	17:1	Heptadecenoic
12:1	Dodecenoic	17:2	Heptadecadienoic
14:0	Tetradecanoic	18:0	Octadecanoic
14:1	Tetradecenoic	18:1	Octadecenoic
14:2	Tetradecadienoic	18:2	Octadecadienoic
15:0	Pentadecanoic	18:3	Octadecatrienoic
15:1	Pentadecenoic	20:0	Eicosanoic
15:2	Pentadecadienoic	20:1	Eicosenoic
16:0	Hexadecanoic	20:2	Eicosodienoic
16:1	Hexadecenoic	21:0	Heneicosanoic
16:2	Hexadecadienoic		

Source: S. D. Senter and R. J. Horvat, "Minor Fatty Acids From Pecan Kernel Lipids," J. Food Sci. (1978).

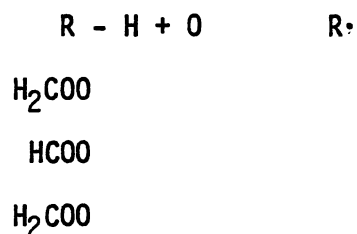
The amount of pecan oil varies more than any other component of the nut. The amount is influenced by physiological conditions as well as the stage of growth and development (Brinson, 1974). Pecan oils are 92 to 97% triglycerides, composed predominately of 18 carbon, unsaturated, and polyunsaturated fatty acids (Senter and Horvat, 1978). The saturated fatty acids, predominately palmitic and steric, range from 3 to 8%. The high amount of unsaturated fatty acids tends to make pecans a good health food (Stein, 1980), but also makes them more susceptible to oxidation and rancidity (Beuchat and Worthington, 1978).

Unsaturated fats undergo atmospheric oxidation (autoxidation), resulting in off-flavors, off-odors, and, in extreme cases, toxic

byproducts (Stuckey, 1981). Autoxidation deals entirely with the double bonds, or the unsaturated portions, in the fatty acid chain (Stuckey, 1981) and is a chain reaction with the three basic steps in the process: (1) initiation, (2) propagation, and (3) termination (Perkins and Visek, 1983).

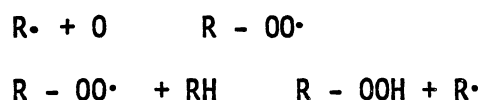
Initiation, the first step, is the formation of a free radical ($R\cdot$) formed by light, heat, enzymes (that is, lipoxidase), or other biological catalysts. This radical "propagates" the reaction by reacting with oxygen to produce a peroxide radical ($R-OO\cdot$). The peroxide radical further propagates with the addition of a hydrogen atom from an unsaturated fatty acid, thus yielding a hydroperoxide and another free radical ($R\cdot$), as shown:

1. Initiation:



The propagation steps forming hydroperoxides are repeated again and again, producing a chain reaction, playing a central role in autoxidation. This cycle is repeated until a "termination" step is reached (Peterson and Johnson, 1978; Perkins and Visek, 1983), forming a nonradical end product ($ROOH$).

2. Propagation:



Volatile carbonyl compounds are produced as a product of lipid oxidation. Aldehydes, ketones, and hydrocarbons have been indicated

as the stimuli of off odors and flavors associated with autoxidation (Peterson and Johnson, 1978). As hydroperoxides are being formed during autoxidation, antioxidants can act as a hydrogen donor. The antioxidant is acting as a "terminator," thereby breaking the propagation cycle. This reaction is usually depicted as:

3. Termination:



where R is the fat containing a free radical and AH is the antioxidant (Rudolph, 1971). The antioxidant radical must be inactive and cannot initiate further reaction (Stuckey, 1981). To be permissible in foods, the antioxidants used must be listed as "Generally Recognized as Safe" by the Food and Drug Administration (FDA), and the amounts used are limited to 0.02% (200 ppm) (Stuckey, 1981).

Effect of Maturation

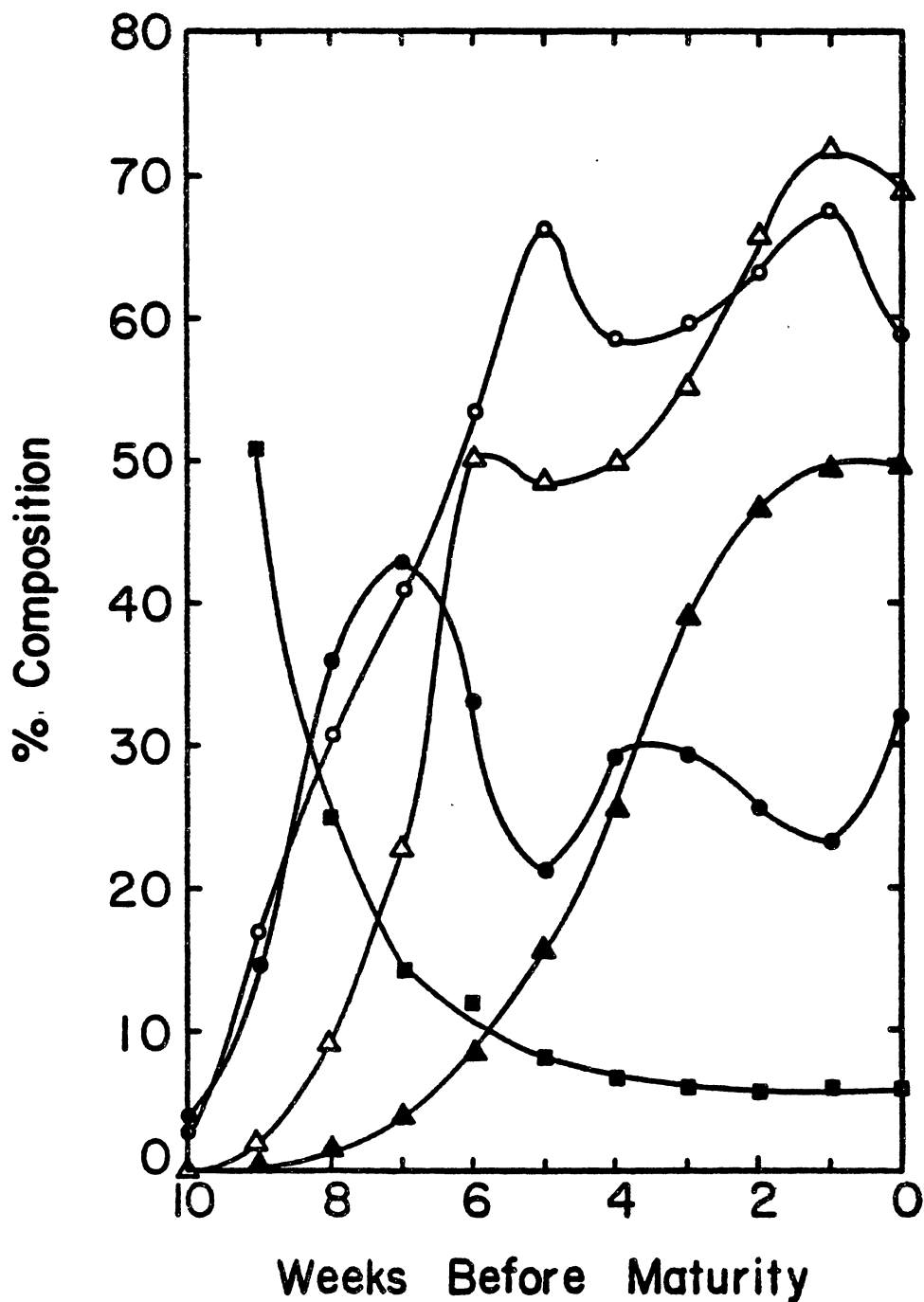
Early studies of the morphological and anatomical properties of pecans defined the developmental changes which occur in pecan nuts from flowering (Woodroof and Woodroof, 1926, 1927). As the pecan develops from the early prefilling stage to maturity, there are changes in oil, protein, and carbohydrate contents (Thor and Smith, 1935, 1939). According to Finch (1933, 1934, 1935), the problems of pecan filling and maturity are related to the drying of the shuck and to the tree's ability to accumulate carbohydrate.

The development of nuts have been divided into two growth periods (Thor and Smith, 1935). The first growth period, from blossoming until late August or early September, consists basically of the structural formation of the shuck and the shell. The second period, filling

the shell, is of major importance (Thor and Smith, 1935). During this filling period, the cotyledon tissue is first present as a gel layer within the seed coat. This gel contains a high concentration of sugar and little fat (Finch, 1933, 1934, 1935). Filling occurs rapidly with the formation of oil, protein, minerals, and acid-hydrolyzable polysaccharides. Total sugar content is not reached until the formation of the oil content has stopped (Thor and Smith, 1935, 1939).

Rudolph's (1971) research, conducted over a two-year period, illustrated the rapid build-up of pecan oil (Figure 1). His sampling period began 10 weeks before maturity, just before gel formation. At that point, the Stuart variety contained less than 1% oil. At nine weeks before maturity, there was 1 to 2% oil, and this oil was 50% palmitic acid and 18% each oleic and linoleic acids. In the period eight-to-six weeks before maturity, the oil concentration rose to 50% and leveled off. During this period, there was a sharp decline in palmitic acid, to about 13%, and an increase in oleic and linoleic to approximately 40% each of the total fatty acid. In the following weeks, Rudolph observed a steady increase in the total oil content, with an increase of oleic acid to 67% and a decrease of linoleic acid to 20%.

Hammer and Hunter (1946) studied the mineral content in relation to physical changes during the pecan kernel filling. They found that the prefilling period, from about August 25 to September 15, is a critical period in the development of the kernel. During this period, 63% of the oil, 43% of the ash, and 71% of the protein are formed in the kernel.



Variation in Palmitate, ■, Oleate, ○, and Linoleate, ●, (as % of Oil) and Variation of Oil Content (% Dry Wt., ▲, % Wet Wt., ▲)

Source: C. J. Rudolph, "Factors Responsible for Flavor and Off-Flavor Development in Pecans" (1971), 183.

Figure 1. Variation in Pecan Oils During the Maturation of Stuart Variety Pecans, 1969 Crop

Lewis and Hunter (1944) indicated that during the filling period, quantities of nitrogen, potassium, magnesium, and phosphorus accumulated in the nut, while significant quantities of these same elements were lost from the supporting shoots. Hammer and Hunter (1946) further noted that all substances entering the nut had to pass through the shuck, and that approximately 70% of the minerals, phosphorus, potassium, calcium, and magnesium remained in the shuck. They also concluded that the rapid rate of potassium accumulation during the early filling stage indicated its importance in the translocation or transformation of stored minerals in the kernels.

Rudolph (1971) concluded that fats are formed as saturated fatty acids, which, as the nut matures, are desaturated; but this mono- and di-unsaturation is influenced by environmental factors, with no consistent year-to-year pattern.

Through the use of thin, layer chromatography, Pyriadi (1967) detected the presence of tocopherol and carotenoids in pecan nut oil and concluded that these constituents played an important role in resistance to oxidative deterioration of the unsaturated oil (Lambertsen, Myklestad, and Brekkan, 1962; Pyriadi, 1967). Tocopherol is found throughout the plant kingdom, primarily in fats and oils (Eisner and David, 1966), the richest source being wheat germ oil. Pecan oil is an abundant source of tocopherols, with 170 μg of γ tocopherol per gram of nut and 15 μg of α tocopherol per gram of nut (Lambertsen, Mylestad, and Braekkan, 1962).

Rudolph (1971) conducted further studies of tocopherol content during maturation of the pecan. He observed that the tocopherol content was elevated at six weeks before maturity to approximately 600 μg

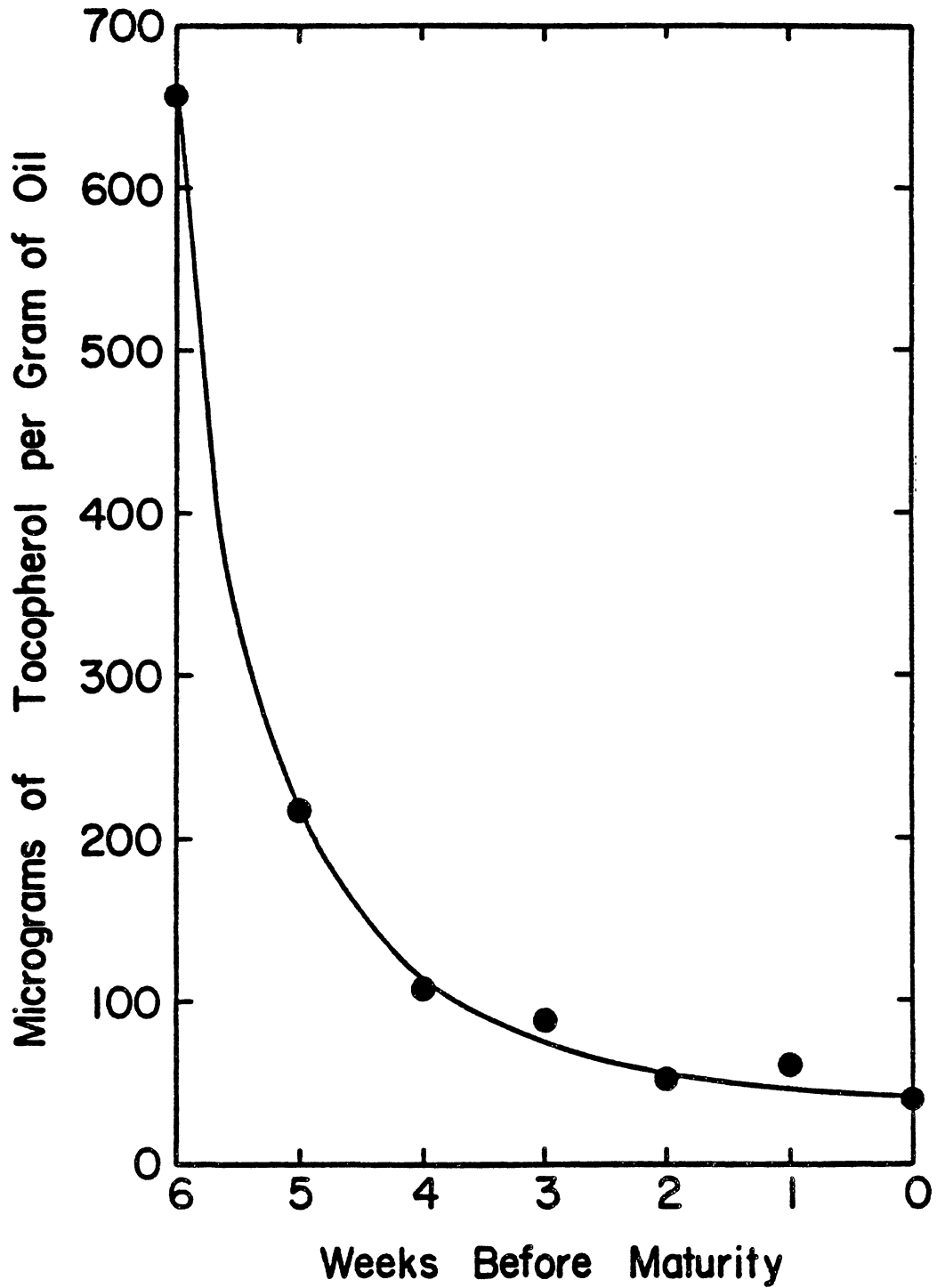
per gram of pecan oil, followed by a rapid and continual decrease to approximately 100 µg per gram of oil at maturity (Figure 2). Contrary to the conclusions drawn by Pyriadi (1967) and others, it is the opinion of some researchers that tocopherol is not of prime importance in determining the oxidative stability of pecans (Odell, 1970; Rudolph, 1971).

Maintaining Pecan Quality

Before 1931, pecans were a seasonal food product. Since then, much has been learned about how to properly harvest pecans and protect them from mold, souring, insects, staleness, discoloration, and rancidity, so that now if proper procedures are followed, pecans can be a year round food.

Pecan nuts are harvested over an eight-to-ten week period, from late September to mid December (Heaton and Beuchat, 1980), and as many as 50% may be left ungathered (Atwood, 1949). Mechanical harvesting, which includes shaking the mature nuts from the trees, allows for complete harvest at an earlier date and minimizes the effects of adverse weather conditions on the fallen pecans.

After harvest, pecans left under ambient conditions normally become rancid in four to six weeks (Holaday, Pearson, and Slay, 1979); thus, the most common method of maintaining quality of both in-shell and shelled pecans is refrigeration (Woodroof, 1967; Heaton, 1974). Before 1961, freezing was thought to be detrimental and impractical for pecans, but because of a large crop in that year, processors were encouraged to freeze nuts for storage (Woodroof, 1967). Experiments have shown that refrigeration storage of $<0^{\circ}$ C and 65 to 68% relative



Source: C. J. Rudolph, "Factors Responsible for Flavor and Off-Flavor Development in Pecans" (1971), 189.

Figure 2. Variation in Tocopherol Content During the Maturation of Pecans, Stuart Variety, 1968 Crop

humidity arrests insects, prevents molding, retards development of staleness and rancidity, and preserves natural color, flavor, and texture of pecans for up to eight years. Refrigeration at 0°C or less does not damage pecans nor does it increase the deterioration rate upon thawing (Woodroof, 1967).

Kernel moisture is approximately 30% when nuts dehisce (shuck splits), but gradually decreases before abscission (falling from the tree) (Heaton and Woodroof, 1970). High moisture in pecans is a major source of deterioration and can result in their becoming inedible within two weeks (Woodroof, 1967). Drying pecans to a kernel moisture of about 4.5% is necessary to avoid quality deterioration. Pecan quality begins to deteriorate from the time the nuts drop from the tree; it is best for pecans to be harvested, dried, and graded promptly, then stored under refrigeration, with proper nut moisture and relative humidity levels, within one month of harvest.

Frozen pecans are brittle and should be "tempered" by increasing the temperature slowly over a period of time (Woodroof and Heaton, 1967). Broken pecan pieces have a shorter shelf life due to an increase of exposed surface area (Wagner, 1980), and tempering will reduce pecan breakage. Also, pecans that are low in moisture are brittle. To increase the yield of unbroken halves and larger pieces, unshelled pecans may be soaked in water before shelling, although this will require that nut meats be dried back to a moisture level of about 4.5%.

Pecans may be stored shelled or in the shell, although shelling pecans has been found to reduce the storage life by one-half (Woodroof, 1967). If pecans are too moist when stored in the shell, the

excess moisture can result in leaching of tannins, which are water soluble, from the shell lining and corky middle. Tannins will cause nut meats to have a bitter flavor (Wagner, 1980; Polles, Hanny and Harvey, 1981). Shelling pecans before storage reduces the weight and volume to about one-half, and faulty nuts may be graded out before storage. Pecans are known to be "odor eaters"; they will absorb odors present in storage rooms which lower their quality (Stein, 1980). They will absorb off odors shelled or in the shell, as well as in ambient or refrigeration storage.

Numerous processes have been developed to maintain the fresh taste of nut meats without refrigeration. Many of these have had at least some degree of success. Dark or opaque packaging will reduce the penetration of ultraviolet light waves (Kays, 1979). Sucrose-based syrup coatings provide barriers to oxygen penetration (Godkin, Beattie, and Cathcart, 1951). Other methods for excluding oxygen include flushing with CO₂ and heat sealing (Holaday, Pearson, and Slay, 1979), replacing oxygen with nitrogen (Kays, 1979), and using monoglyceride coatings containing antioxidants (Shea, 1965; Luce, 1967; Senter and Forbus, 1979). Also used are steam conditioning (McGlamery and Hood, 1951; Forbus and Senter, 1976) and dielectric heat treatments (Senter, Forbus, Nelson, and Horvat, 1984). Heaton and Woodroof (1955) found that when pecan meats were ground to a fine consistency for pecan butter, keeping time (as determined by flavor and aroma) was extended with the use of antioxidants. They found that heating pecans to an internal temperature of 180° C for three minutes inactivated oxidative enzymes, but produced a slightly cooked flavor.

The existence of mycotoxins also affects the storage life of pecans. Micotoxins are described as:

. . . a group of toxic chemical compounds produced by certain strains of a number of species of fungi when they grow under favourable conditions on a wide variety of different substrates. As their generic name implies, these compounds are toxic to man and animals, causing diseases collectively known as mycotoxicoses. Fungi capable of producing such compounds are usually described as toxogenic (Food and Agriculture Organization of the United Nations, 1979, p. 3).

The existence of mycotoxins has been known for two centuries, but it is since the genesis of interest in the specific group of mycotoxins, aflatoxin, in 1961, that these compounds have attracted considerable attention (Food and Agriculture Organization of the United Nations, 1979). Strains of Aspergillus flavus and Aspergillus parasiticus have been reported in processed pecans (Lillard, Hanlin, and Lillard, 1970; Doupnik and Bell, 1971). Aflotoxins produced by strains of Aspergillus have even been found on pecans sold in retail markets (Lillard, Hanlin, and Lillard, 1970). Koehler, Hanlin, and Beraha (1975) examined 5,608 pecan samples and reported 148 isolates. Of these isolates, 93% of the A. parasiticus and 54% of the A. flavus were capable of producing aflotoxin.

Mold and aflotoxigenic aspergilli incidence do not appear to be associated with particular pecan harvesting procedures or with particular cultivars (Beuchat, 1978). However, the unbroken shell provides a protective barrier to contamination by the fungi Aspergillus flavus and Aspergillus parasiticus (Schroeder and Storey, 1976; Schroeder and Cole, 1977; Beuchat, 1978).

Although more research is needed, measures have been developed to control mold growth and toxin contamination; however, mold on shelled

nuts has been observed during processing and storage. Correct environmental conditions need to be more uniformly maintained in order to control mold growth (Beuchat, 1978).

Pecan Color

The testa (seed coat) of pecans tends to darken with age; therefore, color is a major consideration in the determination of quality pecans. The USDA has set color standards for grading pecans: (1) light, (2) light amber, (3) amber, and (4) dark amber. A "light" color is taken as an indication of a fully mature nut that has been properly harvested, processed, and stored. Nuts with this rating receive a premium price in the retail market.

At least 50% of shelled pecans in the market are darker than desired (Woodroof and Heaton, 1967; Kays and Wilson, 1977). These darker pecans are generally associated with rancidity, but in reality there are a variety of attributes other than age and rancidity that lead to darker kernel color.

Some cultivars have a naturally darker color but are not necessarily lower in flavor quality (Kays, 1979). Woodroof (1967) attributed color darkness to seasonal differences within varieties of cultivars and growing differences such as availability of moisture, location of trees, and horticultural practices. Heaton, Worthington, and Shewfelt (1975) found that color quality is highest when nuts are harvested soon after maturation (Kays, 1979). Delayed harvest, as well as early frost (Kays, 1979) result in exposure to damage from weather, producing dark pigmentation and bitterness (Heaton, 1974; Heaton and Beuchat, 1980). The earlier the nuts are harvested after

developing their color, the greater the assurance of their having a consistently high quality rating (Kays, 1979) with brighter and more uniform color (Wagner, 1980). Therefore, mechanical harvesting, which speeds harvest, helps to minimize color darkening. Artificial earlier harvest can be accomplished with the use of an ethylene-releasing chemical sprayed on both the tree and the fruit (Hinrichs and Hopfer, 1970). According to Heaton (1974), pecans left unharvested until temperatures fall below 4°C are prone to severe quality damage, including discoloration.

Insect damage can also cause a darkening of kernel color. The damage may be limited to the area adjacent to the insect's penetration, or it may affect one or both cotyledons (Kays, 1979). Such damage usually results in the total loss of the nut.

Ammonia gas, used in refrigeration, is another factor affecting kernel color which causes an irreversible black discoloration to the testa (Woodroof, 1967; Heaton, 1974). Kays and Wilson (1977) investigated the feasibility of lightening the color of dark pecans, which are otherwise of high quality, to increase their market value. Pecans soaked in dilute aqueous solutions of up to 0.125 M phosphoric acid proved to be the most effective, with no detectable affect on flavor.

While color is not a definite means of determining pecan quality, it is used extensively since color is easy to measure (Kays, 1979). The grower needs to be concerned about the kernel color, since most shellers gauge the price paid predominately by color (Kays, 1979).

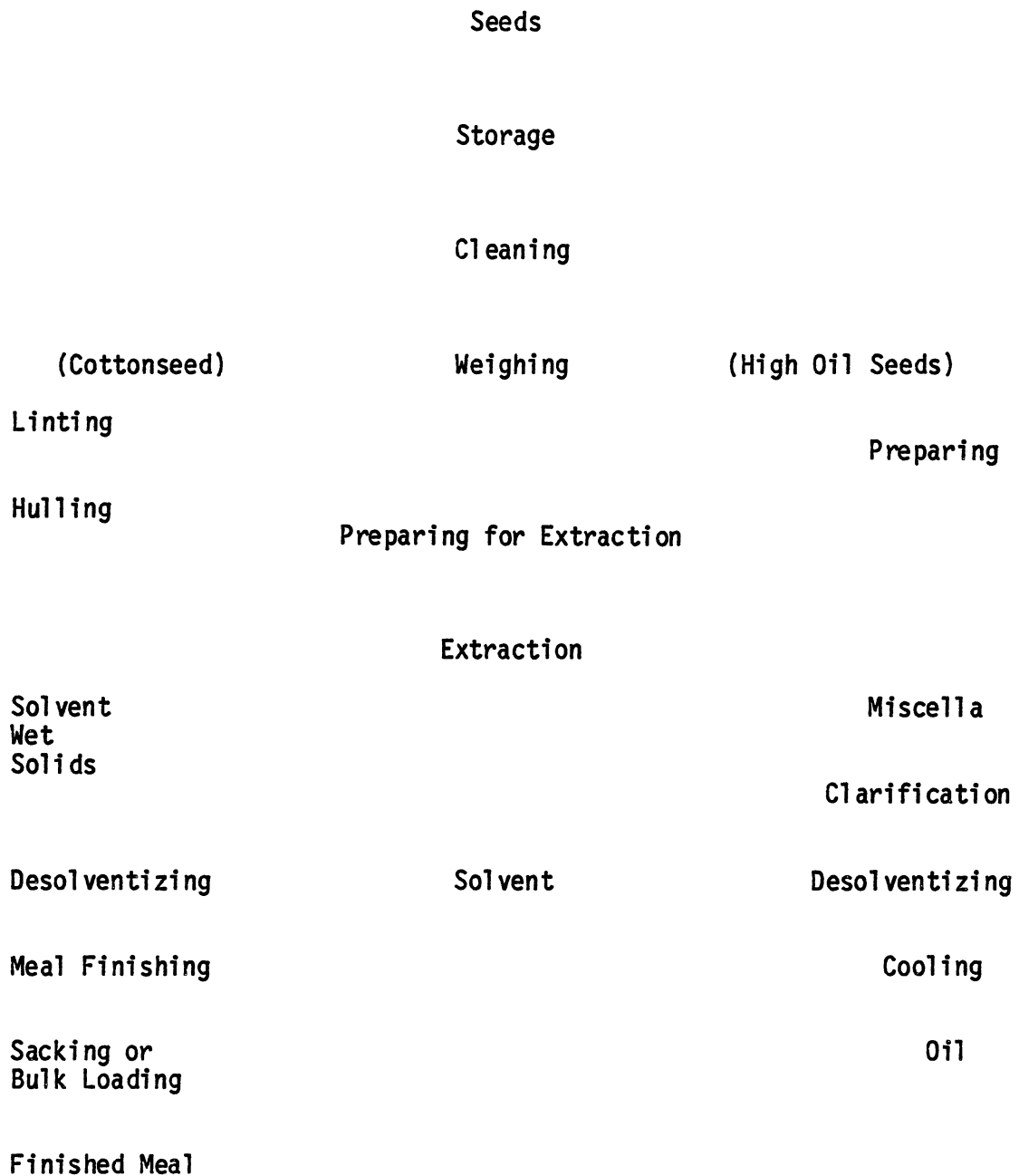
Oil Extraction

Oil can be obtained from foods by two general methods: screw

pressing (the oil is pressed or squeezed from the oil source) and solvent extraction (a fat solvent is used to dissolve the fat from the matrix and then evaporated off, leaving the oil). Oil is recovered more thoroughly and efficiently using solvents and this is the method used for most edible oils.

Procedures for solvent extraction of oil from food products are well developed (Deiler and Frads, 1910; Woodroof, 1967; Pyriadi and Mason, 1968; Beuchat and Worthington, 1978; Bhuchar, Agrawal, and Sharma, 1981). Soxhlet and Szombathy (cited in Bhuchar, Agrawal, and Sharma, 1981) introduced the Soxhlet extractor in 1879, and by 1949, Karnofsky (1949) described several different commercial extractors in production. Newer versions have since been introduced (Bhuchar, Agrawal, and Sharma, 1981; Francis, 1979; Ayres, Branscomb, and Rogers, 1974), but the basic steps in solvent extraction remain as outlined by Karnofsky in 1949 (Figure 3).

Although often updated and improved, most extraction methods are basically similar but vary, depending on the solvents used and the final product. The oil solvent, n-Hexane (Hexane) is probably the most commonly used extractant in both research and commercial applications (Pyriadi, 1967; Pyriadi and Mason, 1968; Holaday and Barnes, 1973; Gutcho, 1979; Bhuchar, Agrawal, and Sharma, 1981; Balentine, 1984). Hexane can also be mixed with other solvents, such as acetic acid, as in the process patented by Hensarling, Jacks, and Yatsu (cited in Gutcho, 1979) in 1976. Extraction may be by immersion or percolation. In the immersion-type, the solids are agitated in the solvent; in the percolation-type, the solvent is sprayed through fixed beds of solids (Woodroof, 1967).



Source: G. Karnofsky, "The Mechanics of Solvent Extraction," J. Am. Oil Chem. Soc. (1949).

Figure 3. General Solvent Extraction Process

Peanut oil is routinely extracted, and the remaining oil-extracted peanut flour has attracted a great deal of interest as a high protein food source (Woodroof, 1966; Rhee, Cater, and Mattill, 1972). Researchers such as Ory and Conkerton (Anonymous, 1982) prepared peanut flour which contained 50 to 60% protein in order to raise the nutritional value of snack foods. Other researchers have produced edible peanut flour and have found the bland flavor and light tan color facilitates its incorporation into food products (Ayres, Branscomb, and Rogers, 1974).

In 1938, researchers prepared a transparent, bland, odorless, pecan oil comparable to olive oil (Woodroof, 1967). There was no oil rancidity reported in 12 months, but no stability reports were given on the pecan meal. In 1949, Kester reported that 300 tons of pecan oil were produced annually for specialty products, including cosmetics and salad oils, and almost 20 years later this amount had not increased (Brinson, 1974). Little, if any, pecan oils are currently produced for such purposes. Woodroof (1967) reported that, although pecans were a rich source of oil, extraction was not economical, except for specialty items. The percentage of oil recovery was also small compared to cottonseeds, soybeans, corn, and peanuts. Pecan oils are still extracted (Woodroof, 1967; Pyriadi and Mason, 1968; McWatters and Cherry, 1977), but mainly for analytical purposes.

Extraction methods using hot, distilled water as the solvent were reported by McWatters and Cherry (1977) with the purpose of defining the emulsion and foaming properties of the pecan flour. Other researchers have used water as an extraction medium (Rhee, Cater, and Mattil, 1973; Holaday and Barnes, 1973; McWatters and Heaton, 1974;

McWatters and Cherry, 1975) to determine additional functional properties such as fat and water absorption, texture modification, color control, and whipping properties of the flour. The high protein pecan flour could be important in food systems for both nutritional and functional properties. Using hot water as an oil extractant is less efficient than using a fat solvent such as hexane and causes loss of water soluble nutrients. However, water extraction is sparing of the oil solvent soluble vitamins A and E (Heston, 1930; Woodroof, 1967).

Summary of Review

Although Oklahoma possesses the conditions necessary for ideal pecan growth, problems associated with poor quality continue to keep pecans an underutilized and seasonal food product. Methods for maintaining quality have been developed, but pecans of inferior quality still reach the marketplace. Solvents used to extract other food oils have been used with pecans to produce pecan oil or pecan flour. Some attempts have been made to develop these pecan components in order to increase year round marketability; however, not nearly enough has been done. Further study is needed in developing products made from pecan components and in testing their quality and acceptability. Also, no work has been reported where fat was extracted from various sizes of pecan pieces.

CHAPTER III

INTRODUCTION

Pecans (Carya Illinoensis) are a leading horticulture crop in the southwestern United States (Senter and Horvat, 1976), yet keeping quality is difficult to maintain. Because of poor keeping quality, pecans have become a seasonal food product.

Pecan nuts contain high levels of oils, from 60 to 70% or more. Pecan oils are 92 to 97% unsaturated triglycerides, composed mostly of 18 carbon unsaturated and polyunsaturated fatty acids; oleic and linolic acids predominate. The saturated fatty acids, mostly palmitic and steric, range from 3 to 8% (Senter and Horvat, 1978). The high amount of unsaturation makes pecans a recommended health food (Stein, 1980), but also makes them more susceptible to oxidation and rancidity (Beuchat and Worthington, 1978). Unsaturated fatty acids undergo autoxidation, initiated from light, heat, oxygen, enzymes, or other biological catalysts (Perkins and Visek, 1983), which results in off-flavors, off-odors, and in extreme cases, toxic byproducts (Stuckey, 1981). Smaller pecan pieces autoxidize faster than larger pecan pieces (Forbus and Senter, 1976).

Since pecan kernels are semiperishable, they should be refrigerated (<0° C) to maintain optimum quality (Woodroof, 1967; Heaton, 1974). As the cost of energy increases, proper pecan storage is more costly. Researchers have attempted to improve the quality of

pecans and decrease the cost of storage by recommending proper harvesting procedures, storage conditions, and marketing practices (Woodroof, 1967; Woodroof and Heaton, 1967; Heaton et al., 1975). Numerous studies have been conducted in search of alternative storage conditions, with only minimal degrees of success. Some of these areas of investigation include: packaging material to provide a barrier to ultraviolet light waves (Kays, 1979); sucrose-based syrup coatings to provide barriers to oxygen (Godkin et al., 1951); package flushed with CO₂ and heat sealing (Holaday et al., 1979); replacing oxygen with nitrogen and heat sealing (Kays, 1979); and using monoglyceride coatings containing antioxidants (Shea, 1965; Luce, 1967; Senter and Forbus, 1979). Also used are steam conditioning (McGlamery and Hood, 1951; Forbus and Senter, 1976), and dielectric treatments (Senter et al., 1984). Although improvements have been made, it is estimated that 50% of the pecans sold in retail outlets are sold below USDA quality standards (Williams et al., 1973).

Not only are pecans susceptible to oxidation and high storage costs, pecan producers must also deal with erratic production caused by late frosts, which destroy entire crops, and alternate-year bearing. This has created marketing as well as managerial problems for pecan producers (Blake and Clevenger, 1980). Horticulturally, no single factor will alleviate alternate bearing (Sparks, 1975). However, it has been recommended that growers and producers try to predict these changes in production so that they can plan financial and marketing strategies accordingly (Blake and Clevenger, 1980).

Pecan production is gradually increasing (Peterson and Johnson, 1978), but there is evidence that per capita consumption is actually

declining (Stebbins, 1980). Thus, we see an increase in supply with no growth in demand; this leads to an imbalance in the market. Stebbins (1980) indicated that one reason for low consumption of pecans is that poor quality pecans are allowed on the market. Since the instability of pecan oil is the prime reason for the poor quality, it has been the subject of much research.

Procedures for solvent extraction of oil from food products are well developed (Deiler and Frads, 1910; Woodroof, 1967; Pyriadi and Mason, 1968; Beuchat and Worthington, 1978; Bhuchar et al., 1981). The Soxhlet extractor was introduced in 1879 (Bhuchar et al., 1981). Since then, several different commercial extractors have been introduced and many improvements have been made, but the basic method of oil seed extraction using oil solvents remains largely as developed and outlined by Karnofsky in 1949 (Karnofsky, 1949; Rhee et al., 1972, 1973). The oil extraction solvent used varies, depending on the purpose and final objectives sought. The solvent n-Hexane has been used by researchers and commercial oil producers for vegetable oil extractions, including peanut and pecan oils (Pyriadi, 1967; Pyriadi and Mason, 1968; Holaday and Barnes, 1973; Gutcho, 1979; Bhuchar et al., 1981).

In 1938, researchers prepared a transparent, bland, odorless pecan oil comparable to olive oil (Woodroof, 1967). Even after 12 months, there was no rancidity in the oil, but no stability reports were given on the pecan meal. In 1949, Kester reported that 300 tons of pecan oil were produced annually for specialty products, including cosmetics and salad oils; 20 years later, this amount had not

increased (Brinson, 1974). Today, pecan oils are extracted mainly for analytical or research purposes.

This present study was conducted to identify a process which would partially extract oil from pecan pieces, determine acceptability using a partially trained taste panel, and determine the subjective differences among various sizes of pecan pieces, both treated and untreated.

Materials and Methods

Pecan halves were from native pecans grown and harvested in Oklahoma in 1984, and were provided by the Oklahoma Pecan Commission. U.S. Standard Sieve Series and a Syntron Sieve Shaker were provided by the Oklahoma State University Civil Engineering Department. The pecans were stored at 0° C in a cold storage space used only for research pecans to avoid flavor contamination from other foods. HPLC grade n-Hexane (hexane) (CH₃(CH₂)₄CH₃) was obtained from Allied Fisher Scientific.

Preparation of Pecans

Pecans were removed from cold storage as needed and were chopped into pieces with a Sunbeam food processor (Model 14-21). Kernels were chopped one cup at a time and accumulated in batch sizes of 10 to 20 cups in five-gallon covered plastic containers.

U.S. Standard Sieve Series was used to separate pecan pieces by size. The sieves were stacked so that the largest sieve opening was on top and the smallest on the bottom. The six sieve sizes used were: 1/4 inches (6.34 mm), No. 4 (4.76 mm), No. 8 (2.38 mm), No. 10

(2.0 mm), No. 16 (1.19 mm), and No. 20 (0.85 mm). A bottom pan collected all smaller particles. Approximately two cups of pecan pieces at a time were placed in the top sieve on the Syntron sieve shaker (Model TSS31). The shaker dial was set on 50 (approximately 90 volts) and set to run for three minutes, separating the pieces into the sieve sizes. Only the pecan pieces remaining in the sieve size Nos. 1/4, 8, and 16 were used as the large, medium, and small pecan pieces in this research.

Extraction Process

Sized pecan pieces (approximately 100g) were rinsed twice with hexane, 150 ml of hexane for the first rinse and 150 ml for the second. After the second rinse, the pecan pieces were placed in a dehydrator (Excalibur Model-301) and dried at 145° F for 72 hours. All extractions were done at ambient conditions of temperature, relative humidity, and light sources (sunlight from windows or artificial fluorescent light) with adequate fan-driven ventilaton. The hexane, with dissolved oil, was distilled and refined to determine the amount of fat extracted (distillation and refining of oil) (Appendix A).

Subjective Tests

Prospective panelists were tested to prove their ability to detect the four basic flavors and to discriminate among four levels of sweet solutions. The final panel consisted of two males and eight females, ranging in ages from 21 through 42. During a training session they were familiarized with the flavors and odors characteristic

of pecans; the research evaluation criteria, score cards, and test procedures were explained.

The instrument developed to evaluate the pecan pieces was a modified magnitude estimation scale (Muskowitz, 1974) which allowed panelists to express small and large differences in sensory evaluation. The vertical lines on the evaluation score cards were 5 cm in length and results were recorded on a scale of 0-50, with the most desirable rating being 50 for attributes. (Copies of instructions for the panelists and the score card appear in Appendix B.)

Panelists were given three sizes and two treatments each testing day, with three replications. Samples were provided in white souffle cups and deionized water was provided for mouth cleansing. Panelists sat in separated booths in the testing room. The panel was conducted in the month of May in ambient room conditions and lighting.

Objective Tests

Fatty acid composition of the extracted pecan oil was determined according to the procedure of Mason and Waller (1964), modified by Rudolph (1971). Reagents used to methylate the fatty acids were: 4 ml sodium benzene, 1 ml 2,2-dimethoxypropane, and 0.5 ml 10±2% methanoic HCl. Analysis of fatty acids was carried out with a Perkins-Elmer 990 gas chromatograph equipped with a glass column (7.5 ft x 1/4 in O.D.) packed with 20% diethylene glycol succinate on 100/120 Chromosorb W. An isothermal program was utilized with column temperature, 180° C; injector temperature, 250° C; flame isolation detector, 250° C; and nitrogen carrier flow at the detector of 40 ml/min.

The percentage of oil present in the research pecans was established using the official AOAC method for fat/oil determination. Finely ground pecan samples (approximately 1 g) were weighed, wrapped with two layers of Whatman (No. 1) filter paper, and extracted with petroleum ether for 24 hours in a Goldfish extraction apparatus. The ether was removed and the samples were cooled in a dessicator and were subsequently weighed. Moisture determination was established by placing chopped pecan samples in a drying oven 90° to 100° C and drying to a constant weight.

Oil remaining in the three sizes of treated pecans was determined by difference. Hexane rinses from weighted lots were placed in dried, tared boiling flasks and evaporated using a Unitized Heater (Precision Scientific 65500). Weight of the remaining fat was compared to the total fat content and used to calculate the fat extracted and the amount of fat remaining in the various sizes of pecan pieces.

Research Design

A factorial arrangement of treatments in a split-plot experimental design was used for subjective data developed in this research (Snedecor and Cochran, 1974; Bartz, 1981). The main unit treatment factor was the panelists. The subunit treatment factors were the treatments and sizes of pecans. The dependent variables were the taste panel sensory evaluations of these attributes: nutty aroma, absence of off-odors, texture, sweetness, full nut flavor, absence of off-flavors, lack of unpleasant aftertaste (30 seconds after swallowing), and overall acceptability. F tests from an analyses of variance procedure were used to determine if significant differences existed

among pecan sizes and the pecan treatments, followed by Duncan's Multiple Range Tests to determine the location of significant differences, with a significance level of $P < .05$.

Results and Discussion

Subjective Evaluation: Smell

NUTTY AROMA, as indicated in Table I, was greater in the medium and large pecan pieces which were rated as having more full nut aroma than the small pieces, but with no significant difference. Table II indicates that the treated pecans had a slightly higher mean value than untreated pecans, but with no significant difference.

OFF-ODOR ratings were high, indicating little off-odors in all pecans tested, but the large pieces did have a significantly higher mean level than the small pieces, indicating that the large pieces had less off-odor than the small (Table I). There was no difference in off-odor detected between the treated and untreated pecans, as seen in Table II.

There was a significant interaction between panelist and size for off-odors ($P = .0125$) and between panelist and treatment for off-odors ($P = .0130$), as seen in Figures 1 and 2. This interaction indicates that the panelists did not respond in the same manner to the three sizes, or to the two treatments, when assessing off-odors.

Subjective Evaluation: Taste

TEXTURE was perceived by the panel as being different, due to the size of the pecan pieces. As seen in Table I, the small pecan pieces

TABLE I
SENSORY EVALUATION OF PECAN PIECES BY SIZE*

Pecan Size	Smell		Taste				Acceptability	
	Aroma	Off-Odors	Texture	Sweetness	Full Nut Flavor	Off-Flavors	Unpleasant Aftertaste	Overall Acceptability
Large	21.2 ^a	45.4 ^a	42.2 ^a	34.5 ^a	37.1 ^a	37.2 ^a	36.6 ^a	37.7 ^a
Medium	22.1 ^a	42.8 ^{ab}	36.3 ^b	30.7 ^b	36.1 ^a	37.5 ^a	34.3 ^a	36.8 ^a
Small	19.4 ^a	42.1 ^b	14.6 ^c	17.2 ^c	22.6 ^b	22.4 ^b	18.7 ^b	25.7 ^b

*Mean values based on a scale of 0 through 50 where 50 is most favorable and 0 is least favorable.

^{abc}Means within a column followed by a common letter are not significantly different at the P<.05 level as analyzed by the Duncan's Multiple Range Test.

TABLE II
 SENSORY EVALUATION OF PECAN PIECES BY TREATMENT*

Pecan Treatment	Smell		Taste				Acceptability	
	Aroma	Off-Odors	Texture	Sweetness	Full Nut Flavor	Off-Flavors	Unpleasant Aftertaste	Overall Acceptability
Treated	21.2 ^a	43.3 ^a	31.2 ^a	30.2 ^a	33.7 ^a	33.8 ^a	35.1 ^a	32.0 ^a
Untreated	20.7 ^a	43.6 ^a	30.8 ^a	24.8 ^b	30.1 ^b	30.9 ^a	31.7 ^b	27.7 ^b

*Mean values based on a scale of 0 through 50 where 50 is most favorable and 0 is least favorable.

^{abc}Means within a column followed by a common letter are not significantly different at the P<.05 level as analyzed by the Duncan's Multiple Range Test.

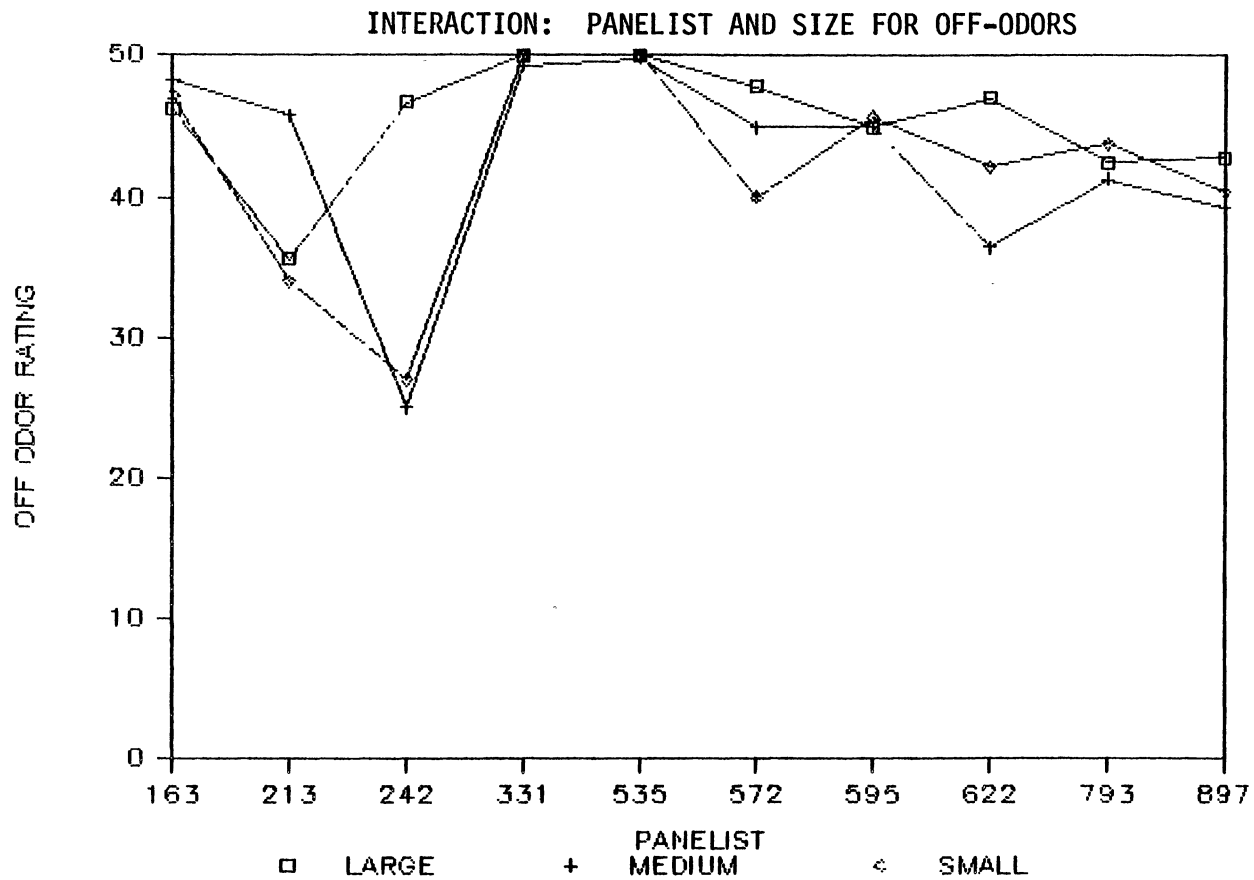


Figure 1. Graph of Individual Panelists' Mean Response (Three Replications) to the Large, Medium, and Small Sizes of Pecans Showing the Panelist/Size/Off-Odor Interaction ($P=0.0125$)

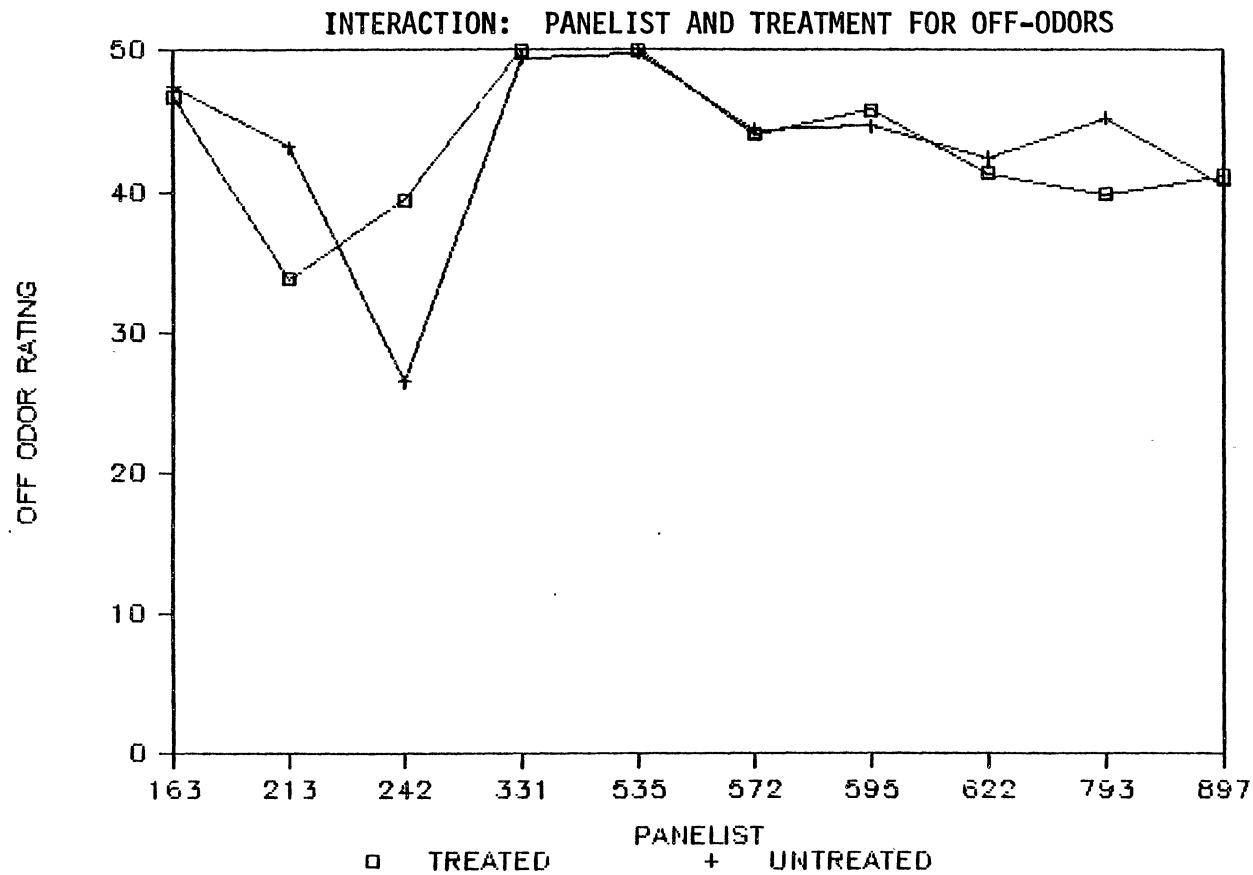


Figure 2. Graph of Individual Panelists' Mean Response (Three Replications) to the Large, Medium, and Small Sizes of Pecans Showing the Panelist/Treatment/Off-Odor Interaction ($P=.0130$)

were rated as being significantly lower (mean rating of 14.6) than the large pieces (mean rating of 42.2). The small pieces tended to have a dry, powdery mouth feel and are probably better suited for use in manufacturing and baking rather than being eaten alone. There was no significant difference perceived in texture between treated and untreated pecans, but treated pecans had a somewhat higher mean value than did the untreated (Table II). There was also a significant interaction ($P=.0003$) between size and panelist for texture, indicating that the panelists did not respond to size in the same manner for texture (Figure 3).

SWEETNESS was another attribute evaluated. Significant differences were observed in sweetness due to size (Table I), with large pieces (mean value 34.5) perceived as sweeter than the small pieces (mean value 17.2). The mean rating for the treated pecans was 30.2 and for the untreated the mean rating was 24.8 (Table II), with the treated being rated the sweetest in all sizes. It appears that when the pecan oil is removed, the percentage of carbohydrate remaining in the pecan will increase, leaving the pecan with a sweeter taste.

FULL NUT FLAVOR in large and medium sized pecan pieces was significantly greater (Table I) than the small by a significant difference, with 33.7 and 30.1 for large and medium, compared to 22.6 for small. There was also a significant difference between the treated and untreated pecans (Table II), with the treated having the higher mean value of 33.7, compared to the mean value of 30.1 for the untreated.

OFF-FLAVORS were determined to be significantly different (Table I) due to size, with large and medium pieces having less off-flavor

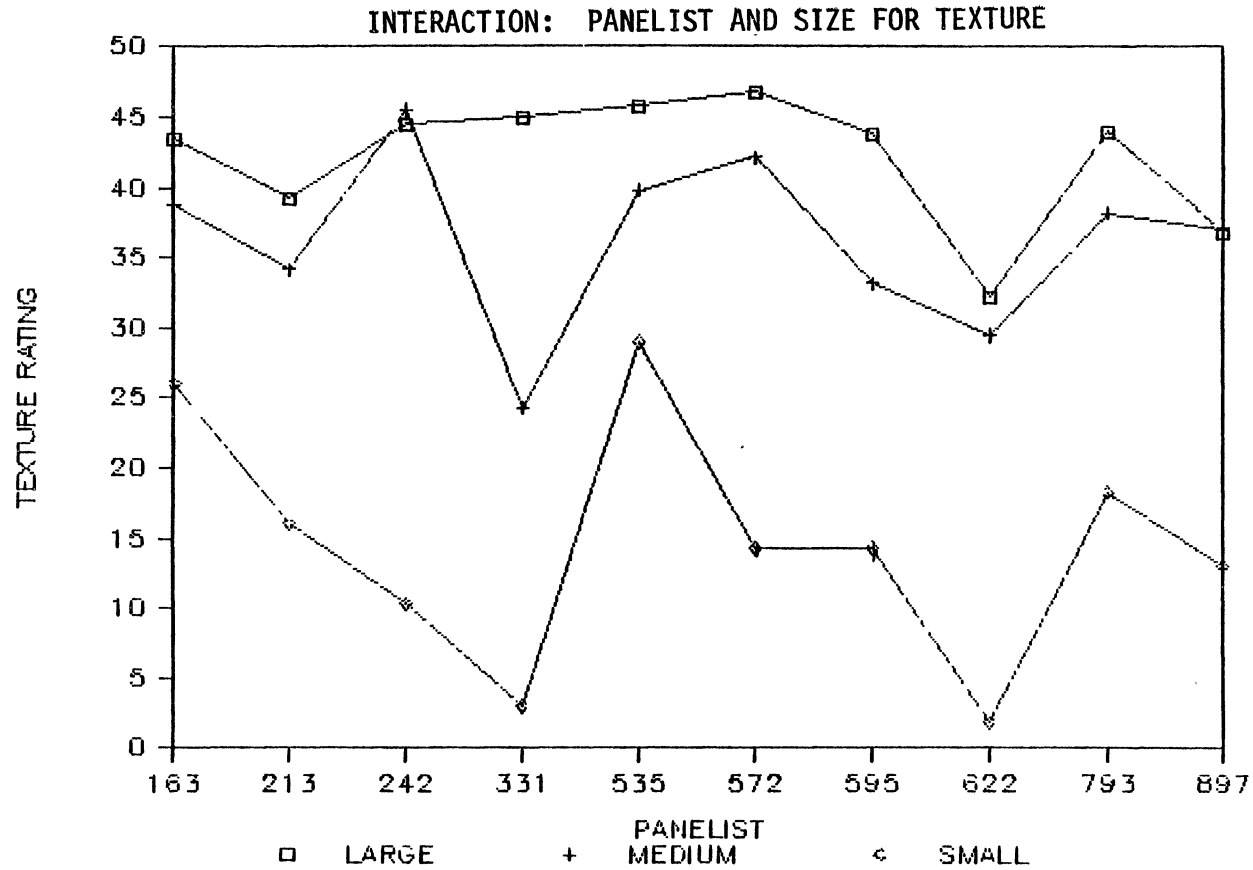


Figure 3. Graph of Individual Panelists' Mean Response (Three Replications) to the Large, Medium, and Small Sizes of Pecans Showing the Panelist/Size/Texture Interaction (P=.0003)

(mean values 37.2 and 37.5, respectively) than the small pecan pieces (mean value 22.4). There was no significant difference between the treated and untreated samples (Table II), although the treated samples had less off-flavor (mean rating 33.8) than the untreated samples (mean rating 30.9). There was also a significant difference in off-flavors due to the interaction of the panelists and size (with a $P=0.0074$), implying that the panelists, with respect to off-flavors, did not respond in the same manner to the sizes (Figure 4). The off-flavors detected by panelists decreased each testing day for both treated and untreated samples (Table III). Apparently, as the panelists became more familiar with the flavor of pecans, they identified as less off-flavor.

AFTERTASTE was determined by asking the panelists to detect aftertaste in 30 seconds after swallowing the pecan pieces. The large and medium pieces (with mean scores of 36.6 and 34.3, respectively) produced significantly less aftertaste than the small pieces (mean score 18.7) (Table I). Also, the treated pecans had significantly less aftertaste than untreated, with means of 35.1 and 31.7, respectively (Table II).

There were significant differences in interaction between panelists and size for aftertaste ($P=0.0003$) and between panelists and treatment for aftertaste ($P=0.0044$), implying that the panelists did not respond in the same manner to size or treatment with respect to aftertaste (Figures 5 and 6). Panelists indicated that, as in off-flavor, less aftertaste was observed each day (Table III). Also, the treated pieces had less aftertaste in every size than did the untreated pieces for all three sizes (Table IV).

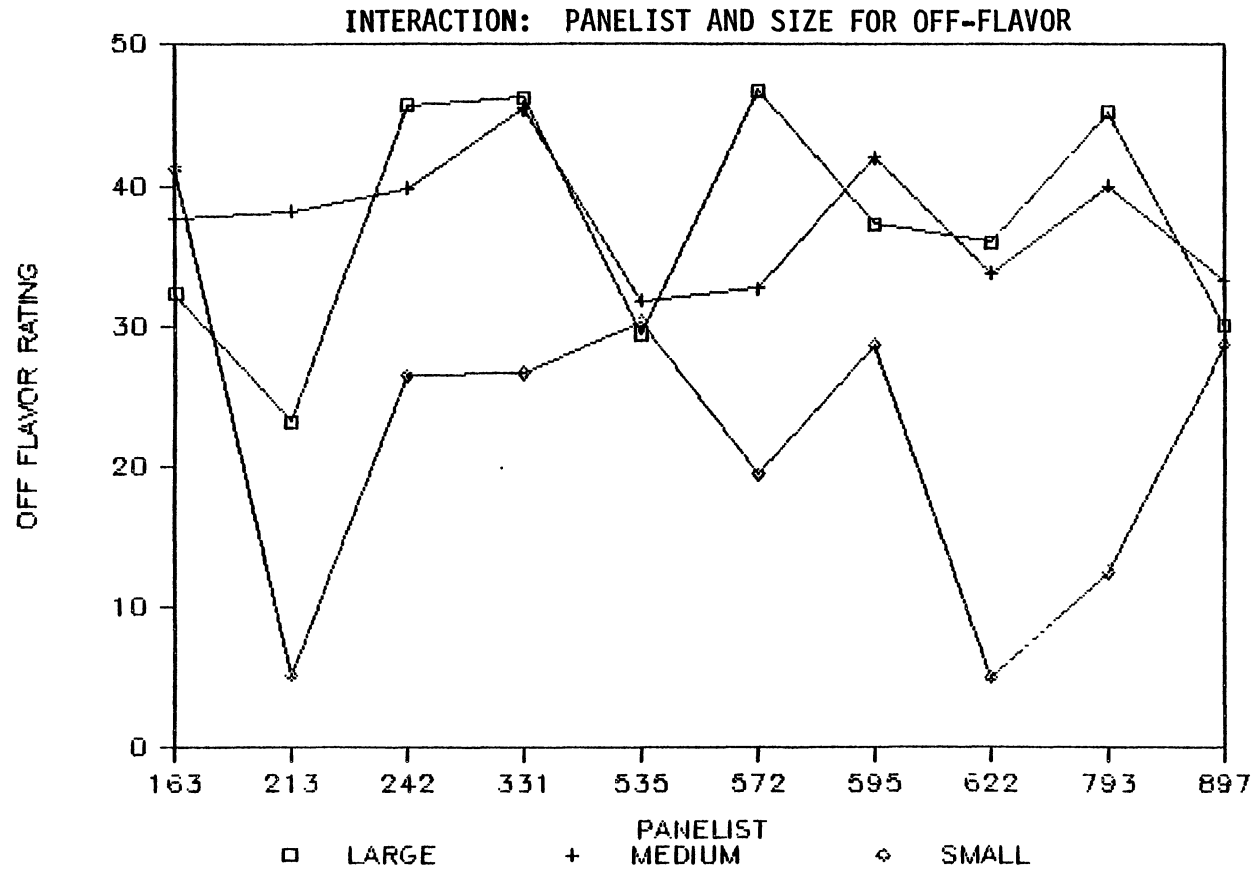


Figure 4. Graph of Individual Panelists' Mean Response (Three Replications) to the Large, Medium, and Small Sizes of Pecans Showing the Panelist and Size for Off-Flavor Interaction ($P=.0074$)

TABLE III
 MEANS OF ALL RESPONSES, SIZE, AND TREATMENT FOR
 DAYS MAY 17, 18, AND 19

Day	Aroma	Off-Odors	Texture	Sweetness	Full Nut Flavor	Off-Flavors	Unpleasant Aftertaste	Overall Acceptability
May 17	19.7	42.8*	33.2	27.5	33.7	32.7	34.9*	30.8
May 18	19.9*	43.8*	30.6	27.1	30.1	32.5	32.9	30.1
May 19	23.1	44.2	29.2	27.9	31.9	32.0	32.5	28.6

n=60

*n=59

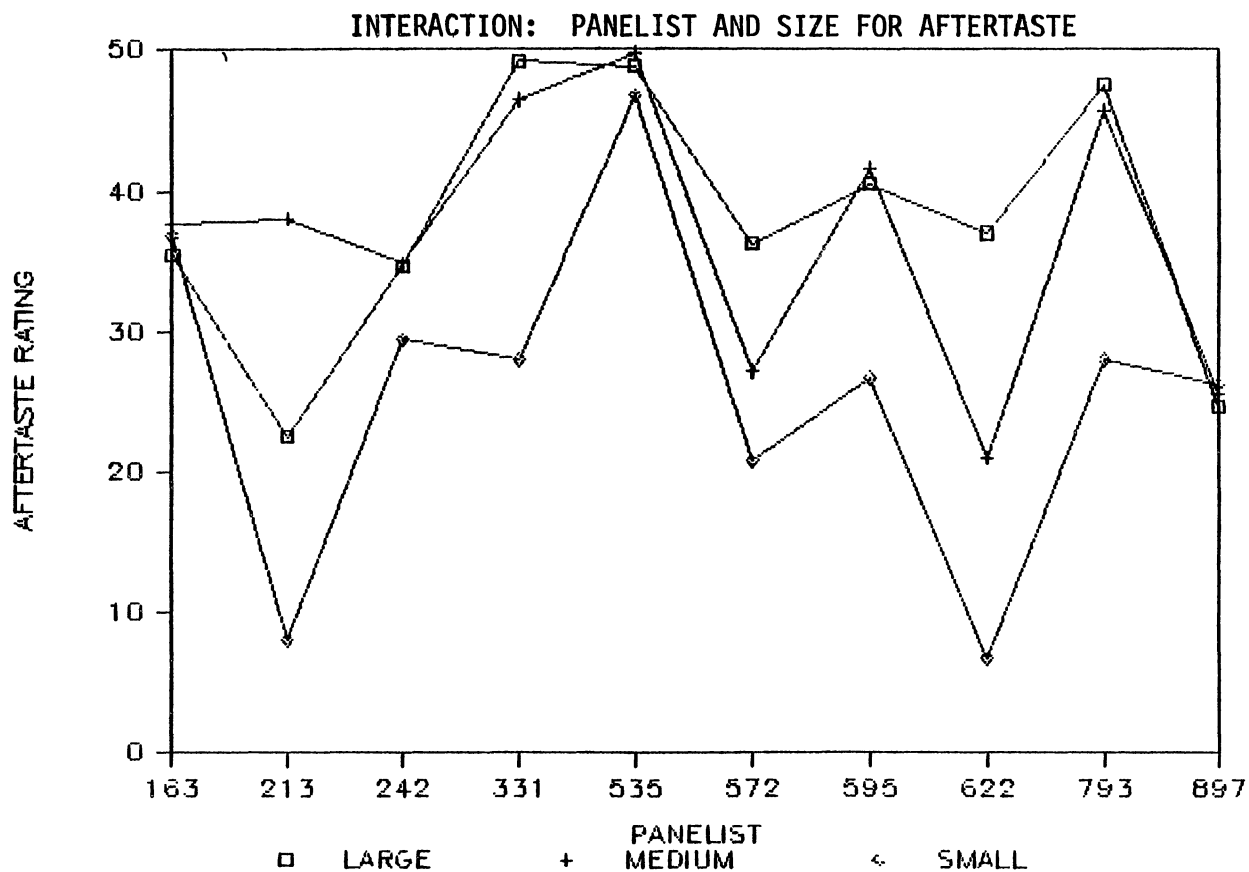


Figure 5. Graph of Individual Panelists' Mean Response (Three Replications) to the Large, Medium, and Small Sizes of Pecans Showing the Panelist and Size for Aftertaste Interaction ($P=.0003$)

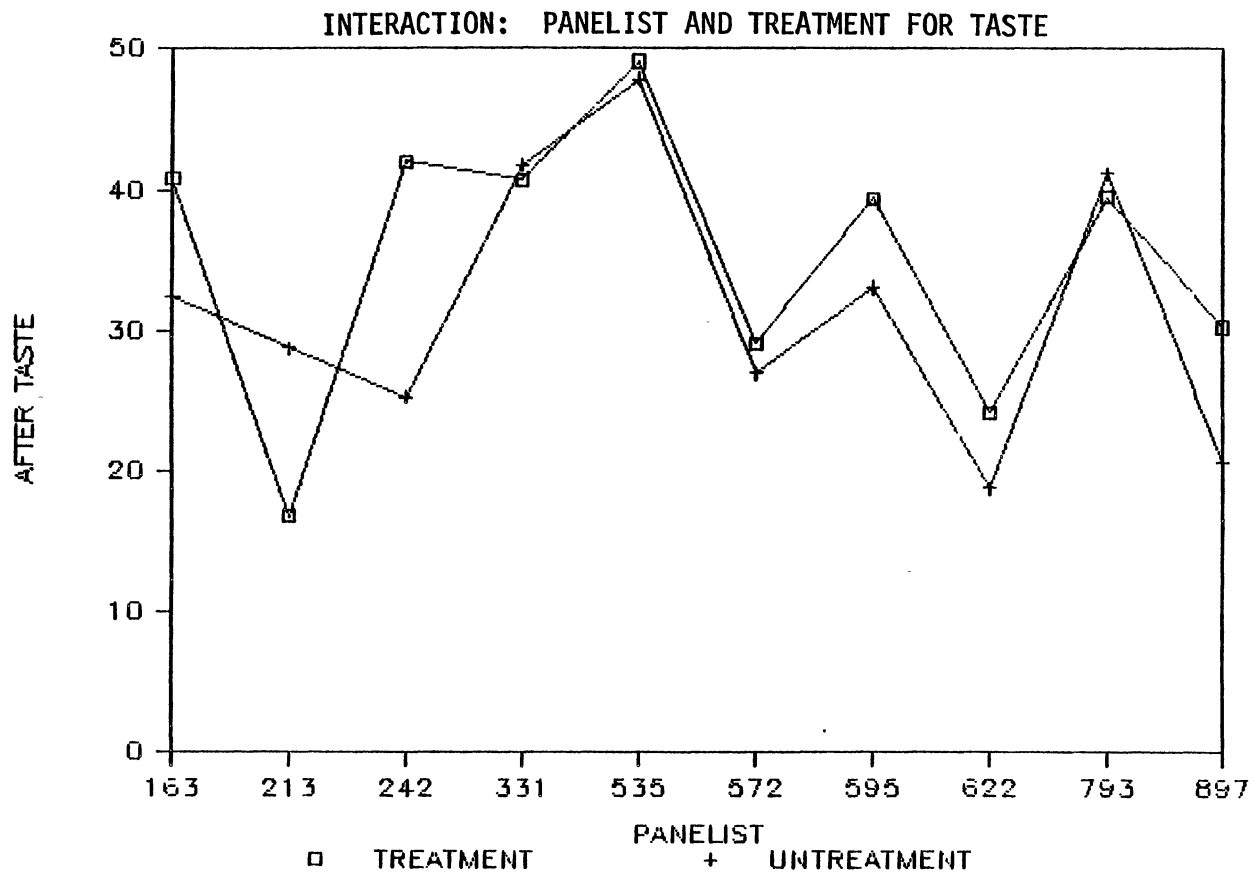


Figure 6. Graph of Individual Panelists' Mean Response (Three Replications) to the Large, Medium, and Small Sizes of Pecans Showing the Panelist and Treatment for Aftertaste Interaction ($P=.0044$)

TABLE IV
MEAN RESPONSES FOR ALL PANELISTS, ALL DAYS

Size	Treatment	Aroma	Off-Odors	Texture	Sweetness	Full Nut Flavor	Off-Flavors	Unpleasant Aftertaste	Overall Acceptability
Large	Treated	22.1	44.1	42.3	36.5	37.2	38.4	38.1	37.5
Large	Untreated	20.3	46.7	42.0	32.4	37.0	36.0	37.2	35.7
Medium	Treated	21.4	43.7	37.5	33.9	39.3	39.2	37.8	37.0
Medium	Untreated	22.7	41.9*	35.0	27.7	32.9	35.8	35.7	31.6
Small	Treated	19.9*	41.9*	13.9	20.1	24.6	23.9	29.2*	21.5
Small	Untreated	19.0	42.2	15.4	14.3	20.5	20.9	22.3	15.8

n=30

*n=29

Subjective Evaluation: Acceptability

The final evaluation made by the panel was overall acceptability. The panel rated the large and medium pecan pieces significantly higher than the small (Table I), with the large and medium pieces having means of 37.7 and 36.8, respectively, compared to the small pieces, with a mean of 25.7. Although there was a significant difference between sizes, all three pecan sizes were rated well within the acceptability range. There was also a significant difference noted between treatments for acceptability, with the treated pieces having a mean of 32.0 and the untreated a mean of 27.7. The treated pecans were more acceptable in all sizes than the untreated pecans (Table IV).

As can be seen in Tables I and II, the panelists tended to prefer the treated over the untreated pecan pieces for virtually all attributes. Also, the panelists uniformly preferred the large pecan sizes.

Subjective Evaluation: Interaction

In statistical analysis of sensory attributes, there were significant differences among the mean scores for interaction among particle size, treatment, and panelist for treated and untreated pecan pieces of fine, medium, and coarse particle size, due to panelist/size/off-odor; panelist/treatment/off-odor; panelist/size/texture; panelist/size/off-flavor; panelist/size/unpleasant aftertaste; and panelist/treatment/unpleasant aftertaste (Table V).

These areas of interaction indicated that there were differences in the way the panelists perceived the pecans. There was more

TABLE V
 PROBABILITY VALUES SHOWING THE ACTUAL PROBABILITY
 THAT THERE IS NO INTERACTION

Interactions	Smell		Taste				Acceptability	
	Aroma	Off-Odors	Texture	Sweetness	Full Nut Flavor	Off-Flavors	Unpleasant Aftertaste	Overall Acceptability
Size/Treatment	.8412	.2735	.3224	.8435	.2423	.9802	.2085	.5952
Panelist/Size	.8731	.0125*	.0003*	.3448	.1319	.0074*	.0003*	.2764
Panelist/Treatment	.3754	.0130*	.7221	.3458	.1593	.1502	.0044*	.1390
Panelist/Size/ Treatment	.0884	.8357	.9355	.7072	.6846	.6664	.5834	.7102

*Probabilities that are <0.005

interaction due to size than to treatment and most instances of interaction were associated with the panelists' detecting unpleasant flavors or odors, indicating that the panelists varied in their sensitivity to the unpleasant attributes of off-odor, off-flavor, and unpleasant aftertaste.

Objective Tests

TOTAL FAT present in the research pecans was determined through ether extraction. The mean percentage was 71.6 (n=3, SD=.19).

FATTY ACID composition of the research pecan comparison of retention times of the peaks of the gas chromatography and measurement of the areas under the peaks revealed the presence of the following fatty acids:

18:3	=	Trace	(Linolenic)
18:2	=	19.6%	(Linoleic)
18:1	=	43.7%	(Oleic)
18:0	=	14.0%	(Stearic)
16:1	=	8.0%	(Hexadecadenoic)
16:0	=	13.6%	(Palmitic)
14:0	=	1.1%	(Mysteric)

The levels of fatty acids in pecans differ widely, due to variations of maturity and growth conditions. The research pecans had a lower amount of unsaturated fatty acid than has been reported by others, but oleic acid (18:1) was the major fatty acid, and the proportion of fatty acids was similar to other reported data (Senter and Horvat, 1978).

MOISTURE content analysis of the research pecans indicated 2% moisture. According to the research literature, most pecans should range from 3.5 to 4.5% moisture; therefore, these particular pecans seemed slightly lower in moisture than the norm.

PERCENT FAT remaining in the pecan pieces varied with size, with more oil remaining in the large pieces, which retained 57.5%, the medium 53.6%, and the small retaining the least amount of oil, 34.3%.

Conclusions

Analysis of the sensory data showed that the hexane extraction (treated) pecans were rated higher than the unextracted pecans (untreated). This was true for all sizes of pecans. Apparently, the extraction process did not remove the natural pecan flavors, nor did it add unacceptable flavor of its own. Also, the panelists preferred the large pecan pieces over the small, whether treated or nontreated. The larger pecan pieces command higher retail prices and are the favorite in the retail market, so there is a prejudice in favor of large pieces that probably affected the panelists' judgment. Since small pecan pieces become rancid faster than the large sizes of pecans (Wagner, 1980), partial fat extraction might be investigated as a means of prolonging the shelf-life of pecans, particularly the smaller sizes. However, implications are that the small pieces, treated or untreated, would be better used in manufacturing rather than eaten directly as a snack food.

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CHAPTER IV

HYPOTHESES TESTING, SUMMARY, AND RECOMMENDATIONS

The purpose of this research was to develop a fat extraction process and to determine the effects of fat extraction on pecan kernels. Three pecan sizes were chosen for this study. The independent variables were the three sizes of the pecans and the two treatments tested. The dependent variables were the subjective evaluations by a 10-member taste panel. Objective evaluations included fatty acid analysis and total fat and moisture determinations. Subjective data were analyzed using analysis of variance and Duncan's Multiple Range Tests, with a significance level established at $P < 0.05$.

Hypotheses Testing and Summary

The first hypothesis (H_1) stated that there would be no significant differences between mean response levels of interaction between particle size and treatments. The sensory evaluation included the following attributes: nutty aroma, off-odors, texture, sweetness, full nut flavor, off-flavors, unpleasant aftertaste, and overall acceptability. In statistical analysis of sensory attributes, there were significant differences among the mean scores for interaction among particle size, treatment, and panelist for treated and untreated pecan pieces of fine, medium, and coarse particle size, due to panelist and size for off-odor, texture, off-flavors, and unpleasant

aftertaste; also due to panelist and treatment for off-odors and unpleasant aftertaste (Table IV). There was no interaction between treatment and size. Based on these results, the researcher rejected H_1 .

The second hypothesis (H_2) stated that there would be no significant differences between mean response levels of extracted pecan pieces and untreated pecan pieces. The sensory evaluation included the following attributes: nutty aroma, off-odors, texture, sweetness, full nut flavor, off-flavors, unpleasant aftertaste, and overall acceptability. Average mean scores for sweetness, full nut flavor, unpleasant aftertaste, and overall acceptability differed significantly ($P < .05$), due to pecan treatment. For each of these four attributes, the panelists preferred the treated pecans. Based on these results, the researcher rejected H_2 for these attributes but did not reject it for the other four. For three more of the attributes--aroma, texture, and lack of off-flavors--the mean scores rated higher for the treated pecans than the untreated, although not by a significant difference. Therefore, the hypothesis was not rejected for these attributes. The mean scores of several variables in both treated and untreated pecans increased with each testing day. This might indicate that the panelists became more familiar with increased exposure.

The third hypothesis (H_3) stated that there was no significant difference between mean response levels of untreated pecan pieces and partially oil-extracted pecan pieces of fine, medium, and coarse particle size. The sensory evaluation included the following attributes: nutty aroma, off-odors, texture, sweetness, full nut flavor, off-flavors, unpleasant aftertaste, and overall acceptability. In statistical analysis of sensory attributes, there were significant

TABLE IV
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Panelist/Size	.8731	.0125*	.0003*	.3448	.1319	.0074*	.0003*	.2764
Panelist/Treatment	.3754	.0130*	.7221	.3458	.1593	.1502	.0044*	.1390
Panelist/Size/ Treatment	.0884	.8354	.9355	.7072	.6846	.6664	.5834	.7102

*Probabilities that are <0.005

differences among the mean scores of pecan pieces. For the attributes of off-odors, texture, sweetness, full nut flavor, off-flavors, unpleasant aftertaste, and overall acceptability, the large pecan pieces scored significantly higher than the small pieces. Based on these results, the researcher rejected H_3 for each of these attributes. For the remaining attribute, aroma, the medium and large scores were both somewhat higher than the small, but not significantly higher; therefore, the hypothesis was not rejected for this attribute.

It was concluded from this study that the panelists preferred the hexane-extracted pecans to the untreated pecans and that they preferred the larger sized pecan pieces.

Areas of interaction indicated that there were differences in the way the panelists perceived the pecans. There was more panelist interaction due to size than to treatment and most instances of interaction were associated with the panelists' detecting unpleasant flavors or odors, indicating that the panelists varied in their sensitivity to the unpleasant attributes of off-odor, off-flavor, and unpleasant aftertaste.

Recommendations

Partial fat extraction of pecan pieces was very acceptable. Further studies will indicate if partial fat extraction will prolong pecan shelf-life to increase marketability.

Changes in basic laboratory techniques could be investigated to prolong autoxidation such as: laboratory equipment uniformity (metal, glass, plastic), temperature of oil solvent, vacuum oven, different

temperatures of heat in the dehydrator, vacuum sealing of extracted pecans, quantity produced, and storage temperature and humidity.

Cell microscopy of pecans might be informative in order to determine the difference of cell structure. This would determine inner cell and outer cell effects from the extraction process. Amino acid analysis of treated and untreated pecans would indicate any reduction of amino acid levels due to extraction.

The extracted oils should be studied for shelf-life, use of antioxidants, and food applications. Other possibilities include adding an antioxidant to pecan oil and reincorporating it into the extracted pecan for extending the shelf-life.

The small, extracted pecans were less favorable to the panelists than were the larger pecan pieces. The smaller pecan pieces have a lower market value, indicating a need for research into manufactured food sources for the small sizes, including pecan flour.

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APPENDIXES

APPENDIX A

EXTRACTION PROCEDURE

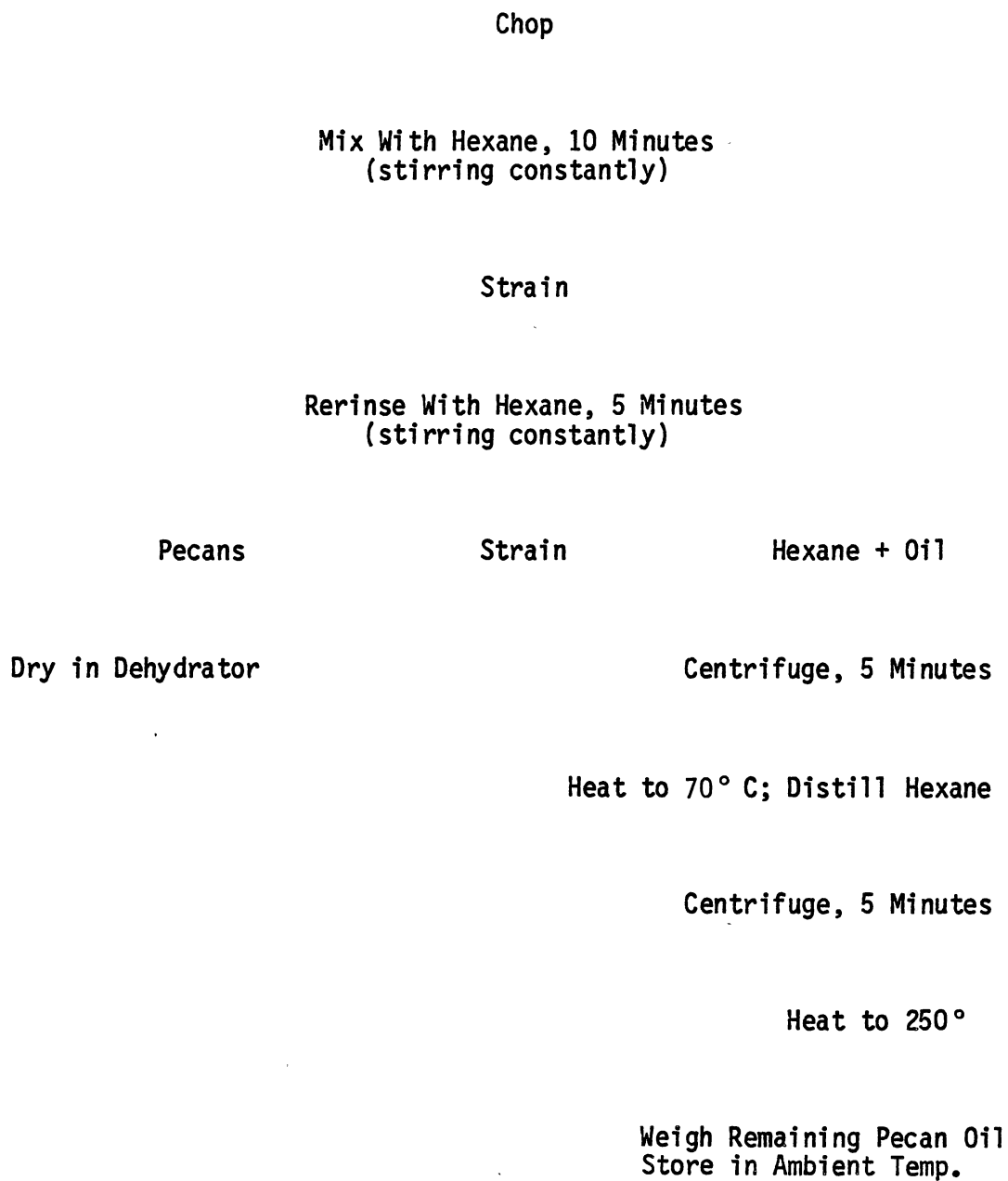


Figure 4. Extraction Process

Ether Extractable Fat

1. Prewrite numbered beakers and numbered glass thimbles with cotton and keep these paired.
2. Place the beakers into the dessicator and leave them there until needed, at least 24 hours.
3. Place thimbles on scale, insert about 1 g double-wrapped, pecan butter down into the thimble. Be very accurate in weights.
4. Place weighed thimbles and beakers on ether extraction apparatus.
5. Place about one tin thimble full of petroleum ether (ONLY) in each beaker and attach beaker.
6. Turn circulating water on and turn burners on.
7. Run for 24 hours with periodical checking for leaks, etc.
8. Remove thimble from apparatus, catch ether in tin thimble and discard this ether.
9. Evaporate any remaining ether in beaker.
10. Place beakers in dessicator for 24 hours.
11. Weigh beakers for final weight.

CHEMISTRY 1225
DISTILLATION OF PETROLEUM

SPECIAL APPARATUS: Distilling flask (200 mL); condenser (straight tube, with jacket connected by rubber stoppers); thermometer (360° C).

MATERIALS: Copper wire (small pieces); petroleum (crude oil); potassium permanganate solution (0.05%); sodium hydroxide (6 N); sulfuric acid (6 N and concentrated).

PREPARATORY QUESTIONS:

1. Define: (a) distillation; (b) fractional distillation; (c) "flash point."
2. Is crude oil (petroleum) a mixture or a compound?
3. In the fractional distillation of petroleum, what are: (a) the boiling point limits of the gasoline fraction? (b) The naphtha fraction? (c) The kerosene fraction? (d) The lubricating oil and wax fraction?
4. What would happen to a dilute solution of potassium permanganate if it were to be shaken with a mixture containing: (a) sulfides (such as hydrogen sulfide)? (b) Unsaturated hydrocarbons (such as ethylene)?

PROCEDURE:

A. Obtain the apparatus listed above and assemble it as illustrated in Fig. 1 (on last page). A is a 200-mL distilling flask resting on a wire gauze (F) and supported on a ring stand. The side arm of the flask passes through a 1-hole cork, which fits tightly in the neck (L) of the condenser (B). The condenser is supported firmly in an inclined position by means of a clamp and ring stand. Connect the lower side tube (M) of the condenser to the water faucet by means of a rubber tube and run a second rubber tube from the upper side tube (N) to the sink. Be sure all connections are tight. Turn on a slow stream of water, allowing it to run into the lower end of the condenser and out at the upper end. *Obtain 100 mL of crude oil. Note its color and odor and test its solubility by adding 2 or 3 drops to 10 drops of water and another 2 or 3 drops to 10 drops of ethyl alcohol. (Obs. 1.) Finally, introduce the remainder of the crude oil into the distilling flask, being careful not to allow any of it to run out the side arm. This is best done by using a funnel with a stem long enough to reach below the side arm. Carefully insert a 360° C thermometer through a cork. (CAUTION: Ask your instructor how to avoid breaking your thermometer while doing this.) Fit the cork tightly into the neck of the flask, adjusting the bulb of the thermometer so that it is just below the side arm.

Be sure that the apparatus is placed so that any air currents in the room will not blow fumes from the lower end of the condenser toward the distilling flask; then place a 50-mL graduated cylinder (E), in which is resting a small funnel (D), under the lower end of the condenser tube. Let the end of the tube drop as far down into the funnel as possible.

When all is ready, begin to heat the flask carefully and gently. Observe carefully the temperature at which the first drop of distillate falls into the graduated cylinder and record this temperature in the table at the end of the experiment as the "initial boiling point" of the crude oil. Continue the distillation, watching the temperature carefully. When the thermometer reads exactly 75° C, note and record in the table the volume of distillate that has collected in the cylinder. Observe the continued rise in the temperature, and for each interval of 25° observe and record the amount of distillate collected in the graduated cylinder.

*Have your instructor check your apparatus before adding crude oil.

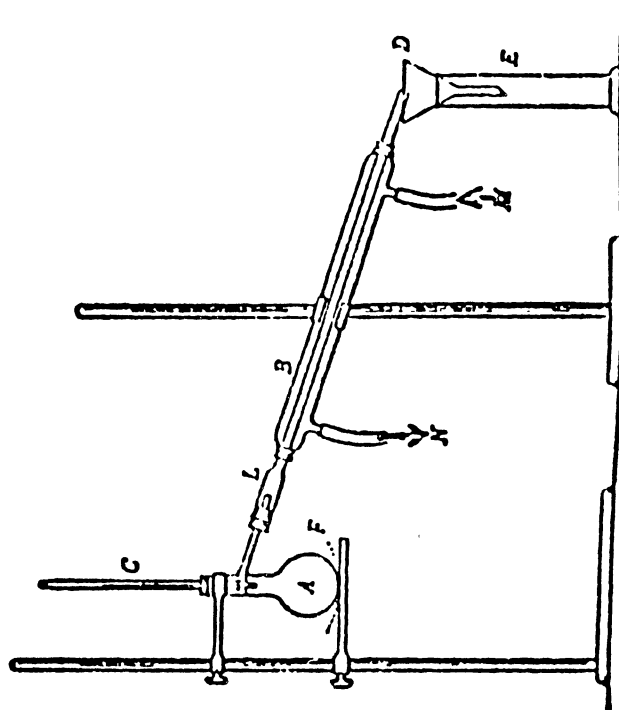


Fig. 1. Distillation Apparatus

APPENDIX B
SUBJECTIVE EVALUATION

**QUESTIONNAIRE FOR
RANKING**

NAME: _____ CODE # _____

DATE: _____

Rank these samples for sweetness. The sweetest sample is ranked first, the second sweetest sample is ranked second, the third sweetest sample is ranked third, and the least sweetest sample is ranked fourth.

Place the code numbers on the appropriate lines provided.

most sweet	second sweetest	third sweetest	fourth sweetest	least sweet
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**QUESTIONNAIRE FOR
DIFFERENTIATING FLAVORS**

Code # _____

Date: _____

Taste the liquid in each coded cup one at a time. Note the difference in taste of each solution. Place the code number of the cup on the line corresponding to the taste description you believe describes the type of solution tasted. Allow the solution being tested to contact all areas of the tongue. Please rinse after each trial by swishing distilled water around in your mouth.

SWEET _____

SOUR _____

SALTY _____

BITTER _____

PECAN EVALUATION

Instructions to Panel Members

At each session, you will be asked to evaluate six different pecan samples. Please evaluate the odor of the samples before you evaluate the taste. Evaluate the pecans in the order of the questionnaire, left to right, at the top of the score-sheet.

Make horizontal lines on the vertical line to indicate your rating of the pecan description. For each different cup of pecans you will be given a new evaluation sheet in which to mark your responses. There will be a total of six cups of pecan pieces and six evaluation sheets per person.

When tasting pecan pieces, it is important that you place several pieces of pecans in your mouth at one time.

Distilled water will be provided for rinsing purposes. Please use it to rid your mouth of one sample before evaluating the next sample.

For at least one hour before evaluation sessions, please try to avoid smoking, eating, drinking coffee or tea or chewing gum, as these may alter your sense of taste.

Thank you for volunteering your time and effort for our research project.

PECAN EVALUATION

Panelist Code # _____

Product Code# _____

Date _____

NUTTY AROMA
Full Nut Aroma



No Aroma

OFF ODORS
No Off Odors



Strong Off Odors

TEXTURE
Crisp Texture



Soft, Mushy

SWEETNESS
Sweet Taste



No Sweet Taste

FULL NUT FLAVOR
Full Nut Flavor



No Nut Flavor

OFF FLAVORS
No Off Flavors



Strong Off Flavors

UNPLEASANT AFTER TASTE
(30 seconds)

No After Taste



Strong After Taste

OVER-ALL ACCEPTABILITY
Very Acceptable



Not Acceptable

APPENDIX C

OBJECTIVE DATA

$C_{14} = .15 = 1.1\%$
 $C_{16} = 13.6 = 13.6\%$
 $C_{16} = 1.14 = 8.0\%$
 $C_{18} = 1.98 = 14.0\%$
 $C_{18} = 6.16 = 43.7\%$
 $C_{18} = 2.76 = 19.6\%$

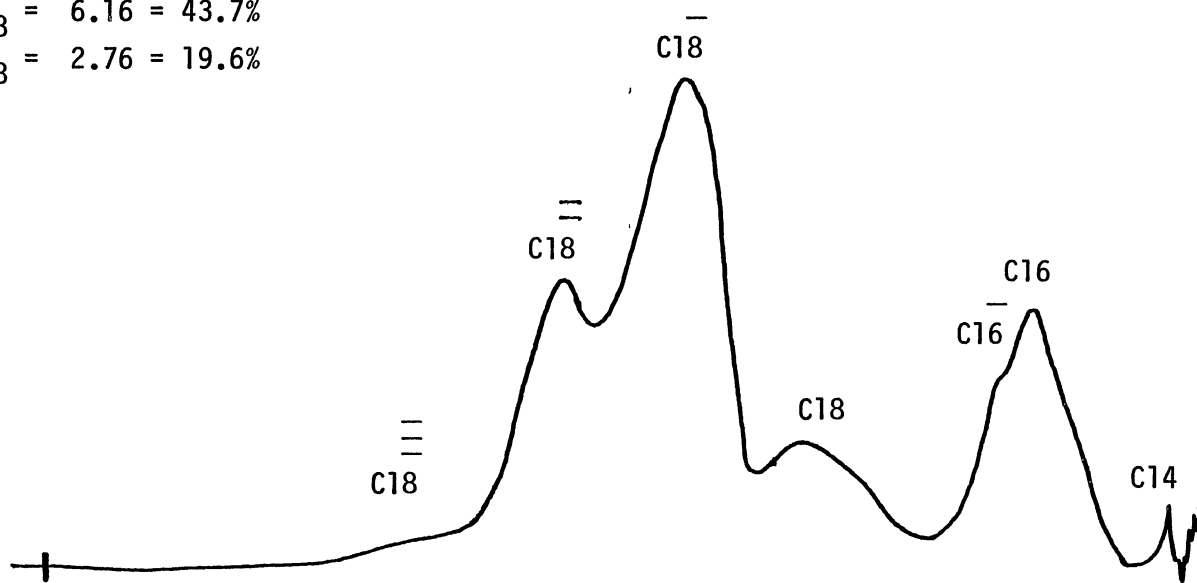


Figure 5. Chromatograph of Pecan Oil Fatty Acids

APPENDIX D
STATISTICAL DATA

TABLE V
SENSORY EVALUATION OF PECAN PIECES BY SIZE*

Pecan Size	Smell		Taste					Acceptability
	Aroma	Off-Odors	Texture	Sweetness	Full Nut Flavors	Off-Flavors	Unpleasant Aftertaste	Overall Acceptability
Large'	T	U	T	T	T	T	T	T
Medium	U	T	T	T	T	T	T	T
Small	T	U	U	T	T	T	T	T

*Mean values based on a scale of 0 through 50 where 50 is most acceptable and 0 is least acceptable.

T=Panelist ratings where treated samples had the higher mean score but not necessarily a significant difference.

U=Panelist ratings where untreated samples had the higher mean score but not necessarily a significant difference.

TABLE VI
 SENSORY EVALUATION OF PECAN PIECES BY TREATMENT*

Pecan Treatment	Smell		Taste				Acceptability	
	Aroma	Off-Odors	Texture	Sweetness	Full Nut Flavors	Off-Flavors	Unpleasant Aftertaste	Overall Acceptability
Treated	L	L	L	L	M	M	L	L
Untreated	M	L	L	L	L	L	L	L

*Mean values based on a scale of 0 through 50 where 50 is most acceptable and 0 is least acceptable.

L=Panelist ratings where large sized pecan pieces had the highest mean score but not necessarily a significant difference.

M=Panelist ratings where medium sized pecan pieces had the highest mean score but not necessarily a significant difference.

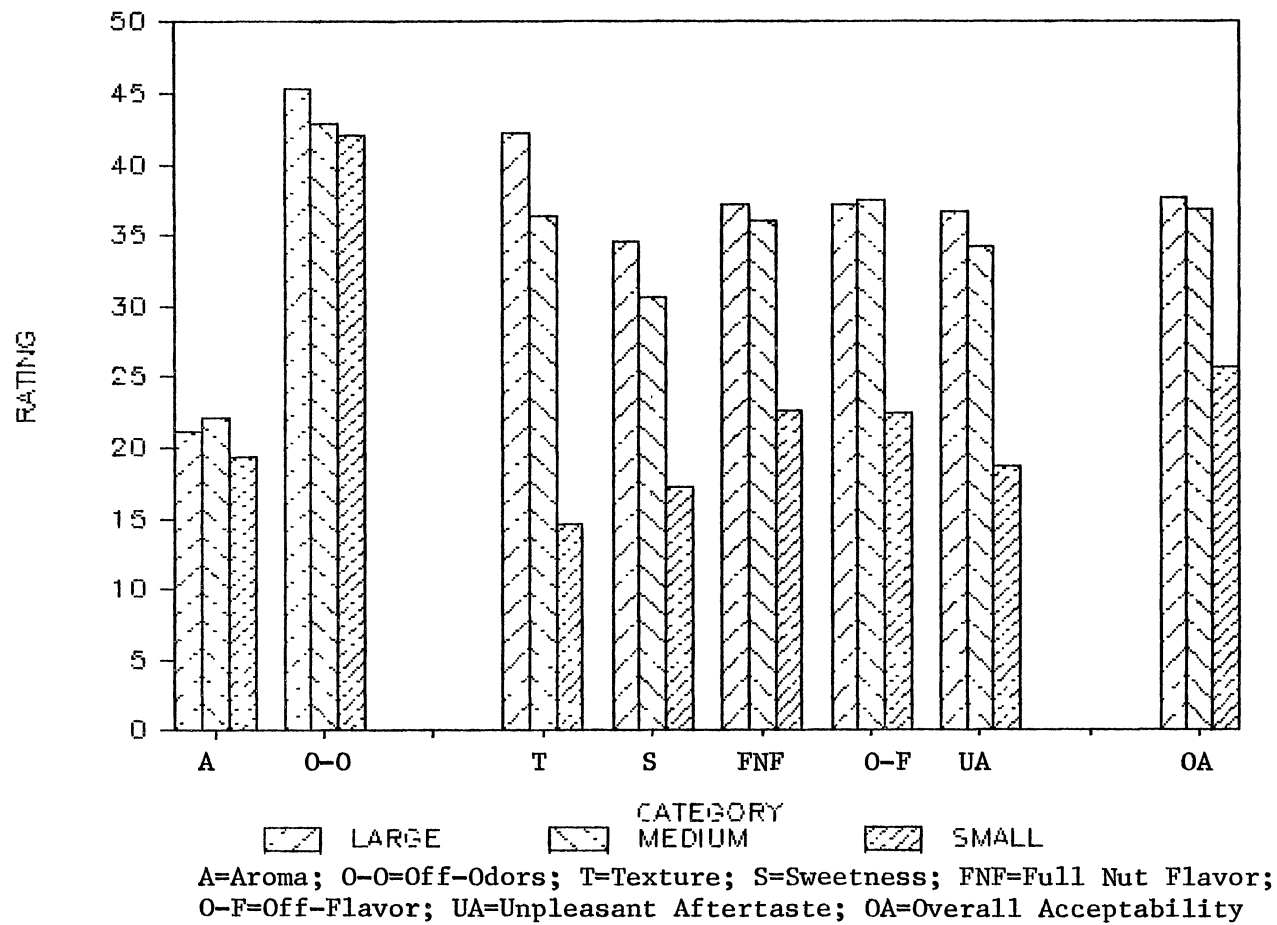


Figure 6. Evaluation of Pecan Pieces by Size

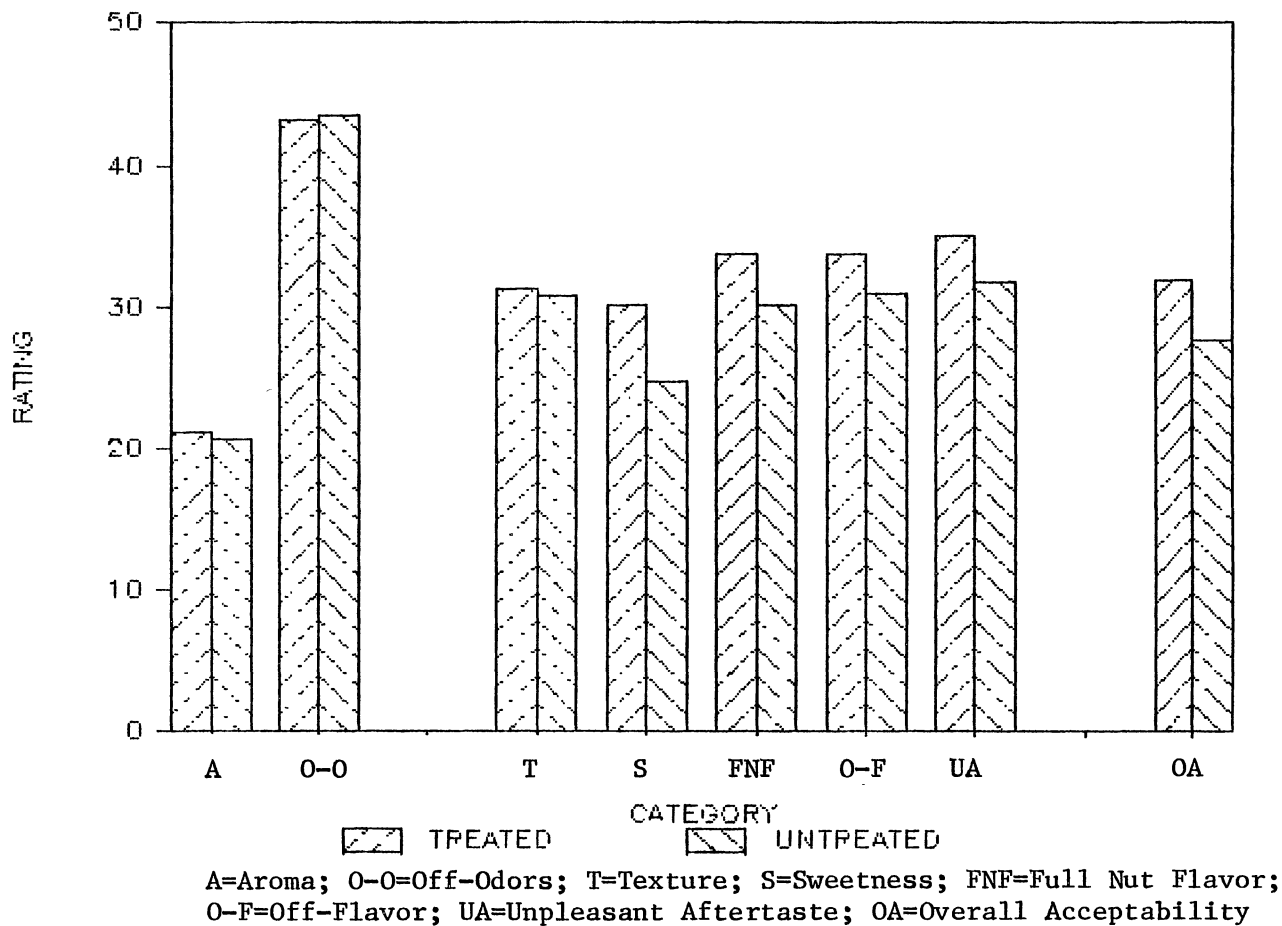


Figure 7. Evaluation of Pecan Pieces by Treatment

VITA ²

Marilyn Baugh Waters

Candidate for the Degree of

Master of Science

Thesis: PARTIAL OIL EXTRACTION OF OKLAHOMA PECANS

Major Field: Food, Nutrition and Institution Administration

Biographical:

Personal Data: Born in Bartlesville, Oklahoma, September 1, 1939, the daughter and first child of Walter and Nadine Baugh; one brother, Thomas Walter Baugh; three children: Jamie Lyn Waters, Jana Susanne Waters, and Robert Thomas Waters.

Education: Attended public schools in Bartlesville, Oklahoma, and Oklahoma City, Oklahoma; graduated from College High School, Bartlesville, Oklahoma, in May, 1957; received Bachelor of Science degree with a major in Home Economics Education and Community Services from Oklahoma State University in May, 1981; completed requirements for the Master of Science degree at Oklahoma State University in July, 1985.

Professional Experience: Graduate Teaching Assistant, Oklahoma State University, August, 1982 to May, 1984; Graduate Research Assistant, Oklahoma State University, May, 1984 to August, 1985; completion of qualifying experience for the American Dietetic Association, May, 1985.

Professional Organizations: American Dietetic Association, American Home Economics Association, Institute of Food Technologists.

Awards: Grant, Oklahoma Pecan Growers Association, 1984 and 1985.