

EFFECT OF LIQUID NITROGEN IN PACKAGING
AND FORMULATION CONCEPTS IN ACID FOODS.

By

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AND FORMULATION CONCEPTS IN ACID FOODS.

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*To my parents
Anju Babu & Sujatha*

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Title of Study: EFFECT OF LIQUID NITROGEN IN PACKAGING AND FORMULATION CONCEPTS IN ACID FOODS.

Major Field: FOOD SCIENCE

Abstract Chapter 1: The objective of the research was to eliminate paneling of the PET bottles due to hot filling. BBQ sauce was hot-filled at 54°C (130°F) in plastic PET, 567 g (20 oz) bottles. The PET bottles paneled, or were misshapen, due to the heat and pressure of hot-fill process. Liquid Nitrogen gas was used as a processing aid to optimize the headspace pressure to prevent paneling. The objective of the research was to eliminate paneling of the PET bottles due to hot-filling. A micro dosing system was used to inject nitrogen into the container just after filling and immediately prior to capping. Headspace pressure was measured using a custom-designed pressure sensor. Nitrogen dosage was plotted against headspace pressure. The relationship between nitrogen dosage and headspace pressure was linear, with a coefficient of determination of 0.84, a slope of, 4.24 kPa/ms, and an intercept of 12.45 kPa. Results were analyzed by using analysis of variance. Visual inspection of the bottles for defects resulted in the determination of the optimum headspace pressure of 30 kpa (4.4 psi).

Abstract Chapter 2: BBQ sauce formulated with sugar and high fructose corn syrup (HFCS) was evaluated for consumer preferences. The objective of this study was to substitute sugar with HFCS and perform sensory evaluation. Five sensory parameters were evaluated using a 9-point hedonic rating scale to perform sensory analysis and determine the consumer preferences. SAS software version 9.2 was used to identify the nuances. It was determined that BBQ sauce formulated with either HFCS or sugar did not possess a significant difference.

Abstract Chapter 3: BBQ sauce made with sugar and high fructose corn syrup (HFCS) was evaluated for color, over 13 weeks in ambient and oven temperatures (50°C). The objective of this work was to determine if there was a significant difference in color between formulations of BBQ sauce with sugar and HFCS initially and over time. Minolta colorimeter was used for the analysis. Results were statistically analyzed using analysis of variance. It was concluded that BBQ sauce made with HFCS or sugar did not have significant color difference initially or during the time period studied.

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CHAPTER I

INTRODUCTION

BBQ Sauce

Before the time of refrigeration, it was discovered that smoking, salting, or drying meat would preserve it longer. Which eventually lead to the culinary tradition of cooking meat low and slow over indirect flame with the very first BBQ sauce being made with pepper and vinegar which eventually got modified to today's flavorful BBQ. (Meat Head Goldwyn 2018).

American BBQ sauce history is diverse in origin which dates back to the path from the culture of Caribbean cooking brought into Spain and landed in the west by the early settlers which was further seasoned with European flavors to arrive at the current day BBQ sauce. The first origin of BBQ sauce is linked to Caribbean islands with the meaning of BBQ being 'Sacred Fire Pit, cooked over life fire'

The most common meat and food additive ‘BBQ sauce’ has not originated in America and the place it was first developed is not known (Aramouni et al. 2013). Records reveal that it was used in the USA in the late 1800’s that was first brought by the slaves and creoles. While there are several flavors of BBQ sauce depending on regional tradition, currently available in the market, the base formulation is with tomato ketchup or tomato paste with ingredients varying widely within individual countries.

Head Country BBQ INC

Head Country Inc’ located in Ponca City, Oklahoma was started in the year 1947 as a family owned business. The history has its beginnings in the World War II by Donovan “Bud Head” a cook on a Navy destroyer, who served his own recipe sauce to the men on the ship. After coming home from the Navy, Bud and his wife Freda produced the popular sauce from their ranch house and neighbors would line up with their fruit jars. By 1977, it became too much to handle, so Bud passed along his secret recipe to his nephew ‘Danny Head’. Bud never imagined his sauce would become a staple at backyard BBQ’s across 26 states and 18 foreign countries (personal communication Mr. Paul Schatte)

Head Country is now Oklahoma’s # 1 selling BBQ sauce and the Ponca City Plant produces an average of 6000 gallons of the sauce per day. This business is

now a \$12 Million industry and holds the reputation of providing the best quality ready to eat products with an excellent array of barbeque (BBQ) sauce, seasonings and marinades for over 60 years, focusing on retail, food service and casual and family dining events. This local business contributes significantly to the Oklahoma economy and strives to keep the jobs and dollars in the state.

According to Neilson recent survey Head Country BBQ sauce is #10 in the top 50 sauces marketed, even though they are in only 12% of the grocery stores, unlike most of the major brands labeled under worldwide companies. Head Country celebrates 70 years this year and has only 27 employees (Deana Craig 2017)

The company's major product line is BBQ sauce. Based on the demands of local and global customers, this product is now available in 6 flavors. This company has grown significantly from just a regional favorite to expanding to reach throughout the country and internationally. The global customers include customers in Sweden, Norway, France and Head Country is now focusing on targeting Asian markets. This traditional product has made its way not just on the food tables but in the heart of the customers due to the various factors including quality, shelf life and convenience of the product size.

From 3,500 to 9,000 gallons of sauce made every day, up to 30,000 glass units are filled each day. Due to the potential safety concerns for employees and consumers in using glass for BBQ sauce, Head Country has initiated the use of plastic bottles in replacement of glass. However, with the magnitude of advantages of plastic, the limitations of using plastic bottles includes shrinkage of bottles due to hot-filling & labeling of the bottles due to the lack of sturdiness of the PVP bottle.

High Fructose Corn Syrup:

High fructose corn syrup (HFCS) manufacturing process was first developed by (Marshall et al. 1957) and was first introduced into the food industry in the late 1960. Since then HFCS has been used an alternative sweetener to sucrose and a major ingredient of foods for human consumption. HFCS is a liquid source sweetener obtained from corn starch.

Production of HFCS involves wet milling of corn and shelled process after a series of chemicals and enzymes use. This chemical reaction hydrolyzes corn starch to corn syrup where shelled corn is cleaned for processing with sulfuric acid, the residues of this reaction are glucose and glucose isomerase. The end product is classified into two different grades HFCS 42 (42% fructose and 58% glucose) and HFCS 55 and HFCS-55 (55% fructose and 45% glucose) (Parker

et al. 2010a). These two distinguished HFCS that are available in the market are numbered of 42 and 55 based on the different sweetener concentrations, with 55 being more concentrated.

Prior to use of sucrose, honey was the only sweetener, (Bray et al. 2004) however, the introduction of high fructose corn syrup (HFCS) has subsided the use of sucrose in the current days. HFCS is a major sweetener and additive widely used in variety of processed foods, yogurts, breads (Parker et al. 2010a) and beverages with regular soft drinks being the major contributors of HFCS use (Guthrie and Morton 2000). In the US, HFCS is found in almost all foods containing caloric sweetener (Bray et al 2016). HFCS is increasing in the food manufacturing due to its low cost compared with sugar” (Kelishadi et al. 2014) and several advantages compared to sucrose which makes this sweetener more commonly used in the food industry. These benefits include its cost effective, no hassle import from other countries due its abundance in the US, prohibits the metal shaving adulteration which is a severe problem in the manufacturing industry with use of sugar. Furthermore, it has easy access to usability.

Head Country currently utilizes sucrose to produce BBQ sauce. The company is interested in researching the alternative use of sucrose to HFCS in the face of economic challenges. One of the goals of my research is intended to perform

sensory evaluation with varying composition of HFCS and sucrose and provide feedback to the management on the transformation to HFCS.

Objectives

The objective of this study was to determine optimum bottling conditions with the use of liquid nitrogen along with other objectives listed as below.

1. Economics and Product Quality:

- a). Determine if it is possible to substitute **HFCS** for sucrose in the BBQ sauce.
- b). Conduct sensory panels using an expert panel to detect differences and preferences for BBQ sauce with HFCS and sucrose formulations.
- c). Study the color changes of BBQ sauce with HFCS and sucrose.
- d). Conduct accelerated shelf life studies on BBQ sauce with HFCS and sucrose formulations.

e). Calculate the cost savings associated with the transformation from glass to plastic containers for BBQ sauce.

2. Optimum Bottling conditions:

a. Nitrogen: Determine the optimum pressure for 20 oz. (567 gm) & 40 oz. (1134 gm) plastic bottles.

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CHAPTER II

Optimization of Head Space Pressure Using Liquid Nitrogen in Hot-Packed BBQ Sauce

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Abstract: BBQ sauce was hot-filled at 54°C (130 °F) in plastic PET, 567 g (20 oz) bottles. The PET bottles paneled, due to the pressure of hot-fill process. Liquid Nitrogen gas was used as a processing aid to optimize the headspace pressure to prevent paneling. The objective of the research was to eliminate paneling of the PET bottles due to hot-filling. A micro dosing system was used to inject nitrogen into the container just after filling and immediately prior to capping.

Headspace pressure was measured using a custom-designed pressure sensor. Nitrogen dosage was plotted against headspace pressure. The relationship between nitrogen dosage and headspace pressure was linear, with a coefficient of determination of 0.84, and slope of 4.24 kPa/ms, and an intercept of 12.45 kPa. Results were analyzed by using analysis of variance. Visual inspection of the bottles for defects resulted in the determination of the optimum headspace pressure of 30 kpa (4.4 psi).

Keywords: BBQ sauce, headspace, pressure, paneling, PET bottle, optimize

1. INTRODUCTION

Plastic is steadily replacing glass in food manufacturing facilities to reduce costs and environmental impact and improve safety. Shipping costs are significantly reduced for plastic containers compared to glass because of the weight difference. Glass containers weigh about 7 times more than a similar plastic container (274 grams versus 39 grams). Both glass and plastic may be recycled, but glass requires more energy to produce and recycle (Hannon, 1973). Furthermore, glass is brittle compared to plastic and is prone to breakage during manufacturing operations like receiving, production and shipping. Broken glass is an adulterant in food products and a potential safety hazard to plant personnel and consumers.

Plastic bottles have enormous benefits for packaging foods, but these benefits have not been realized for most hot-packed items like BBQ sauce. The hot-packing process poses container deformation issues resulting from the relatively high filling temperatures (compared to cold pack) and the headspace vacuum. The high temperatures and forces on the container as a result of the vacuum combine to deform the bottle. Deformation of plastic bottles due to heat and pressure is known as “paneling” in the industry (see fig. 1). The aim of this study was to use liquid nitrogen gas as a processing aid to optimize the headspace pressure in PET bottles filled with BBQ sauce.

Liquid nitrogen is classified as a processing aid, is an inert gas and is safe for consumers.

The demand for processed food with a longer shelf life is continuously growing. New methods of packaging are in use and being developed to accommodate longer shelf life. Newer packing methods include aseptic & hot filling (Manfredi and Vignali 2015) and MAP (modified atmospheric packaging). MAP uses combinations of gases like nitrogen, oxygen and carbon dioxide, depending on the food products packaging material and configuration.

Nitrogen gas purging is a prominent anti oxidation technique that has been widely used for wine and beverages, along with various products like vegetables, fruits, meat and snacks. Nitrogen is also used in processing and packaging fresh cut and minimally processed vegetables (Koseki and Isobe 2005) fruits (Rodríguez-Hidalgo et al. 2010) almonds (Raisi et al. 2015) and extension of herbs (Hu et al 2004).

The role of bottling is significant in the BBQ sauce manufacturing process. It provides convenience for shipping and handling, however, due to the nature of the hot filling operation, paneling of the plastic

bottles is a common challenge. The result of paneling is an aesthetically unappealing bottle with a distorted shape that further results in wrinkled labels. Paneling is commonly observed in the plastic bottles that are hot filled and sealed prior to cooling (Berk 2018). There are two common solutions to paneling. The first is to use a rigid container. The rigid container must be strong enough to withstand the forces of the vacuum, which occurs from hot filling, without visibly yielding. Glass, steel and aluminum are examples of rigid containers. The second solution to paneling is to pressurize the container with an inert gas, like nitrogen. The pressurizing gas has the opposite effect of the vacuum, and can reduce or eliminate the vacuum, or pressurize the container. If the headspace pressure is too high, the bottle or its seal may burst, with the potential to spill the BBQ sauce into the environment, or on the consumer.



Fig. (1). Ordinary plastic bottle of BBQ sauce (left) and paneled (deformed) plastic bottle of BBQ sauce (right).

Oxygen present in the headspace can react with the product and alter the organoleptic properties like color. Liquid nitrogen dosing reduces the oxygen content in the headspace by displacing it. The reduced oxygen atmosphere in the bottle helps to enhance the quality and shelf life of the product (Ansari and Datta 2003).

While extensive research has been conducted on the use of nitrogen gas and MAP in food packaging, information on the use of liquid nitrogen gas filling for hot-packing of BBQ sauce has not been published. The objective of this research was to optimize the optimum dose of liquid nitrogen required to overcome paneling of the plastic container.

2. MATERIALS AND METHODS:

The experiment was conducted at a BBQ sauce production plant in Oklahoma. The diagram shown in figure 2 describes the process flow of the experiment.

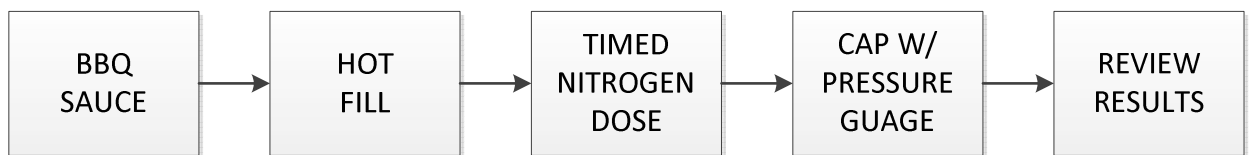


Fig. (2). Process flow of experimental setup.

Freshly BBQ sauce from the production line at Head Country, Inc., Ponca City, Oklahoma was provided for the experiment. The BBQ sauce was filled into plastic PET bottles (V Pet, Garland, TX). Fill mass was 567 grams (20 oz). Fill temperature was 54°C (130 F) +/- 2 °C and was monitored using a

calibrated thermometer. Immediately after filling, a dosage of liquid nitrogen was deposited into the head space of the bottle using the automated Nitrodose System (model MD 157, Vacuum Barrier Corporation, Woburn MA). The automated nitrogen dosage system had the capability to control the timing of the liquid nitrogen dosage application to 25, 26, 28, 30, 32, 34 and 36 ms. An image of a container of BBQ sauce receiving a dose of nitrogen is shown in figure 2. After the dosage, the bottles pass through an induction sealer where the eddy current seals the bottles.



Fig. (3). Nitrogen gas dosed into a container of BBQ sauce just after hot filling.

The head space pressure of the bottles was measured using a touch type gauge (model 1706210252, SSI technologies, Janesville, WI). The

gauge was mounted on a screw type metal lid (see figure 4 and 5) which was directly placed by hand on the BBQ bottle after the dose of liquid nitrogen was applied. Pressure readings from the dosage times starting from 25 ms to 36 ms were recorded and analyzed to identify the optimum head space pressure. A minimum of five replicates per dosage were recorded and analyzed as shown in table 1.

Observation was used to determine the optimal headspace pressure for the filled BBQ sauce bottles. The key factors observed were:

1. Deformed or paneled containers
2. Label application and appearance of the applied label.
3. Shape of the seal on the finished product after the product cooled to room temperature.



Fig. (4). Pressure gauge integrated with container cap to measure headspace pressure of a bottle.



Fig. (5). Detail of pressure gauge sensor attachment to a screw-on container cap.

Table (1). Headspace pressure (kPa), measured at given liquid nitrogen dose times (ms) for BBQ sauce filled into PET bottles at 54□.

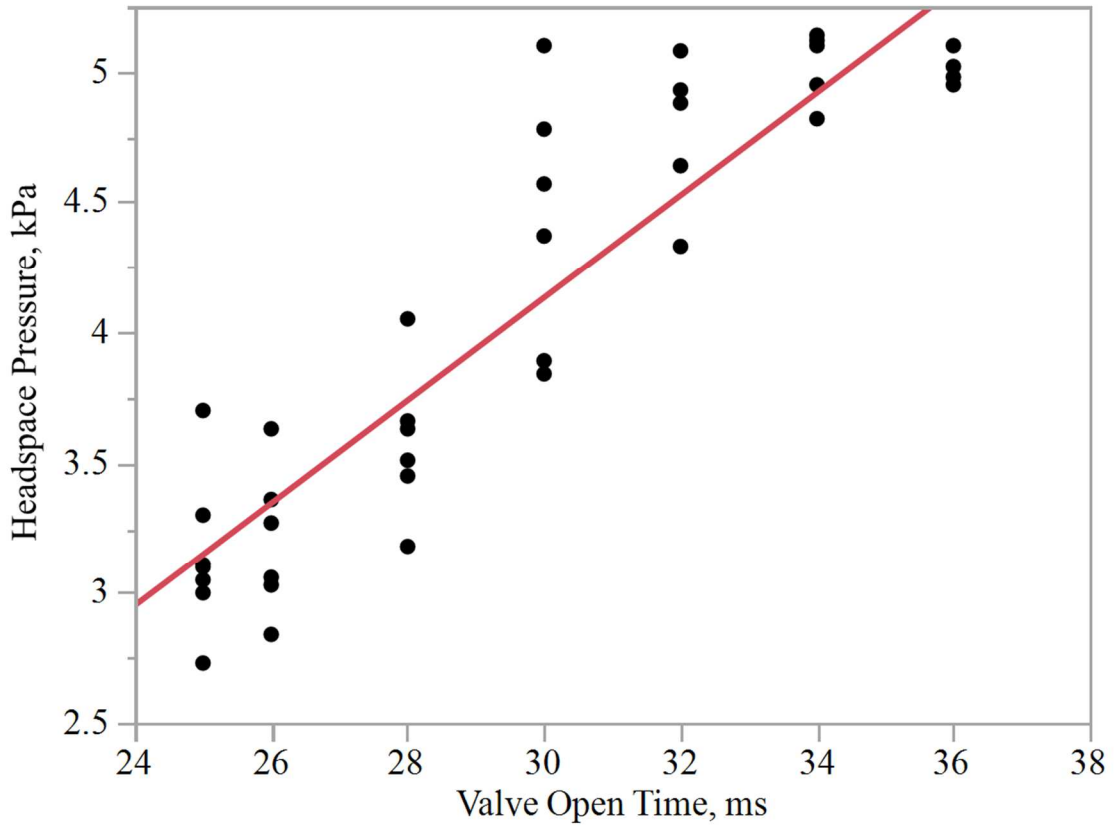
Replicates	Valve Open Time (ms)						
	25	26	28	30	32	34	36
1	3.3	3.03	3.63	5.1	4.33	5.1	5.02
2	3.1	2.84	3.66	3.89	4.93	5.14	4.95
3	3.0	3.36	3.18	4.37	4.88	5.12	5.1
4	3.7	3.63	3.45	4.78	4.64	4.82	4.98
5	3.11	3.27	4.05	3.84	5.08	4.95	5.1
6	2.73	3.06	3.51	4.57	4.64	-	4.98
7	3.05	-	-	-	-	-	-
Average	3.14	3.20	3.58	4.43	4.75	5.03	5.02
Std. Dev.	0.30	0.28	0.29	0.50	0.27	0.14	0.06



Fig. (6). Convex seal due to the bottle receiving excessive dosage of the liquid nitrogen

3. RESULTS

A scatter plot and best-fit line for the data provided in table 1 were made using JMP 14 (SAS institute, Cary NC) and are shown in figure 7. The line of best fit had a coefficient of determination (r^2) of 0.84, with a y-intercept of 12.45 kPa, and a slope of 4.24 kPa/ms. The slope indicated that the container headspace pressure increased 4.24 kPa for each ms that the nitrogen dispensing valve was open (within the range of 25 to 36 ms).



Coefficient of determination, $r^2 = 0.84$

Correlation coefficient, $r = 0.91$

Significance: < 0.0001

Line of best fit:

Slope = 0.197 (kPa/ms)

Intercept = -1.77

Fig. (7). Scatter plot with dosage time of liquid nitrogen (ms) versus head space pressure (kpa).

The use of liquid nitrogen helped in the conversion of glass bottles for filling of BBQ sauce to plastic bottles, this in turn resulted in a tremendous cost savings (table 2). There was a total of 3,205,137 bottles of BBQ sauce produced in 2017 which saved a net cost of \$ 1,171,658

Table (2). Costs Savings in 2017 by converting from Glass to Plastic

SAVINGS	567 gm Bottles	1134 gm Bottles
Materials (glass to plastic)	\$556942	\$ 415328
Freight	112,741	86,647
Total	\$669,683	\$501,975
Per bottle packed	\$0.3267	\$0.4345

4. DISCUSSION

A linear relationship was demonstrated between the time that the nitrogen valve was open and the resulting headspace pressure of hot-filled, sealed, PET BBQ sauce containers. PET BBQ sauce bottles which did not receive a dosage of liquid nitrogen, or had a headspace pressure less than 30 kPa, appeared deflated which was determined by inspecting the bottles on the line and applied labels were unacceptably deformed. Analysis of the data and bottles revealed that the optimum range for headspace pressure was 30 kPa

(4.35 psi). The bottles with a headspace pressure higher than 32 kPa (4.64 psi) resulted in a convex foil seal (Fig 6) and an undesirable spray of sauce upon opening the foil seal of the bottle.

5. CONCLUSION

The optimum pressure for bottle headspace was determined to be 30 kPa. While various pressures ranging from 25 kPa to 36 kPa were investigated, the lower pressure deflated the bottle while the higher pressures lead to an uneven seal on the bottle. This novel approach of pressurizing the headspace with a liquid nitrogen dose made it possible to convert from glass to plastic bottles for hot-filling BBQ sauce. The increased headspace pressure eliminated the deformation challenges associated with hot-filling PET bottles. Furthermore, successful conversion to PET bottles eliminated safety concerns of glass in the production facility and reduced costs.

CONFLICT OF INTEREST

The authors confirm that this article content has no conflict of interest.

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CHAPTER III

Color Analysis of BBQ Sauce with Sugar and HFCS During Storage up to 13 Weeks

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Abstract

BBQ sauce made with sugar and High Fructose Corn Syrup (HFCS) was evaluated for color, over 13 weeks in ambient and oven temperatures (50°C). The objective of this work was to determine if there was a significant difference in color between formulations of BBQ sauce with sugar and HFCS initially and over time. Minolta colorimeter was used for the analysis. Results were statistically analyzed using analysis of variance. It was concluded that BBQ sauce made with HFCS and Sugar did not have significant color difference initially or during the time period studied.

Keywords:

BBQ sauce, Color analysis, HFCS and Sugar.

1. Introduction:

The origin of BBQ sauce dates back to the formation of the first American colonies in the 17th century. BBQ sauce is a ubiquitous flavoring agent used in many foods. Depending on the region, there are several varieties of BBQ sauce available. In the face of financial challenges companies are considering alternative options of use of sweetener for BBQ sauce. HFCS is a top alternative to sugar because of domestic availability, stable price, and low cost. However, color is an important factor for consumers in choosing a brand of the BBQ sauce. The objective of this research was to determine the influence of HFCS on the color of BBQ sauce compared to sugar for a period of 13 weeks at ambient temperature and 50°C.

Experimental Design:

In a post test design with control (standard BBQ sauce) and treatment (standard BBQ sauce with HFCS substituted for sugar). Color of the control and treatment was measured over time using Minolta colorimeter CM-3500d (Konica Minolta, Sensing Americas, NJ 07446) on days 0, 21, 77, and 91. Control and treatment samples were stored at room temperature and 50°C. Three bottles of each (control and treatment) was stored at both temperatures. Color measurements at each bottle was taken in triplicate. See figure 1 for diagram of experiment design. Color was measured through the base of the container by placing the bottle over the aperture of the colorimeter, see figure 2. The containers were not opened during the course of the study.

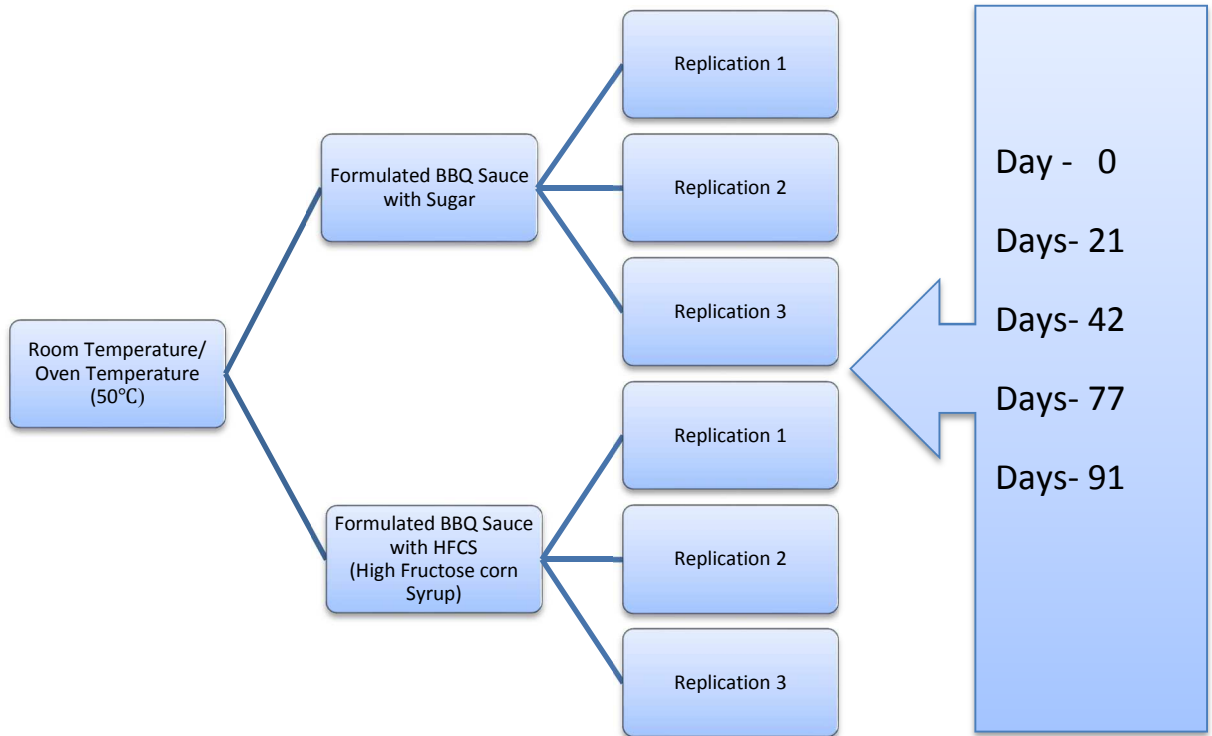


Fig. (1). Experimental design for color analysis of BBQ sauce



Fig. (2). BBQ sauce color measurement with a colorimeter.

2. Materials & Methods:

2.1 BBQ Sauce:

The BBQ sauce samples were formulated per the local company recipe at the site which is proprietary. Cane sugar (Blackhive, AR) and HFCS strength 55 (Sweetener Supply Corporation, IL) was used in the production of the BBQ sauce filled in the 567 ml plastic bottles. Fill temperature was 54°C (130 F) +/- 2 °C and was monitored using a calibrated thermometer, immediately after filling (see materials and methods section of chapter 2). Two sets of similar formulations of 3 replicates were tested.

2.2 Liquid Nitrogen Dosage:

Immediately after filling, a dosage of liquid nitrogen was deposited into the head space of the bottle using the automated Nitrodose System (model MD 157, Vacuum Barrier Corporation, Woburn, MA). The automated nitrogen dosage system had the capability to control the timing of the liquid nitrogen dosage application of 30 ms. The purpose of the nitrogen dose was to provide a final headspace pressure of 30 kPa. After the dosage, the bottles passed through an induction sealer, then a tamper evident shrink band applied.

2.3 BBQ Sauce Storage: BBQ sauce replicates being tested for ambient temperature were stored at compacted boxes at a research lab and another set for testing the accelerated shelf life testing were stored in a dark oven at 50°C.

2.4 Color Measurement:

During storage bottles were removed and color was measured using a Minolta colorimeter (CM-3500d, Konica Minolta, Sensing Americas, NJ 07446). Color was measured through the bottom surface of the BBQ sauce bottle after placing it on the instrument. Measuring aperture size of 33 mm was used for the determination of the color ranges. (L=30.25, a=5.95 and b=0.04). Color was reported using the following three CIE 1976 (International Commission on Illumination) (Konica Minolta Sensing Americas, Inc manual.) L*a*b* coordinates.

- L* [(degree of lightness)]
- a* [red (+) to green (-)]
- b* [yellow (+) to blue (-)]

Table (1). BBQ samples color readings from Minolta Colorimeter

Time Period	Sample	L*	a*	b*
Week 0	Sugar RT	30.60	6.77	2.73
	HFCS RT	30.84	7.47	3.53
	Sugar OT	30.82	6.72	2.86
	HFCS OT	30.34	7.58	4.02
Week 3	Sugar RT	30.88	6	2.42
	HFCS RT	30.23	6.96	3.39
	Sugar OT	30.17	5.13	1.80
	HFCS OT	30.89	5.76	3.00
Week 6	Sugar RT	30.33	7.76	2.51
	HFCS RT	30.09	7.46	2.79
	Sugar OT	30.81	3.82	1.38
	HFCS OT	31.38	4.37	2.16
Week 11	Sugar RT	29.82	5.78	2.6
	HFCS RT	29.96	6.50	3.53
	Sugar OT	31.46	2.51	0.96
	HFCS OT	31.87	3.06	1.31
Week 13	Sugar RT	29.98	5.65	2.28
	HFCS RT	30.38	6.12	2.49
	Sugar OT	31.85	2.53	1.46
	HFCS OT	29.96	3.31	1.55

OT- Oven Temperature, RT- Room Temperature, L*, a*, b* are color co-ordinates.

Based on the Commission Internationale de l'Eclairage (CIE) Color Systems utilize CIE L*a*b* coordinates to determine color. ΔE value is used to determine the variation of color in two experimental samples.

General guidelines for color difference determination (Color Yard Stick) are shown in table 2. (Chung, 2003) (Beckman et al 1998) (Ocean Optics, Mowatt, Applications Scientist, 2015) (Franziska Bührle et al 2017), (Jasia Nissar et al 2017) & (Seunga Kang Ha, 2004).

Table (2). General guidelines for color difference determination (Color Yard Stick)

ΔE^*	Human Eye Perception
≤ 2.0	Not Detectable with unaided Eyes
2-3	Detectable by very close observation of trained experts.
4-10	Detectable at a glance
11-49	Colors are similar
100	Colors are exact opposite

2.5 Data Analysis:

Data obtained from the study was analyzed using the following formula:

- Total color difference formula Delta E was used as referenced in Konica Minolta Sensing Americas, Inc manual.

$\Delta E^* = [\Delta L^{*2} + \Delta a^{*2} + \Delta b^{*2}]^{1/2}$ by using L*, a*, b* color coordinates.

ΔL^* (L* HFCS- L* Sugar) = difference in lightness (+) or darkness (-).

Δa^* (a* HFCS- a* Sugar) = difference in redder (+) or greener (-).

Δb^* (b* HFCS- b* Sugar) = difference in yellow (+) or blue (-).

ΔE^* = total color difference

3. Results and Discussion:

Total color difference between BBQ sauce formulated with sugar and HFCS at room temperature during different time intervals is shown in Table 2 and further demonstrated as histogram and scatter plot in Figures 2 and 3.

Table (3). Total color difference between samples formulated with HFCS and sugar at room temperature using ΔE^* , calculated from $L^* a^* b^*$ color values.

Time Point	Sugar			HFCS			HFCS-Sugar			Total Color Difference
	L^*	a^*	b^*	L^*	a^*	b^*	ΔL^*	Δa^*	Δb^*	ΔE^*
Week 0	30.60	6.77	2.73	30.84	7.47	2.86	0.24	0.70	0.13	0.75
Week 3	30.88	6.00	2.42	30.23	6.96	1.80	-0.65	0.96	-0.62	1.31
Week 6	30.33	6.09	2.51	30.09	7.46	2.79	-0.24	1.37	0.28	1.42
Week 11	29.82	5.78	2.66	29.96	6.50	0.96	0.14	0.72	-1.64	1.8
Week 13	29.98	5.65	2.28	30.38	6.12	1.46	0.40	0.47	-0.82	1.03

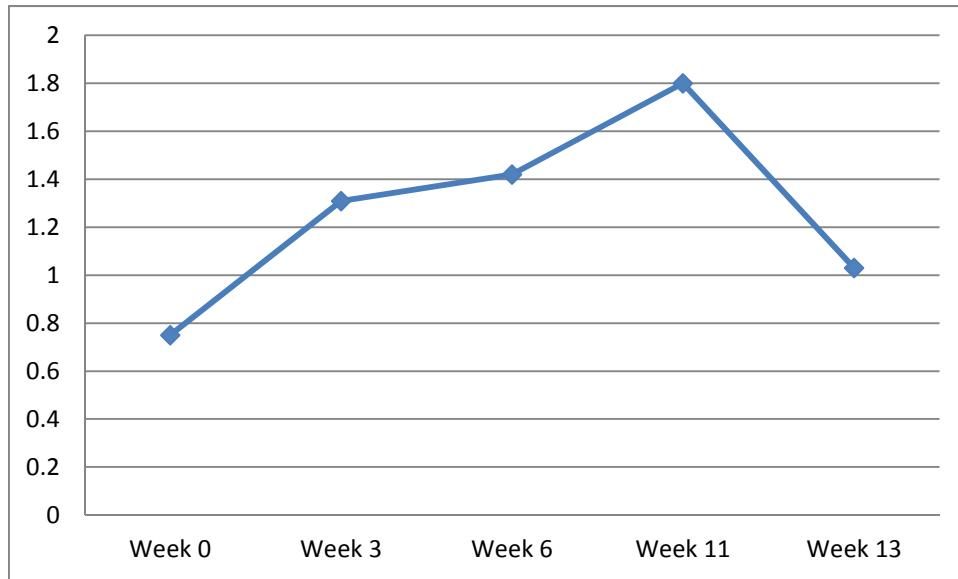


Fig. (3). Scatter plot showing total color difference between BBQ sauce formulated with HFCS and sugar at room temperature over a period of 13 weeks' time points using ΔE^ , calculated from $L^* a^* b^*$ coordinates values.*

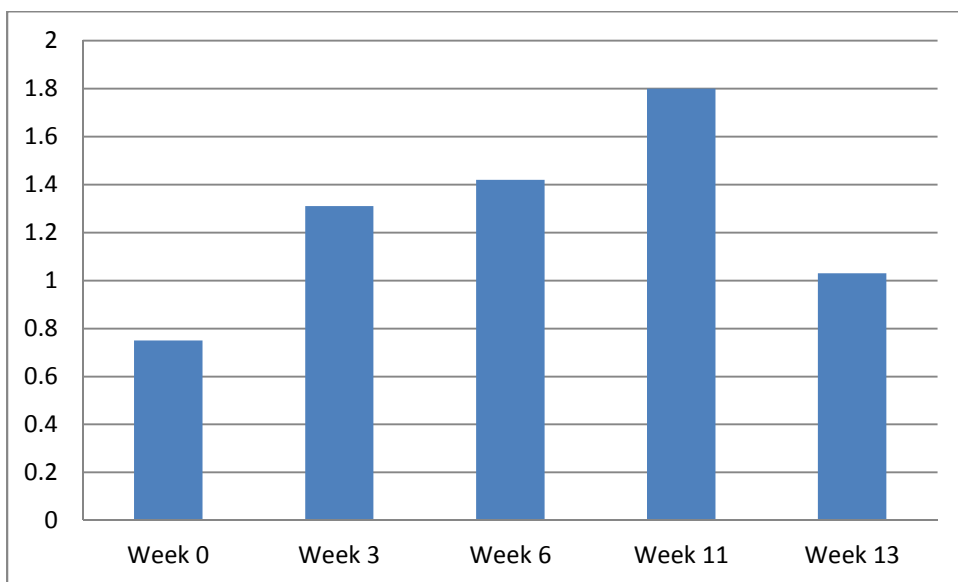


Fig. (4). Histogram showing ΔE^ Total color difference between BBQ sauce formulated with HFCS and sugar at room temperature over a period of 13 weeks' time points using $L^* a^* b^*$ coordinates values.*

Total color difference between BBQ sauce formulated with Sugar and HFCS stored at oven temperature (50°C) during different time points is shown in Table 3. The L*, A*, B* values for the two formulations were collected during Week 0, Week 3, Week 6, Week 11 and Week 13. The color difference between the two samples were calculated using the formula ΔE^* . These results are shown in Table 3 and further demonstrated as histogram and scatter plot in figures 4 and 5.

Table (4). Total color difference between samples formulated with HFCS and Sugar at Oven temperature using ΔE^ , calculated from L* a* b* coordinates values.*

Time Point	Sugar			HFCS			HFCS-Sugar			Total Color Difference
	L*	a*	b*	L*	a*	b*	ΔL^*	Δa^*	Δb^*	
Week 0	6.72	6.72	2.86	7.58	7.58	4.02	0.86	0.86	1.16	1.68
Week 3	5.13	5.13	1.80	5.76	5.76	3.00	0.63	0.63	1.20	1.49
Week 6	30.81	3.82	1.38	30.71	4.37	2.16	-0.10	0.55	0.78	0.96
Week 11	2.51	2.51	0.96	3.06	3.06	1.31	0.55	0.55	0.35	0.85
Week 13	2.53	2.53	1.46	3.31	3.31	1.55	0.78	0.78	0.09	1.11

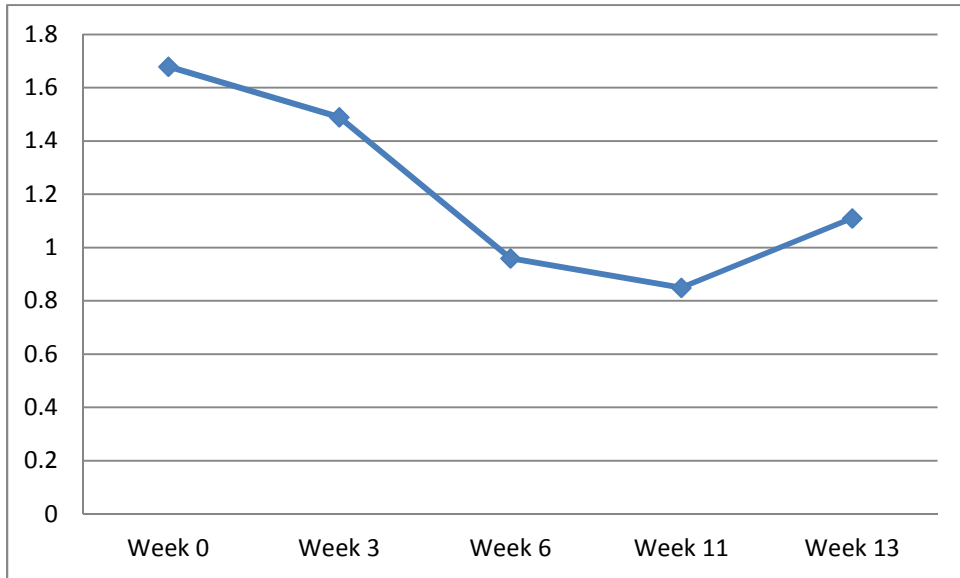


Fig. (5). Scatter Plot showing ΔE^ total color difference between BBQ sauce formulated with HFCS and sugar at oven temperature over a period of 13 weeks' time points using $L^* a^* b^*$ coordinates values.*

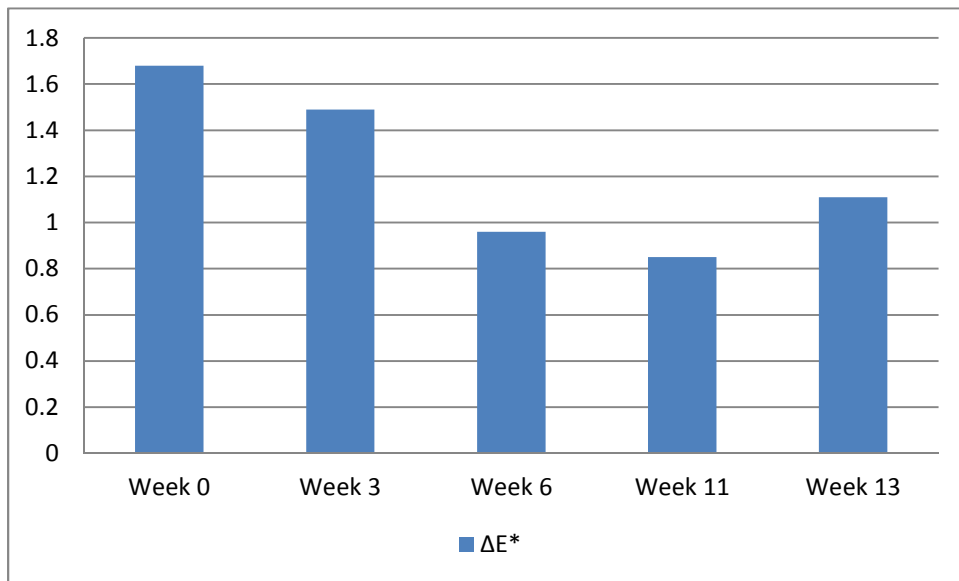


Fig. (6). Histogram showing ΔE^ total color difference between BBQ Sauce formulated with HFCS and sugar at oven temperature over a period of 13 weeks' time points using ΔE^* , calculated from $L^* a^* b^*$ coordinates values.*

Conclusion:

Based on the results derived by using the total color difference formula ΔE^* , BBQ sauce formulated with HFCS did not show a significant color variation compared to the BBQ sauce formulated with sugar stored at room temperature and oven temperatures over 13 weeks (91 days). The result of color difference represented by ΔE^* was less than 2, which cannot be perceived by the human eye. BBQ sauce is a viscous liquid that has a naturally saturated color. In this type of product, the color variation that may be perceived by the human eye,

ΔE^* must be at least 4. Results, indicate there was no significant color difference between samples of BBQ sauce formulated with HFCS or sugar and stored at room temperature or in an oven at 50°C over 91 days.

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CHAPTER IV

Sensory Evaluation of BBQ Sauce Formulated with Sugar and HFCS

Sensory Evaluation of BBQ Sauce Formulated with Sugar and HFCS

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Abstract:

BBQ sauce formulated with sugar and High Fructose Corn Syrup (HFCS) was evaluated for consumer preferences. The objective of this study was to substitute sugar with HFCS and perform sensory evaluation. Five sensory parameters were evaluated using a 9-point hedonic rating scale to perform sensory analysis and determine the consumer preferences. SAS software was

used to identify the nuances. It was determined that BBQ sauce formulated with either HFCS or sugar did not possess a significant difference.

Introduction:

Sensory analysis:

Use of sensory evaluation has steadily increased with the growth of processed foods (Lawless and Heymann 2010). The methods used to score the primary response towards a food product for example human perception of a commodity drives the brand image and price (Svensson 2012). Sensory evaluation is also critical in determining the quality and product development of new launch (Ludovic et al, 2007). While several sensory evaluation methodologies are in use, in this research, discrimination analysis and descriptive analysis are used on a Hedonix 9 scale.

Sweeteners like honey, molasses, syrups and sugars have enhanced the flavors of food for thousands of years with Honey being the principal sweetener prior to the introduction of the fructose (White 1999). High Fructose Corn Syrup (HFCS) is liquid alternative sweetener to sucrose (Sjöström 1957) which is steadily replacing the use of sucrose in the food and beverage industry due to its low cost (Kelishadi et al. 2014). The use of HFCS and health status have been controversial while studies supported the

concept of fructose increases chronic disorders however, other studies have not confirmed the relationship between fructose use with the potential for the diseases. (Moreno and Hong 2013 (Richard A. Forshee, Maureen L. Storey et al 2007).

HFCS have enormous benefits in the food industry like low cost and abundance of availability without the need for importing from foreign countries

BBQ sauce has been the staple food for centuries, while several flavors and formulations exist as regional favorites. Food industries are considering transition from using sugar to HFCS in the formulation due to the economics and availability of HFCS. This study was conducted to assess the consumer preference of using sugar versus HFCS in the BBQ sauce.

Materials & Methods:

2.1 BBQ Sauce:

The BBQ sauce samples were formulated per the local company recipe at the site which is proprietary. Cane sugar and HFCS strength 52 were used in the production of the BBQ sauce filled in the 567 gm plastic bottles. Sauce samples that were formulated using Sugar were

recorded as (NN1) and High Fructose Corn Syrup as (NN9) during the time of sensory evaluation as a blind fold measure. These samples in plastic bottles were stored at room temperature until sensory evaluation was concluded

2.2 Liquid Nitrogen Dosage:

Immediately after filling, a dosage of liquid nitrogen was deposited into the head space of the bottle using the automated Nitrodose System (model MD 157, Vacuum Barrier Corporation, Woburn MA). The automated nitrogen dosage system had the capability to control the timing of the liquid nitrogen dosage application of 30 ms. After the dosage, the bottles pass through an induction sealer where the eddy current seals the bottles with a shrink wrap.

2.3 BBQ Sauce Storage and Sensory Evaluation:

BBQ sauce replicates being evaluated were stored at ambient temperature and the sensory evaluation was performed in lab kitchen. An expert taste panel was used to detect the difference and preferences for BBQ sauce. The panel was requested to rate samples for the following organoleptic properties: sweetness, sour, mouthfeel, color, palatability acceptability. For a total number of 43 panelists coded BBQ samples were presented to assess the sauce (one at a time) to rate

from 1 = “dislike extremely” to 9 = “like extremely” on a 9-point hedonic scale (Peryam, D.R., & Pilgrim, F.J. 1957) (Lawless and Heymann, 2010). The data collected was transferred to Microsoft excel sheet as shown in Table 1.

Data Analysis:

The collected data sensory data based on descriptive analysis by candidates was analyzed using SAS software (SAS Institute, 2004). Data was analyzed using Analysis of Variance (ANOVA) procedure Tukey’s Test as these samples are five dependent variables (the sweet, sour, mouthfeel, color, palatability) were analyzed between two treatments (Sugar, HFCS). Results were deemed statistically significant if $p < 0.05$ (Sabato 2005). Proc Means was used to get summary statistics as shown in Table 2.

Table (1). Sensory Panel Descriptive Analysis data (questionnaire).

Panelist	Sweet	Sour	Mouthfeel	Color	Palatability	Treatment
1	9	7	8	5	8	HFCS
2	9	9	8	7	8	HFCS
3	9	7	8	9	8	HFCS
4	7	7	8	7	8	HFCS
5	8	8	8	6	8	HFCS
6	8	8	4	7	7	HFCS
7	7	6	8	6	8	HFCS
8	5	7	7	7	8	HFCS
9	7	9	6	9	9	HFCS
10	9	8	9	7	7	HFCS
11	7	6	6	8	8	HFCS
12	8	7	7	9	9	HFCS
13	8	5	8	8	8	HFCS
14	8	7	8	9	8	HFCS
15	8	8	9	5	7	HFCS
16	8	8	9	9	7	HFCS
17	6	5	9	5	7	HFCS
18	9	5	8	8	9	HFCS
19	8	5	8	9	8	HFCS
20	9	9	9	9	9	HFCS
21	8	2	6	5	8	HFCS
22	3	2	3	8	3	HFCS
23	5	6	7	9	9	HFCS
24	9	8	9	8	9	HFCS
25	8	8	8	8	7	HFCS
26	6	6	9	9	8	HFCS
27	9	9	9	9	9	HFCS
28	9	8	8	7	7	HFCS
29	8	8	9	9	8	HFCS
30	5	7	8	8	7	HFCS
31	9	8	8	8	8	HFCS
32	4	4	5	7	4	HFCS
33	1	3	6	5	1	HFCS
34	7	8	8	8	8	HFCS

35	7	6	7	9	7	HFCS
36	6	3	5	8	7	HFCS
37	9	6	8	8	7	HFCS
38	7	4	3	6	4	HFCS
39	7	7	8	8	7	HFCS
40	4	1	6	2	3	HFCS
41	9	9	7	7	8	HFCS
42	6	6	5	8	7	HFCS
43	8	8	8	8	8	HFCS
1	8	7	8	5	8	Sugar
2	6	6	6	5	7	Sugar
3	8	7	8	9	8	Sugar
4	8	5	4	7	7	Sugar
5	4	4	8	5	4	Sugar
6	7	6	8	8	7	Sugar
7	8	7	7	6	8	Sugar
8	5	6	7	7	7	Sugar
9	9	6	9	8	8	Sugar
10	8	9	9	6	8	Sugar
11	7	6	7	8	8	Sugar
12	8	5	7	9	9	Sugar
13	8	5	8	8	8	Sugar
14	7	7	8	8	8	Sugar
15	6	3	4	7	4	Sugar
16	9	9	9	9	9	Sugar
17	4	4	4	5	5	Sugar
18	9	5	8	8	9	Sugar
19	8	5	8	9	8	Sugar
20	9	8	8	9	8	Sugar
21	7	2	4	5	7	Sugar
22	8	6	7	8	7	Sugar
23	7	5	8	9	9	Sugar
24	8	8	8	8	8	Sugar
25	7	7	7	6	6	Sugar
26	6	8	8	8	8	Sugar
27	9	9	9	9	9	Sugar
28	7	7	7	8	8	Sugar
29	9	9	9	9	9	Sugar

30	7	7	8	8	7	Sugar
31	7	8	7	8	8	Sugar
32	8	8	5	8	8	Sugar
33	9	5	8	9	9	Sugar
34	8	9	9	8	9	Sugar
35	9	9	9	9	9	Sugar
36	6	5	5	9	7	Sugar
37	7	8	8	8	8	Sugar
38	4	7	4	8	5	Sugar
39	8	8	7	8	8	Sugar
40	8	9	8	6	9	Sugar
41	9	9	8	8	8	Sugar
42	8	9	8	8	8	Sugar
43	8	8	8	8	8	Sugar

Table (2). Sensory attributes of Barbeque Sauce made with Sugar and HFCS.

Attributes	BBQ Sauce Treatments		
	Sugar Sample	HFCS Sample	P-Value
Sweet			0.5524
N	43	43	
Mean (SD)	7.44 (1.3)	7.23 (1.8)	
Sour			0.5071
N	43	43	
Mean (SD)	6.74 (1.8)	6.46 (2.0)	
Color			0.5464
N	43	43	
Mean (SD)	7.65 (1.2)	7.46 (1.5)	
Mouthfeel			0.9451
N	43	43	
Mean (SD)	7.30 (1.5)	7.32 (1.5)	
Palatability			0.2336
N	43	43	
Mean (SD)	7.67 (1.2)	7.27 (1.7)	

Note: SD – refers to standard deviation

Comparison of Sensory attributes (sweet, sour, mouthfeel, color, palatability) of two samples Sugar and High Fructose Corn Syrup. Values shown are the average of three replicates.

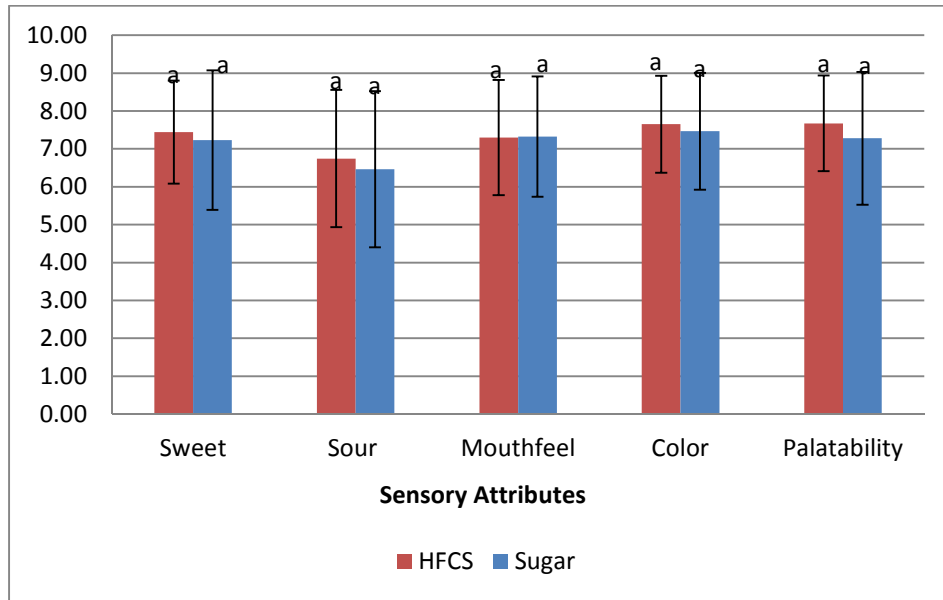


Fig. (1). Histogram comparison of sensory attributes (sweet, sour, mouthfeel, color, palatability) of two samples sugar and HFCS. Values shown are the average of three replicates. Error bars represent standard deviation. Values marked with the same letter are not significantly different (p -value < 0.05 shows significance).

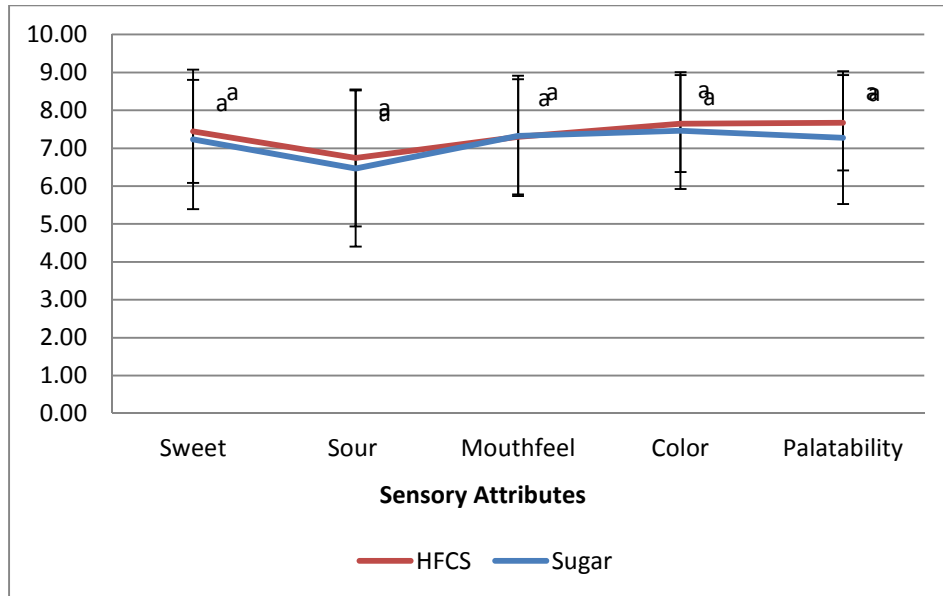


Fig. (2). Scatter plot comparison of sensory attributes (sweet, sour, mouthfeel, color, palatability) of two samples sugar and HFCS. Values shown are the average of three replicates. Error bars represent standard deviation. Values marked with the same letter are not significantly different (p -value <0.05 shows significance).

Results and Discussion:

The results collected from 43 participants, based on descriptive analysis questionnaire were used to determine the nuances of sensory attributes of Sugar and HFCS samples based on p value. If p value is less than 0.05 then it indicates the significance of HFCS formulation over sugar. Questionnaire results as shown in Table1, were used for further sensory analysis. These results indicated there was no significance of one sample over another based

on P-value. The attributes tested were for Sweet, Sour, Mouthfeel, Color, Palatability. The mean of the questionnaire results is presented in Table 2. The results of the analysis presented in Figures 1 & 2.

Conclusion:

It can be concluded from the 9-point hedonic rating scale for taste panel results there is no significant difference ($p < 0.05$) in Sweet, Sour, Color, Mouthfeel and Palatability. This suggests that sensory attributes of Barbeque sauce made with HFCS sample remains similar (with no significant difference) to Barbeque sauce formulated with sugar.

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CHAPTER V

CONCLUSIONS

This research study provides some insights on the Economics, Product Quality and Optimum Bottling Conditions in the production of processed foods especially low acid foods like BBQ sauce.

The major conclusions drawn from this research study are as follows:

- a. It is possible to substitute HFCS for sucrose in the BBQ sauce formulation. Also, based on the sensory and the color evaluation results the formulations did not show a significant difference one over the other.
- b. Sensory panel could not detect the nuances of sauce formulated with HFCS or sucrose.
- c. Color changes of BBQ sauce was studied for a period of 13 weeks there was no significant difference in the color for either of the formulations.
- d. Accelerated shelf life studies of BBQ sauce at 50°C in oven temperatures for color comparison for HFCS and sucrose did not show significant difference.
- e. The cost savings associated with transformation from glass to plastic was calculated to be \$1,171,658 in the year 2017.

- f. Use of liquid nitrogen dosing at 30 kpa (4.4 psi) helps in hot filing of processed foods and results in eliminating paneling and labeling challenges.

APPENDICES

A.1. SAS code used for Sensory Analysis:

```
/*Reading in analysis Excel sheet*/

libname cats "\\Prod-
Isilon.Corporate.ivh\SASDataHome\SASProduction\pfizer\Apixaban\B0661037\Cat";

PROC IMPORT OUT= WORK.sensory
  DATAFILE= "\\Prod-
Isilon.Corporate.ivh\SASDataHome\SASProduction\pfizer\Apixaban\B0661037\Cat\sensory
data.xlsx"
  DBMS=EXCEL REPLACE;

  GETNAMES=YES;
  MIXED=NO;
  SCANTEXT=YES;
  USEDATE=YES;
  SCANTIME=YES;
  SHEET=Sheet2;

RUN;

/*Anova Procedure*/

%macro anva(vr=);
proc anova data=sensory ;
class trt;
model &vr=trt;
means trt/tukey lines ;
run;
%mend;
```

```
%anva(vr=sweet);
%anva(vr=sour);
%anva(vr=mouthfeel);
%anva(vr=color);
%anva(vr=palatability);

/*Summary statistics*/

%macro stat(a=);
proc sort data=sensory;by trt;

PROC MEANS DATA=sensory noprint ;
BY trt ;
VAR &a ;
OUTPUT OUT=st&a N=n MEAN=mean
STD=std stderr=stderr LCLM=lclm UCLM=uclm ;
RUN ;

data st&a;
set st&a;
length variable $50;
variable="&a";
drop _type_ _freq_;
run;

%mend stat;

%stat(a=sweet);
%stat(a=sour);
%stat(a=mouthfeel);
%stat(a=color);
%stat(a=palatability);

data allstat;
set stsweet stsour stmouthfeel stcolor stpalatability;
run;

proc print data=allstat; run;
```

A.2 Statistical Output – Sensory Analysis

Table A.1.1. ANOVA for sensory analysis of sourness.

Source	DF	Sum of Squares	Mean Square	F Value	Pr > F
Model	1	1.6744186	1.6744186	0.44	0.5071
Error	84	316.8837209	3.7724252		
Corrected Total	85	318.5581395			

R-Square	Coeff Var	Root MSE	Sour Mean
0.005256	29.40766	1.942273	6.604651

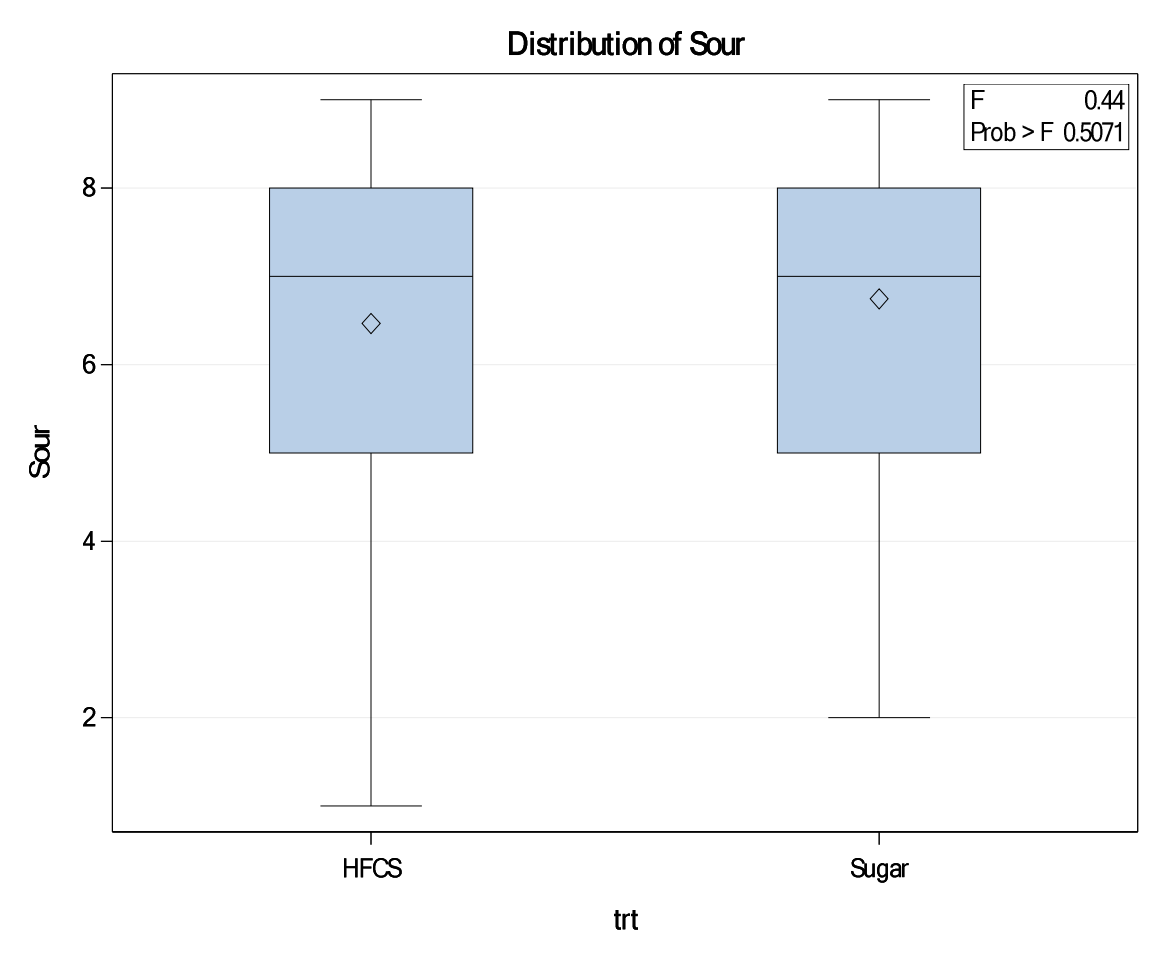


Figure A.1.1. Graph comparing mean values of sourness of HFCS and sugar for sensory analysis.

Sour Tukey Grouping for Means of trt (Alpha = 0.05)

Means covered by the same bar are not significantly different.

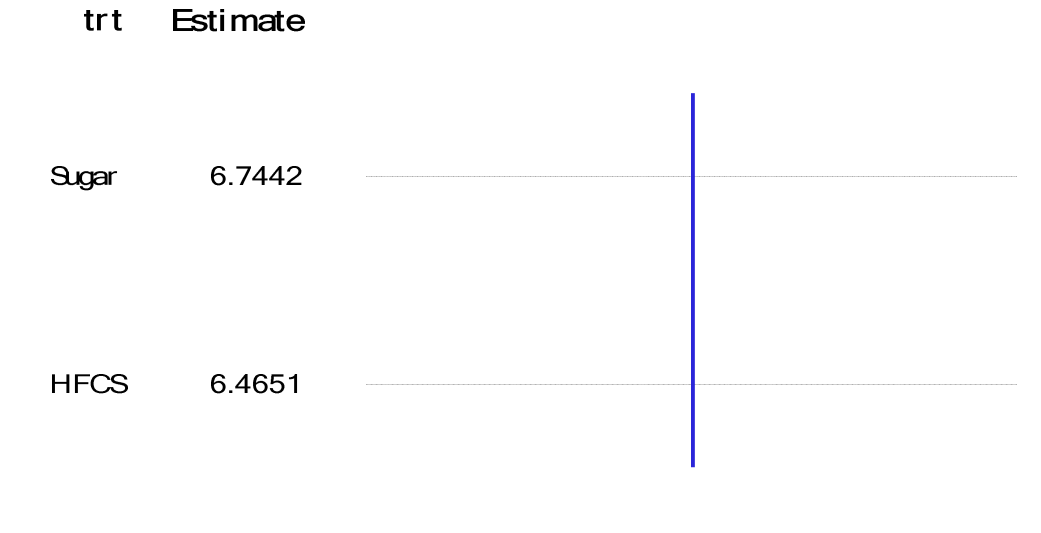


Figure A.1.2. Tukey grouping of mean values of sourness of HFCS and sugar

Table A.1.2. ANOVA for sensory analysis of sweet

Source	DF	Sum of Squares	Mean Square	F Value	Pr > F
Model	1	0.9418605	0.9418605	0.36	0.5524
Error	84	222.2790698	2.6461794		
Corrected Total	85	223.2209302			

R-Square	Coeff Var	Root MSE	Sweet Mean
0.004219	22.17067	1.626708	7.337209

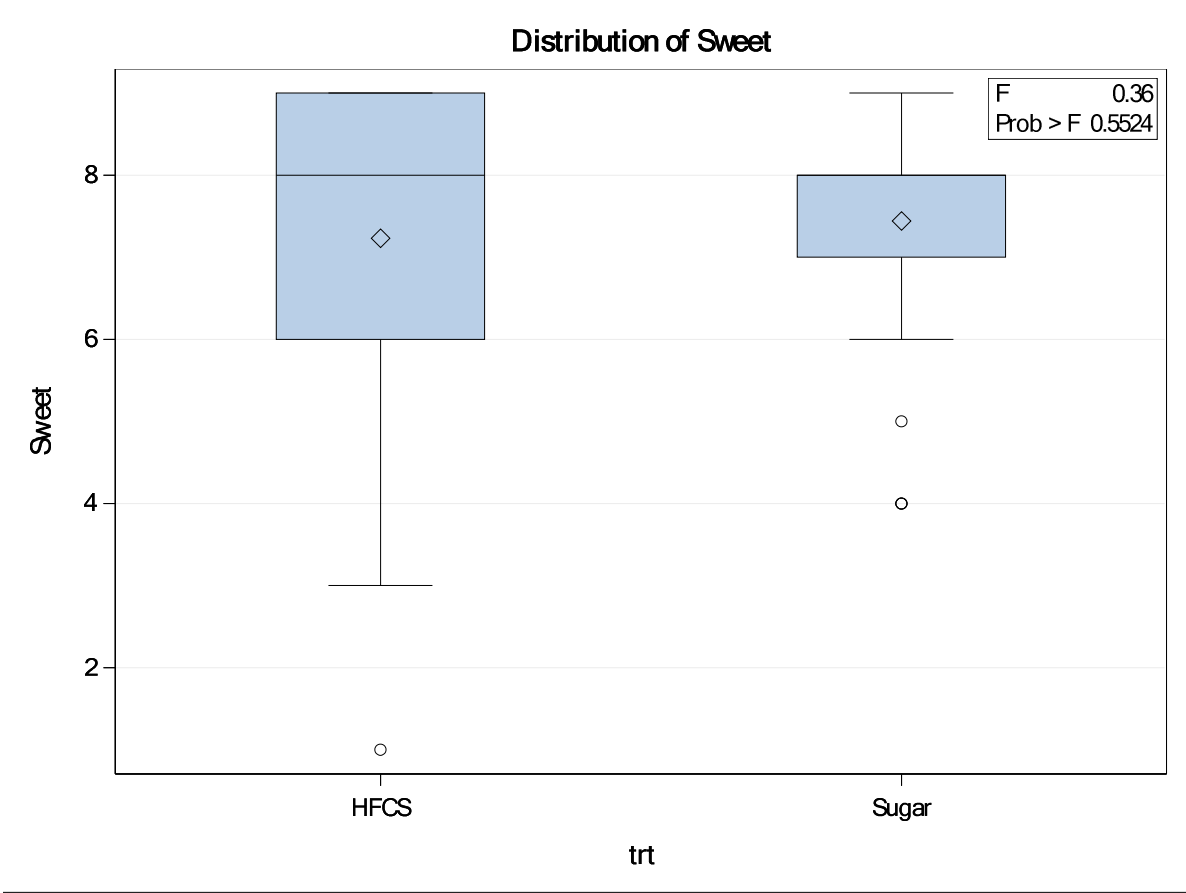


Figure A.1.3. Graph comparing mean values of sweet for sensory analysis of HFCS and sugar.

Sweet Tukey Grouping for Means of trt (Alpha = 0.05)

Means covered by the same bar are not significantly different.

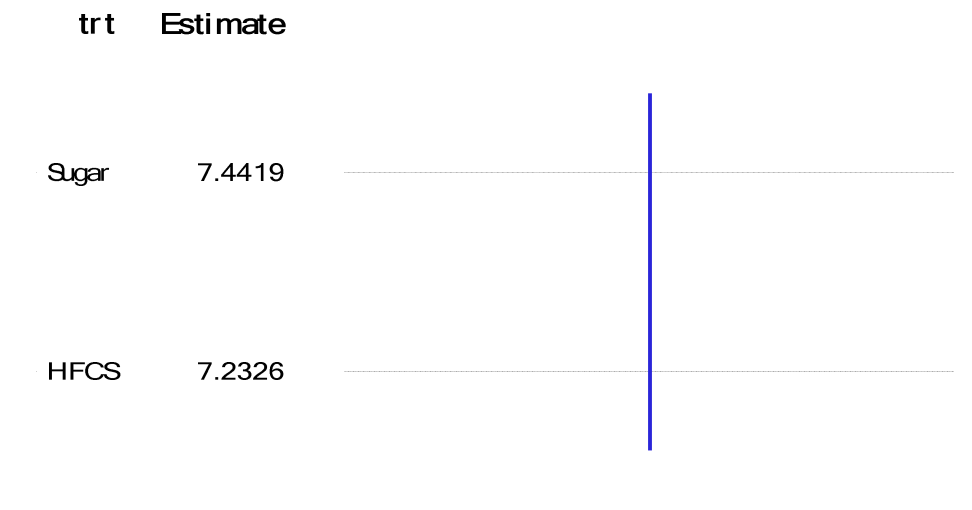


Figure A.1.4. Tukey grouping for mean values of sugar and HFCS for sensory analysis of sweet.

Table A.1.3. ANOVA for sensory analysis of mouthfeel.

Source	DF	Sum of Squares	Mean Square	F Value	Pr > F
Model	1	0.0116279	0.0116279	0.00	0.9451
Error	84	204.5116279	2.4346622		
Corrected Total	85	204.5232558			

R-Square	Coeff Var	Root MSE	Mouthfeel Mean
0.000057	21.33375	1.560340	7.313953

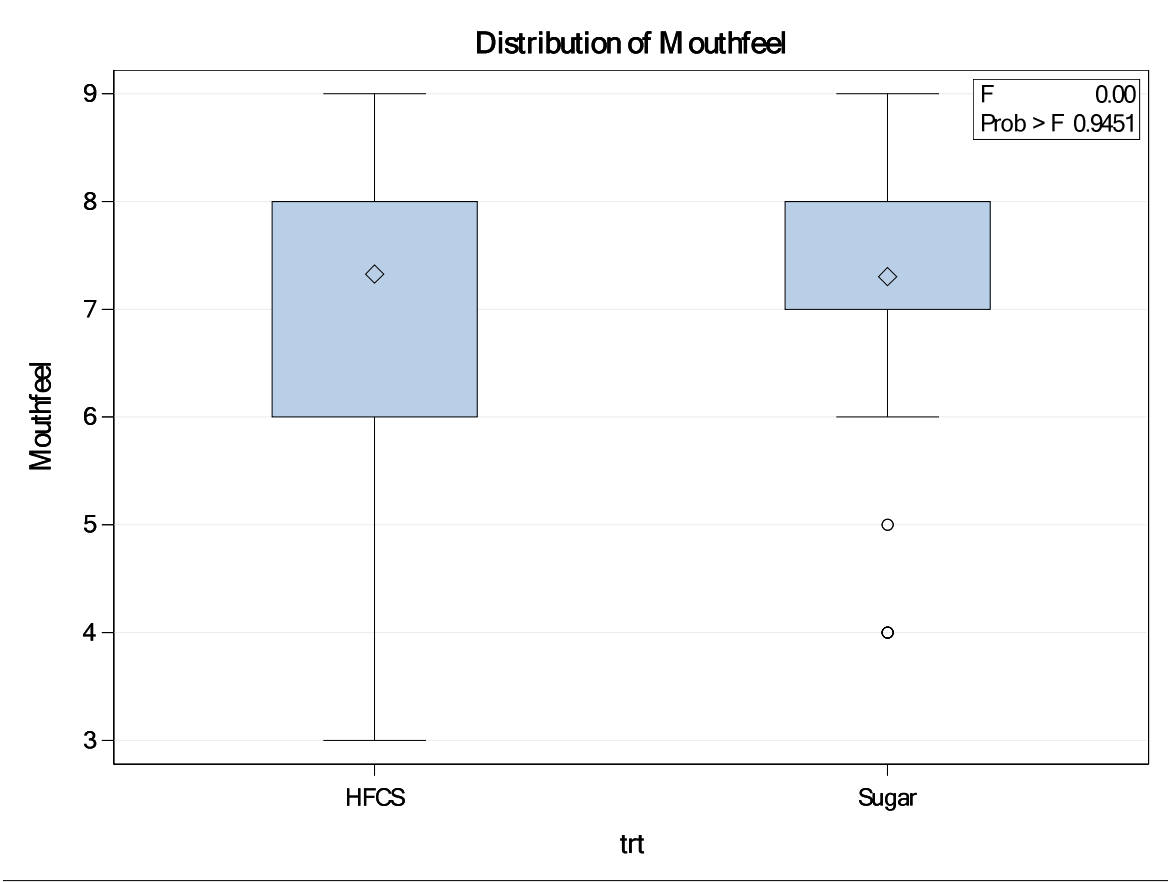


Figure A.1.5. Graph comparing mean values of mouthfeel for sensory analysis.

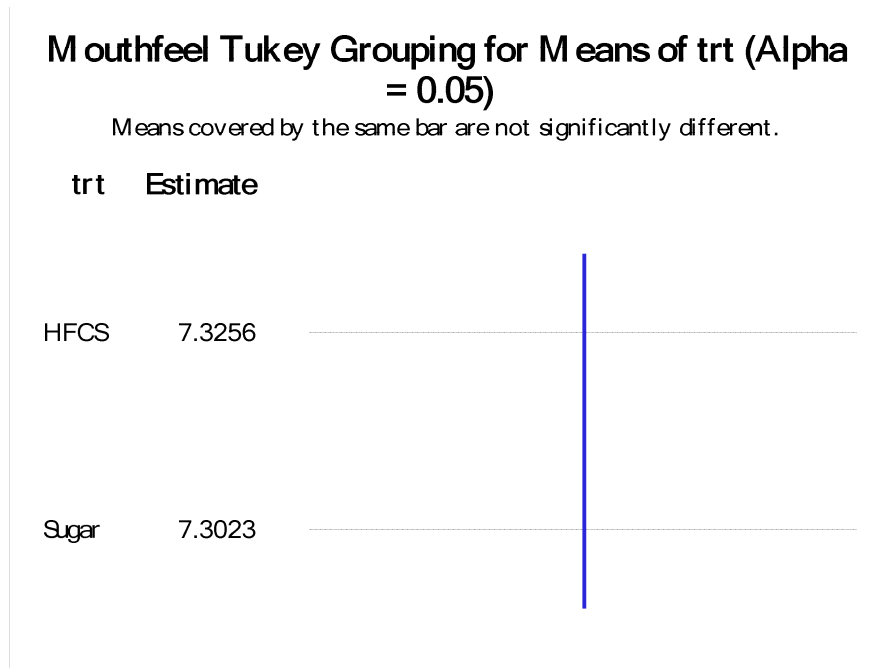


Figure A.1.6. Tukey grouping of mean values of sugar and HFCS for sensory analysis of mouthfeel.

Table A.1.4. ANOVA for sensory analysis of color.

Source	DF	Sum of Squares	Mean Square	F Value	Pr > F
Model	1	0.7441860	0.7441860	0.37	0.5464
Error	84	170.4651163	2.0293466		
Corrected Total	85	171.2093023			

R-Square	Coeff Var	Root MSE	Color Mean
0.004347	18.84791	1.424551	7.558140

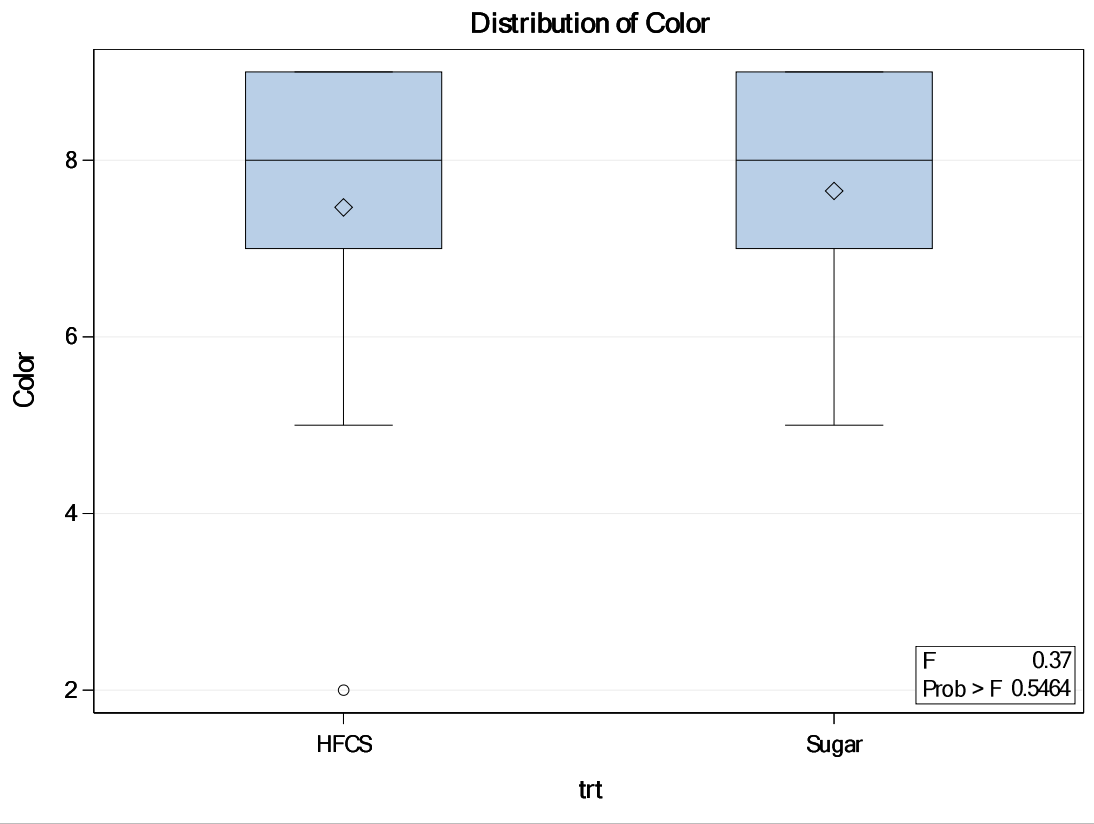


Figure A.1.7. Graph comparing mean values of Color for sensory analysis of HFCS and sugar.

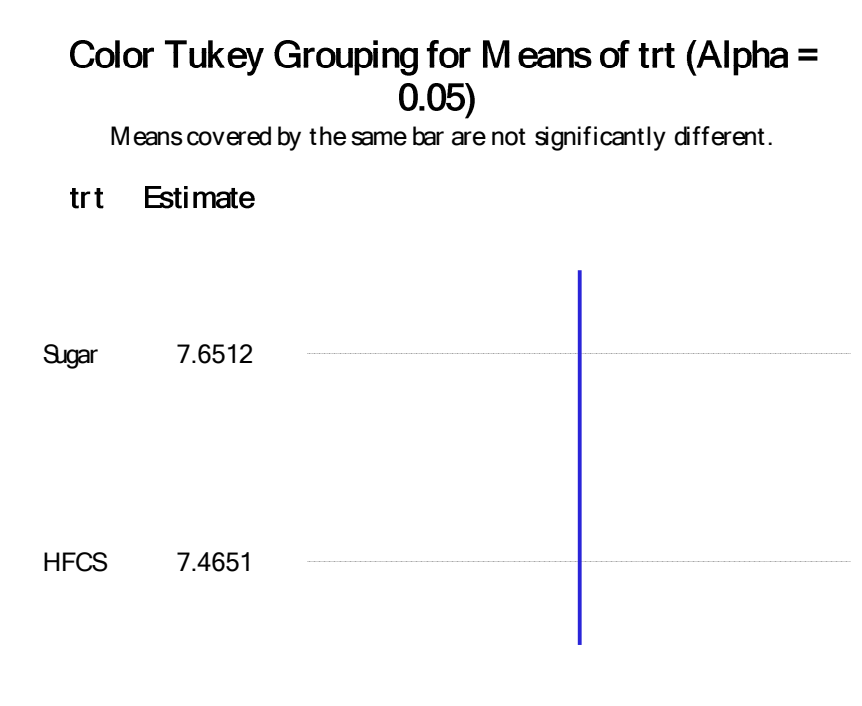


Figure A.1.8. Tukey grouping for mean value of sugar and HFCS for sensory analysis of color.

Table A.1.5. ANOVA for sensory analysis of palatability.

Source	DF	Sum of Squares	Mean Square	F Value	Pr > F
Model	1	3.3604651	3.3604651	1.44	0.2336
Error	84	196.093023 3	2.3344408		
Corrected Total	85	199.453488 4			

R-Square	Coeff Var	Root MSE	Palatability Mean
0.016848	20.43520	1.527888	7.476744

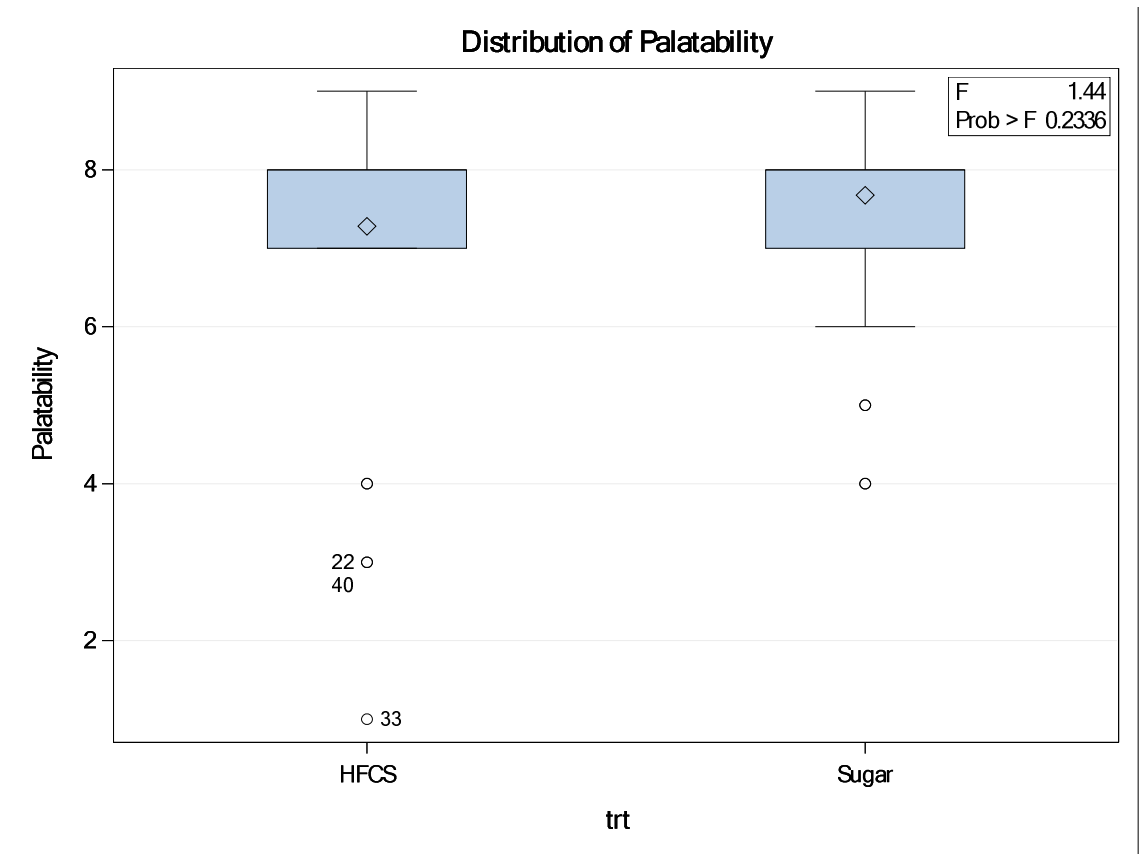


Figure A.1.9. Graph comparing mean values of palatability for sensory analysis of HFCS and sugar

Palatability Tukey Grouping for Means of trt (Alpha = 0.05)

Means covered by the same bar are not significantly different.

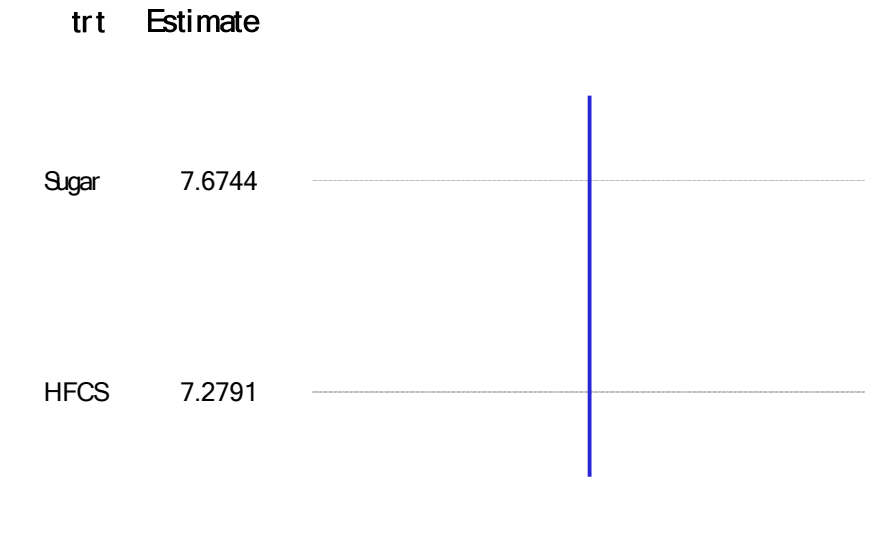


Figure A.1.10. Tukey grouping for mean values of sugar and HFCS for sensory evaluation of palatability.

VITA

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Doctor of Philosophy

Thesis: EFFECT OF LIQUID NITROGEN IN PACKAGING AND FORMULATION CONCEPTS IN ACID FOODS.

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I, 'Praveen Yerramsetti' was born in Kakinada, India on May.20. Son of Anju Babu and Sujatha. I came to US in 2002 and consider US as my second home.

Education:

Completed the requirements for the Doctor of Philosophy in Food Science at Oklahoma State University, Stillwater, Oklahoma in December 2018.

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Completed the requirements for the Master of Science in Biotechnology at Gitam College, Vizag, India in 1999.

Experience:

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2002-2004: Graduate Research Assistant, OSU, Dept of Plant and Soil Sciences

2004-2010: Research Specialist at FAPC, Center for Veterinary Health Sciences, Biochemistry Core Facility, OSU.

2015-Current: Quality Assurance Manager, Head Country BBQ LLC, Ponca city, Oklahoma

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Safe Quality Food (SQF)