

**KWAME NKRUMAH UNIVERSITY OF SCIENCE AND
TECHNOLOGY, KUMASI**

DEPARTMENT OF FOOD SCIENCE AND TECHNOLOGY



**OPTIMIZATION OF SENSORY ACCEPTABILITY OF
MILK CHOCOLATE CONTAINING OKRA PECTIN AS
EMULSIFIER**

By

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A thesis submitted to the Department of Food Science and Technology, Kwame Nkrumah University of Science and Technology in partial fulfillment of the requirements for the award of MSc. Degree in Food Quality Management.

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DECLARATION

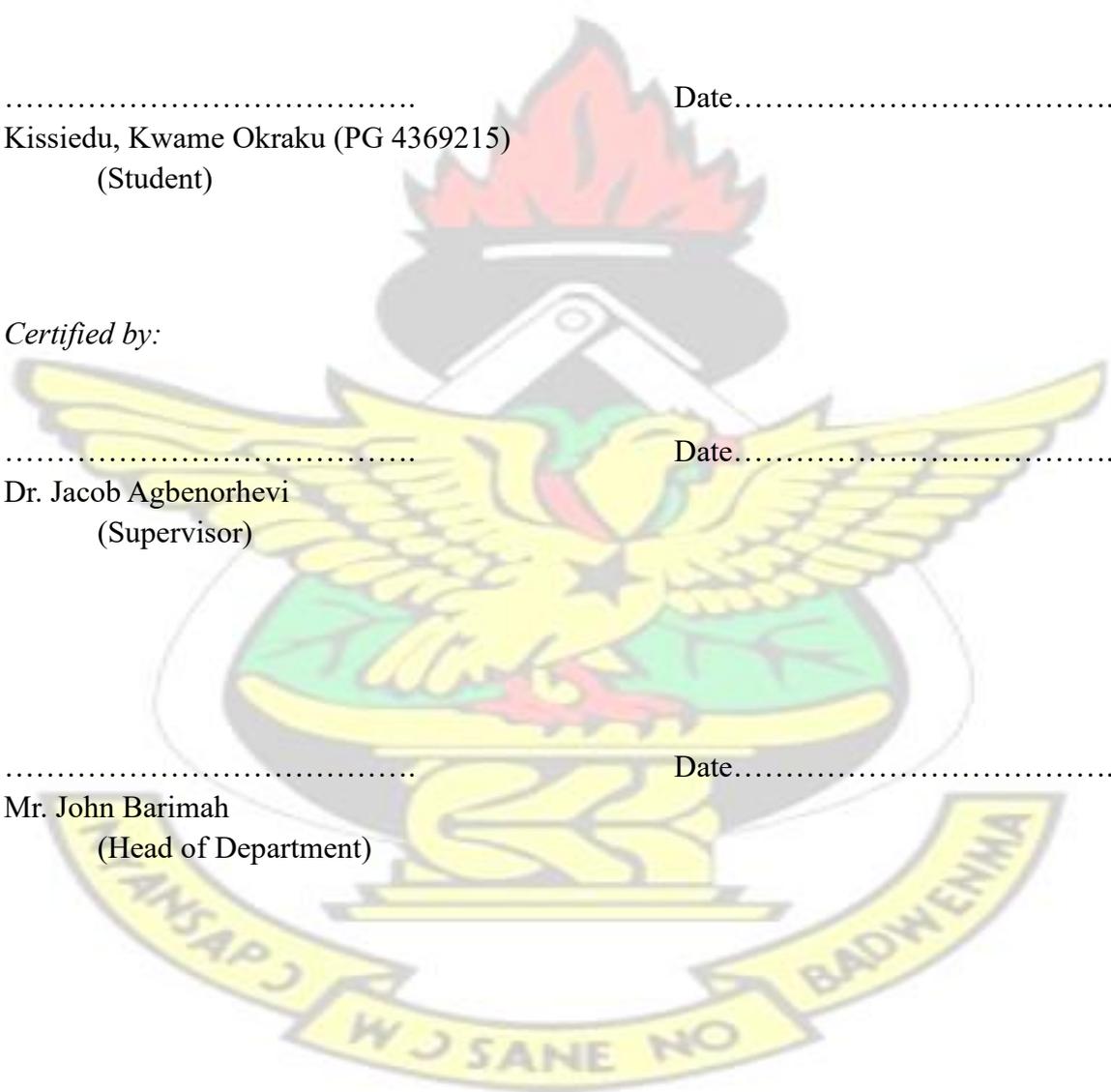
I hereby declare that this submission is my own work towards the Master of Science in Food Quality Management and that, to the best of my knowledge, it contains no material previously published by another person nor material which has been accepted for the award of any other degree of the university, except where due acknowledgment has been made in the text.

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DEDICATION

I wish to dedicate this work to my family; Faustina, Kwaku Ntow and Adwoa Sakyiabea.

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My thank goes to the almighty God for the peace, love and strength to pursue this course.

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Last but not the least, I offer regards and blessings to my wife and children whose love and encouragement helped me to complete this dissertation.

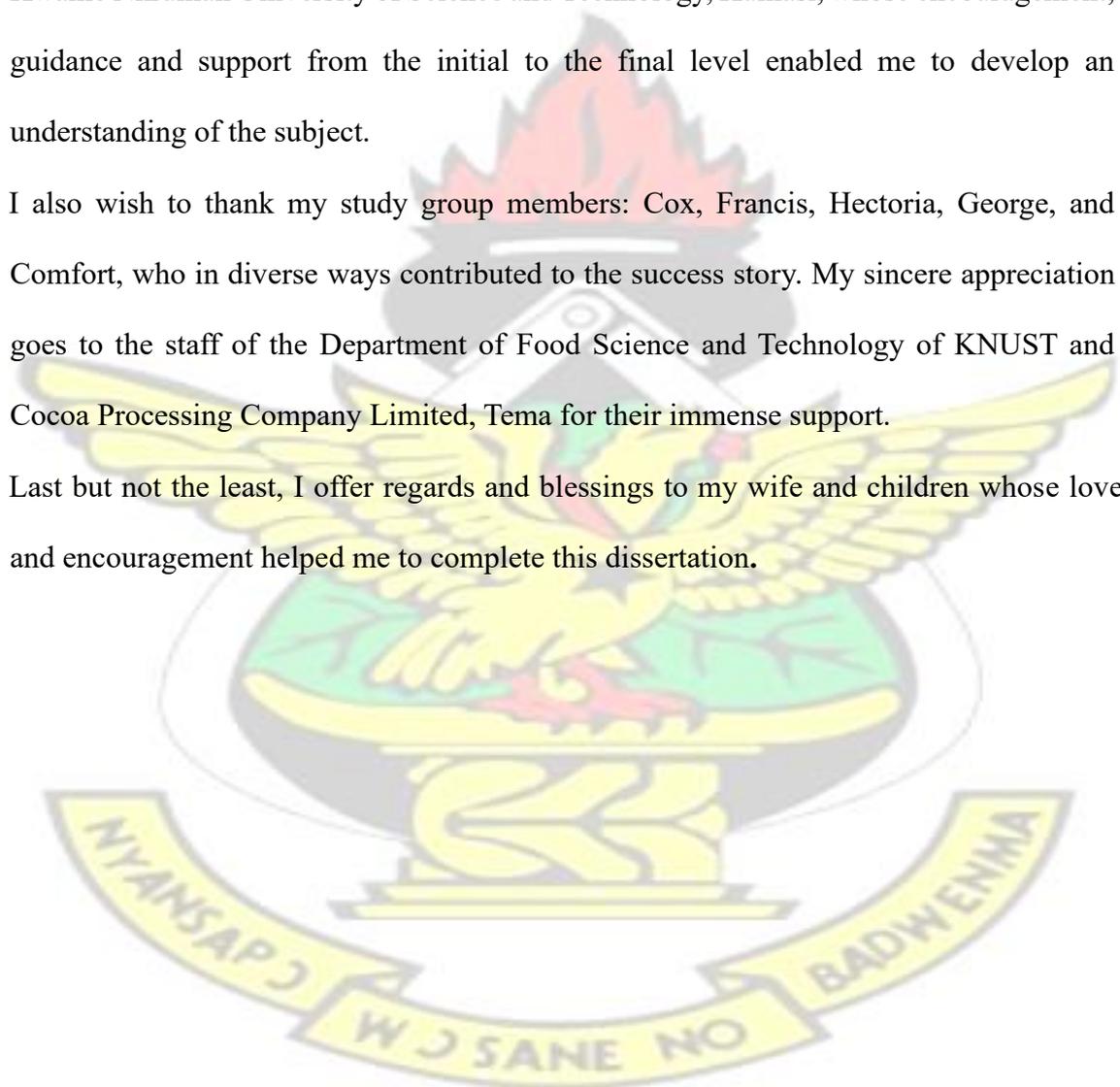


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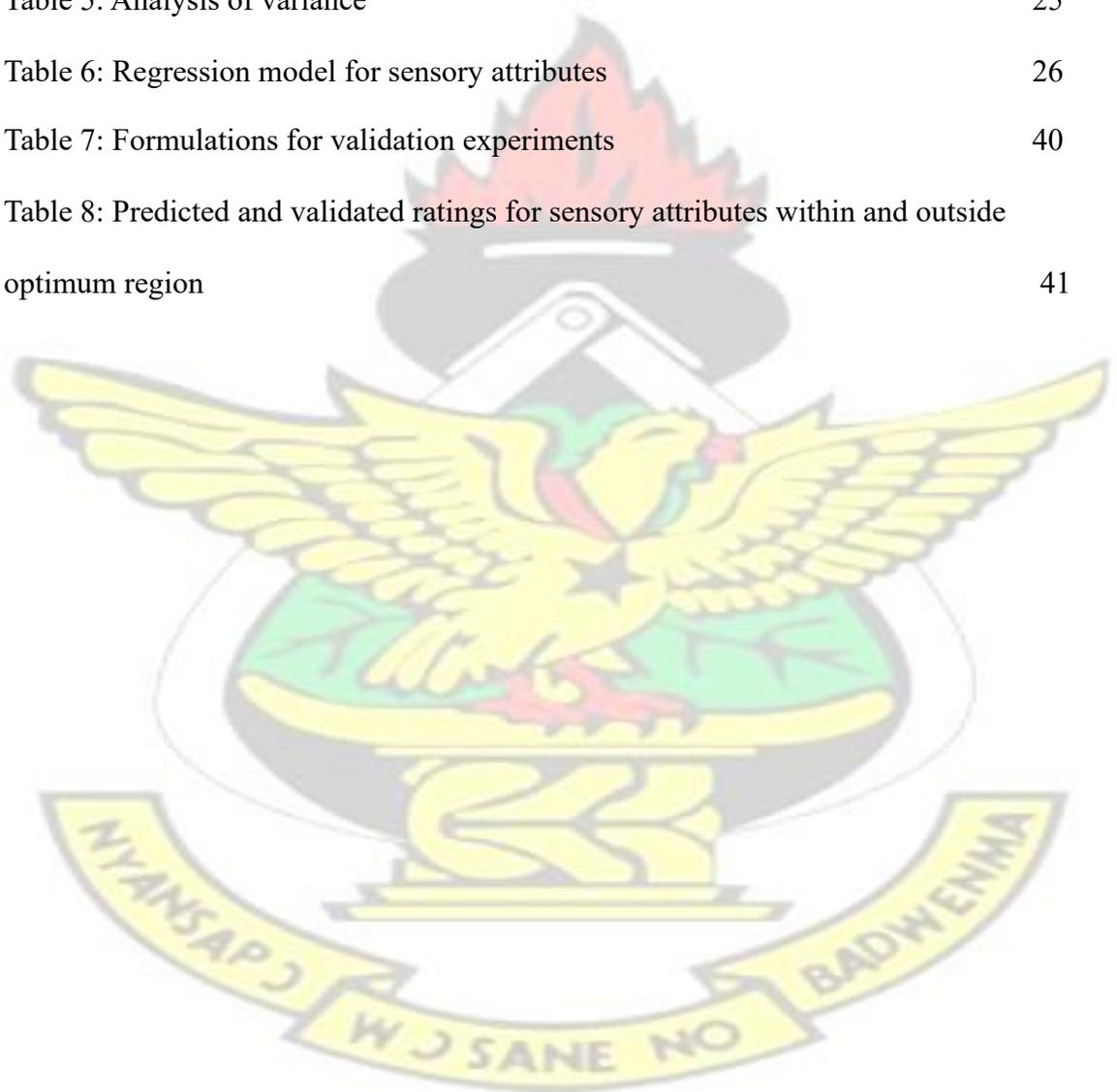
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ABSTRACT

In the preparation of chocolate in Ghana, the only ingredient obtained locally is cocoa. A preliminary study showed the potential of okra pectin as emulsifier in milk chocolate. The present study aims to investigate and optimize the sensory acceptability of milk chocolate when local okra pectin extract is used as an emulsifier or lecithin substitute. A four-component constrained mixture design (Simplex lattice) was used for the recipe formulations using MINITAB (version 17) Statistical Package. The components were sugar, milk, cocoa butter, and cocoa liquor. The lower and upper constraints of the component variables were determined from an existing milk chocolate recipe from the Cocoa Processing Company Limited, Tema, Ghana. The emulsifier, Okra pectin (0.145%) was held constant in all the formulations. The formulated products were evaluated by trained judges for its sensory characteristics such as mouthfeel, aftertaste, flavour, taste, appearance and overall acceptability on a nine-point Hedonic scale. At okra pectin level of 0.145%, the optimal proportion of the ingredients in the milk chocolate based on sensory characteristics were found to be 36.08% sugar, 25.92% milk, 20.00% butter and 18.00% liquor. At these levels, the chocolate samples were either liked moderately or very much, which suggests high overall acceptability (about 90%).

CHAPTER ONE INTRODUCTION

1.1 Background

Chocolate is one of the most popular foods and common confectionery material in the world, people enjoyed for its taste and health benefit. Chocolate is a product of cocoa, made by mixing cocoa masse, cocoa butter, sugar and other ingredients to produce a solid confectionery. West Africa produces more than 70% of the world's cocoa. Unfortunately, the consumption pattern for these producing countries in this region is low. This low patronage may be attributed to the price of the product, purchasing power of citizens in these developing countries and the stability of these products on the shelves. The apparent consumption per capital of 2010/2011 for Ghana for example was only 0.55Kg compared to 5.88kg and 5.66kg for Switzerland and Belgium, respectively (ICC0, 2012).

Different types of chocolate have been developed to suit various uses in the confectionery industry and demands of different markets. There are so many chocolates to choose from, whether imported, domestic, artisanal, mass produced chocolate or filled chocolate. Both dark and milk chocolate are suspension of solid particles from sugar and cocoa dispersed in a fat continuous phase in a process assisted by an emulsifier.

Chocolate has become a product of interest to consumers not only because of its nutritional composition, but also its medicinal properties. It has become a perfect gift for many, but it becomes unattractive and unappealing when the box is unwrapped only to find out that there are gray streaks on the bar compromising its quality. Chocolate can

have a shelf-life of about 12 months, but the stability depends on parameters such as storage temperature, humidity, accessibility of oxygen and ingredients (Tuorila, 1996). During storage and in distribution, oil migration may occur causing a defect called fat bloom. Sugar bloom occurs when the temperature over the surface of the chocolate product drops below the dew point (Čopíková, 1999). The likeness of chocolate by consumers is mostly depends on the sensory characteristics and its acceptance.

In chocolate, the primary emulsifier used is soy lecithin. Mostly, emulsifiers provide control over flow properties when used in chocolates, although they may have other effects such as controlling the viscosity, influencing the fat crystallization and acting as bloom inhibitors. The addition of low levels of emulsifiers can reduce viscosity equivalent to several percent addition of cocoa butter. Therefore, emulsifiers are cost-saving ingredients in chocolate (Hasenhuettl and Hartel, 2008). Different emulsifiers have been used in chocolate manufacturing. Examples are, ammonium phosphatide and polyglycerol polyricinoleate (PGPR), each having different flow properties.

The okra plant, *Abelmoschus esculentus*, is a native plant from Africa, but it could also be found in many areas such as Asia, Middle East and the southern states of the USA. In Ghana, Okra is the fourth most popular vegetable after tomato, pepper and garden egg (Oppong-Sekyere *et al*, 2012). Extract rich in pectins from okra pods has suggested to be a strong candidate for emulsification in an acidic environment (Alba *et al*, 2013).

1.2 Problem statement

Reducing cost and ensuring consistent product quality, maintaining tight recipe control and using no more cocoa butter than necessary are of paramount importance for any chocolate manufacturer. Cocoa butter is the most expensive ingredient in chocolate and accounts for one third of the cost of the final product (Dimick, 1999). Due to the cost of the butter, there are substantial gains to be made from developing more cost-effective chocolate recipes. This is because, cocoa butter saving will typically deliver worthwhile bottom-line effects. About 3.5% less cocoa butter could translate to savings of over €100,000 (\$140,000) when producing 1,000 metric tons (MT) of chocolate and more than €1m (\$1.4m) at a 10,000 MT level (Nieburg, 2014). It is economically prudent therefore, for manufacturers to look more closely at replacing cocoa butter with emulsifiers to realize cost savings.

Lecithin (E322) is the most commonly used type in chocolate. Unfortunately for a developing nation such as Ghana, this ingredient is imported into the country, adding to the cost of production. Some people have concerns about soy lecithin in products because soy is considered a “major food allergen” by the Food and Drug Administration of the United State of America (USFDA, 2009). Another major concern regarding soy lecithin is that, like most soybean products, it is usually derived from genetically modified (GM) soybean plants (Greene, 2013).

Alternative chocolate emulsifiers such as polyglycerol polyricinoleate (E476) and ammonium phosphatide (E442) will equally have to be sourced from another country if they are to be used. The ingredients used in chocolate preparation in Ghana that is obtained locally is cocoa. With this in mind, it is extremely important to investigate the use of indigenous ingredients such as okra pectin as an emulsifier in chocolate. Datsomor

et al., (2016), has investigated the effect of okra pectin as an emulsifier on the yield, textural properties, sensory and consumer acceptability of different chocolate formulations.

With this background, further investigations are therefore needed to better understand the impact of the okra pectin as an emulsifier on sensory properties of milk chocolate, allowing for optimization of the chocolate recipe.

1.3 Purpose of the Study

The purpose of this study was to conduct sensory optimization of milk chocolate when okra pectin is used as an emulsifier and make recommendations for enhancing sensory acceptability of the product.

1.4 Objectives of the Study

The following are the specific objectives of the study to replace lecithin E322 with okra pectin in chocolate production.

- (i) Investigate the effect of okra pectin as an emulsifier in milk chocolate using a mixture design.
- (ii) Evaluate and optimize the sensory acceptability of chocolate developed with okra pectin as an emulsifier/lecithin substitute.

1.5 Research Questions

The study was guided by the following research questions:

- (i) What is the optimum level of milk chocolate when okra pectin is used as an emulsifier?
- (ii) How acceptable is milk chocolate developed with okra pectin as an emulsifier?
- (iii) What measures can be put in place to improve the sensory properties of milk chocolate?

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1.6 Significance of the Study

This study would make an important contribution to literature in the area of optimization and sensory characteristics of chocolate. The finding of this study would reduce the cost of chocolate production in Ghana in the food/chocolate industry. It will also help in the use of locally manufactured ingredients such as okra pectin in chocolates.

1.7 Scope of the Study

The research was limited to the formulation of milk chocolate with minimum cocoa solids of not less than 40%. Due to time constraints, the study focused on the sensory evaluation and optimization of the chocolate samples formulated with okra pectin as lecithin substitute. The okra pectin was extracted from a high yielding Asha genotype. The okra pectin was extracted at the Department of Food Science and Technology, KNUST, Kumasi, whereas the chocolate product development and the analysis were done at the Research and Development Department of Cocoa Processing Company Limited, Tema.

CHAPTER TWO LITERATURE REVIEW

“Cocoa” comes from the word cacao, which is derived from the Mayan and Aztec languages. Some countries in West Africa currently produces more than 70% of the

world's cocoa, with Ivory Coast (39%), Ghana (21%), Nigeria (6%) and Cameroon (5%) being the leading producers (ICCO, 2008).

In Ghana, political economy of cocoa exceeds that of any other commodity. The cocoa bean can be said to grow in about six of the ten regions of the country (Essegbey and Ofori-Gyamfi, 2012). Cocoa bean exports accounts for about 40 percent of Ghana's foreign exchange earnings and provide the second largest source of export dollar (Ashitey, 2012). This provides many households with the fundamental capital base for income and employment.

Currently, Ghana exports about 67.6% of its cocoa to Western Europe. The main importing countries in the region include the Netherlands (33.8%), United Kingdom (12.1%), Belgium (8.9%) and Germany (3.6%). On the other hand, about 7.2% and 3.3% are exported to Japan (7.2%) and United States of America respectively (ISSER, 2011).

The Cocoa Processing Company (CPC) is one of the cocoa processing companies in Ghana. It used to be a subsidiary of COCOBOD but it became privatized with the reformation of the industry. It currently operates with an expanded installed capacity that produces chocolates and cocoa-based sweets and other products for exports and local consumption (Essegbey and Ofori-Gyamfi, 2012)

2.1 Chocolate

2.1.1 Chocolate Ingredients

Chocolate is the generic name obtained by an adequate manufacturing process from cocoa materials which may be combined with milk products, sugars and/or sweeteners, and other additives. Other edible foodstuffs, excluding added flour and starch and animal fats other than milk fat, may be added to form various chocolate products

(Alimentarious, 2003).

Chocolate is prepared from the basic ingredients such as cocoa butter and cocoa liquor, emulsifiers, sugars and a flavor enhancer such as vanillin. The type of chocolate varies greatly on its composition. The main ingredient differentiating milk chocolate from dark chocolate is milk powder that has been added. Milk powder brings about the creamy and smooth flavor of the chocolate. Several different forms of dry milk may be used, including spray-dried milk, roller-dried milk, and milk crumb (Greweling, 2013). White chocolate on the other hand is a milk chocolate without any cocoa particles (meaning no cocoa liquor is added). This gives its distinct white colour.

Sugar is one of the most prevalent ingredients in dark, milk and white chocolates. Its main purpose is to provide sweetness to the bitterness in the cocoa. Nutritive carbohydrate sweeteners such as maltitol in chocolate manufacturing are permitted, but usually crystalline and dense sucrose from sugarcane or sugar beets is by far the sugar most commonly used in chocolate. The sugar in chocolate is not dissolved but is refined to very small particles to create a smooth mouthfeel. The large sugar particles may be milled into small particles prior to being mixed with the batch or may be fully refined together with the other chocolate ingredients.

Emulsifiers are added to improved rheological properties with lower viscosity. The most frequently used emulsifier is soy lecithin but other emulsifiers such as polyglycerol polyricinoleate (PGPR) may occasionally be used.

2.1.2 Chocolate Manufacturing

Traditionally the mixture is kneaded or mixed and refined in two staged process (two roller and five roller refiners). The refined mass is then transferred to a hollow vessel that is opened at the top called a conche. At this stage, the refined mixture is continuously mixed for about 18 hours with more butter, lecithin and vanillin added, after which the chocolate masse is pumped into tanks for use (Minifie, 1989). The masse is tempered for the most stable crystals of the cocoa butter in the chocolate to form before it is deposited into the moulds.

2.1.3 Sensory of chocolate

In confectionery senses of sight, touch, smell, and taste are critical to the consumer's appreciation of products (Voltz and Beckett, 1997). For example, chocolates that does not look glossy or worse still if it is bloomed, it is unlikely to be purchased. Touch on the other hand is related to how a chocolate breaks and also its behavior in the mouth. It therefore includes the snap of a chocolate and how it melts away smoothly.

Chocolate is known to be a highly craved product that has uniquely attractive taste that has benefits to the health (Hill and Heaton-Brown, 1994; Serafini *et al.*, 2003 and Chiva, 1999). It derives its popularity from the ability to arouse sensory pleasure and also trigger positive emotions especially when consumed during depressive moods.

(Macht and Dettmer, 2006; Macdiarmid and Hetherington, 1995).

The technique employed in chocolate processing, distributed particle size and composition of the ingredients or the recipe can have a tremendous effect on characteristics such as the sensory perception, physical properties and the rheological behavior of the chocolate. Servais *et al.* (2002) reported that the particle size distribution and composition has an influence on the rheological properties of chocolates. Afoakwa *et al.* (2008) also indicated that the texture with specific surface area and the rheology can

be influenced by the particle size distribution. The mean particle size is capable of influencing plastic viscosity, yield stress, spread of the product and its hardness. Smaller particles improve organoleptic characteristics (Ziegler *et al.*, 2001) of the chocolate. The plastic viscosity and yield stress goes up due to changes in surface area of particles in contact with fat phase. This means that the ability to optimize the particle size can greatly reduce requirement for viscosity modifiers and also has an improvement on process control (Afoakwa *et al.*, 2008).

Differences in the sensory perception of chocolate can also be linked to the differences in the type of cocoa used, variations in the recipe, whether a milk crumb or milk powder was used, and blending and processing methods.

Important to sensory characteristics is the fat in the chocolate composition. This has an effect on the melting properties and mouth feel. Solid chocolates melt at oral temperature of about 37 °C during consumption giving a smooth suspension of particulate solids in cocoa butter and milk fat (Beckett, 2011; Whitefield, 2005).

2.1.4 Chocolate blooming

2.1.4.1 Sugar bloom

Bloom refers to the gray cast, streaks, or spots that appear on chocolates. There are two types of bloom: sugar and fat bloom. Sugar bloom occurs either through improper storage conditions, especially in a high humidity environment or quickly transferring the product from an area of low to high temperature. These conditions result in moisture on the surface of the chocolate, and this consequently dissolves sugar. As the surface water evaporates, sugar crystals remain on the surfaces, producing a white appearance (Afoakwa, 2010).

2.1.4.2 Fat bloom

Fat bloom is the most popular chocolate defect that makes the chocolate undesirable for consumers, who expect a product to have a glossy surface and desired color, but instead, a bloomed chocolate may visibly appear to be stale and aging, and this is usually identified by a beige coating on the surface of the chocolate. A bloomed chocolate has a characteristic loss of gloss on the surface, giving rise to an appearance that can either be from a uniform dull gray to a marbled aspect, or from small individual white spots to an even larger white spot on the chocolate. This blooming can be attributed to factors such as improper processing conditions, composition, and temperature (Lonchamp and Hartel, 2004).

Cocoa butter is polymorphous in nature. It consists of six different crystal forms (I through VI) with each successive form exhibiting increased stability (Wille and Lutten, 1966). The most desirable polymorph state in a well-tempered chocolate is the form V crystals. It is at this state that the melting and solidification properties are most desirable compared to the other crystal forms. Chocolate bloom is known to occur through any of three different situations (Lonchamp and Hartel, 2004). One of which is when poor tempering of chocolate causes cocoa butter to crystallize in the form IV polymorph, which promptly changes to form V upon cooling and storage. The newly formed form V crystals located at the surface are visible as bloom. Also, when chocolate that has a mixture of different types of TAG. When this happens, the phase behavior of the TAG becomes disrupted leading to the formation of large crystals on the surface (Cebula and Ziegler, 1993). The other scenario is when chocolate that has been properly tempered to the most stable form V crystals is subjected to an elevated temperature or to thermocycling. This may lead to the formation of form VI crystals (Hachiya *et al*, 1989).

2.1.5 Chocolate Emulsifiers

Chocolate has a hydrophilic sugar and lipophilic cocoa particles dispersed in the continuous phase of fat from the cocoa butter (van Nieuwenhuyzen and Szuhaj, 1998). Emulsifiers function is to regulate rheological properties of fats (Johansson and Bergenstahl, 1992). In the chocolate matrix, the sugar particles are coated with the emulsifier. This helps in the facilitation of the flow of the sugar in the continuous fat phase. It also helps in the distribution of the particles evenly throughout the emulsion so that agglomeration does not occur. Emulsifiers can alter the viscosity of specific foods (Walter and Cornillon, 2001). This is even more important when it comes to chocolate manufacturing in processes such as panning, enrobing, molding, or depositing (Rector, 2000). Emulsifier has the capability to reduce viscosity and control the overall rheology of chocolate, thereby allowing chocolate manufacturers to optimize their processes in order to minimize production costs.

The rheological properties of molten chocolate are important for chocolate quality assurance and accurate weight measurements. Molten chocolate behaves as a nonNewtonian liquid, exhibiting non-ideal plastic behaviour with a yield stress, related to amount of energy required to initiate fluid flow, and plastic viscosity, energy required to keep fluid in motion (Beckett, 1999; Afoakwa *et al.*, 2007).

By using the viscometer, the Casson equation can be used to calculate for two important flow parameters. These are the yield value and plastic viscosity (Beckett, 2000). This equation is used to describe the flow properties of chocolate,

$$\sqrt{\tau} = \sqrt{\tau_C A} + \sqrt{\mu_C A} \sqrt{\dot{\gamma}}$$

Where

τ (yield stress) τ_{CA} (Casson

yield stress) μ_{CA} (Casson

plastic viscosity) $\dot{\gamma}$ (shear

rate)

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Plastic viscosity relates to pumping characteristics, filling of rough surfaces and coating properties. On the other hand, yield value relates to shape retention, pattern holding, inclined surface coating and bubbles in processing (Seguine, 1988). Plastic viscosity and yield value are the energy required to keep fluid in motion and the minimum force that must be applied to initiate flow respectively.

Emulsifiers included in the recipe at certain concentrations can facilitate the reduction of the overall fat content from cocoa butter, thereby enhancing functionality of the (Bamford *et al.*, 1970; Walter and Cornillon, 2001). In certain cases, emulsifiers have been used to as replacement for the cocoa butter. (Rector, 2000).

Emulsifiers have also been used to influence the tempering behaviour and the sensitivity to relative humidity and temperature (Afoakwa *et al.*, 2007). Emulsifier also has an effect on solid chocolate. For example, is on susceptibility of the chocolate to fat blooming, stability against the migration of fat and the oxidation from fillings (Schantz and Rohm, 2005).

Different emulsifiers are used in the manufacturing of chocolate. Notable ones are soy lecithin, polyricinoleate (PGPR) and ammonium phosphatide (Schantz and Rohm, 2005; Bamford *et al.*, 1970; Wilson *et al.*, 1998). Additionally, citric acid esters of mono-

diglycerides have also been studied as potential emulsifiers for chocolate formulation (Afoakwa *et al.*, 2007).

The most popular emulsifier used in the manufacturing of chocolate is the soy lecithin (Schantz and Rohm, 2005). It is obtained in the preparation of soya oil. It is a byproduct in the oil preparation that results as a light to medium pasty brown product of a liquid to paste consistency (Surh *et al.*, 2007). It is added at approximately 0.5% (w/w) to reduce the viscosity during processing. At this percentage, it increases the efficiency of conching process. The addition of lecithin reduces the amount of cocoa butter needed to attain the texture needed. Meaning, addition of lecithin can reduce the production cost. Addition of 0.3% lecithin reduces chocolate viscosity and increases chocolate's tolerance to moisture (Afoakwa *et al.* 2007). On the other hand, too much lecithin can cause off-flavors and also increase the viscosity of chocolate.

Polyglycerol polyricinoleate on the other hand is developed from either esterification of polyglycerols with polymerized ricinoleic acid or polycondensation of castor oil and glycerol. It is a mixture with a polyglycerol backbone dominated by di, tri, and tetraglycerols (Wilson *et al.*, 1998). PGPR has the ability to reduce or possibly eliminate the yield value of chocolate tremendously, thereby transforming a non-Newtonian liquid into a Newtonian so that the chocolate can flow more easily. This is an important characteristic for the enrobing and moulding techniques in the chocolate processes (Schantz and Rohm, 2005). PGPR is most efficient in enhancing the flow of chocolate into molds. Air bubbles that may be trapped in the moulds are reduced (Fletcher, 2006).

Both lecithin and PGPR are capable of synergistically working with other emulsifiers, such as ammonium phosphatide and citric acid esters (Stier, 2009). Ammonium

phosphatide is either manufactured synthetically from a mixture of ammonium salts of phosphorylated glycerides or from a mixture of glycerol and partially hardened rapeseed oil (Surh *et al.*, 2007). Rapeseed oil is usually used because it has an advantage in respect to taste, availability, and price (Schneider, 1986).

2.2 Okra (*Abelmoschus esculentus* L.)

2.2.1 The Okra plant

Okra, *Abelmoschus esculentus* belongs to the family Malvaceae. The English term 'okra' came into use in the late 18th century (Arapitsas, 2008). It has different local names in different parts of the world. In Ghana for example the name for okra in the Twi language is nkuruma (Benjawan *et al.*, 2007).

It is an economically important crop that is able to grow in tropical and subtropical parts of the world (Oyelade *et al.*, 2003) including Ghana. Okra (*Hibiscus esculentus*) is one of the most commonly grown vegetables in Ghana. Other vegetables commonly grown include tomato, onion, shallots, egg plant, local, Indian or Gambian spinach, sweet and chillipepper, and hot pepper (Sinnadurai, 1971).

In 2007, the okra production globally was estimated at 4.8 million tons. Ghana contributed to about 2% of the global output (Gulsen *et al.*, 2007). In Ghana, cultivation is mainly in the Northern part of the country but it could be found in almost all markets in the country, whether in its fresh state during the rainy season or in a dehydrated form during the dry season.

It is also a multipurpose crop because of its various uses, whether it is the fresh leaves, buds, flowers, pods, stems or the seeds (Yonas *et al.*, 2014). For example, the immature

fruits (green seed pods), which are consumed as vegetables, can be used in salads, soups and stews, fresh or dried, fried or boiled (Ndunguru and Rajabu, 2004).

2.2.2 Okra pectin

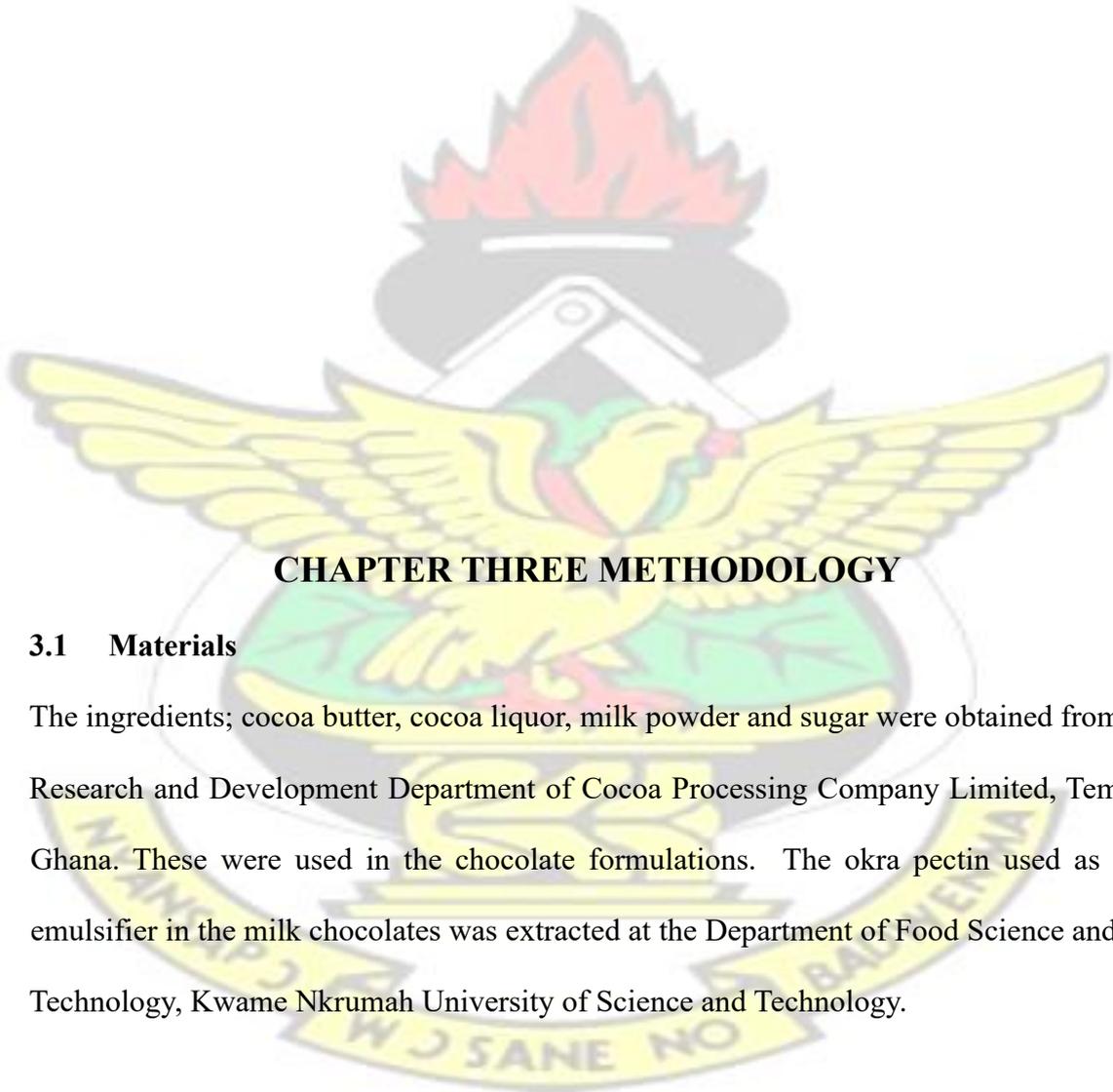
Pectin are commonly used as gelling, stabilizing or thickening agent in many food products such as jam, yoghurt drink, fruity milk drinks and ice cream (Laurent and Boulenguer, 2003).

The thick and slimy texture of okra water-extracts is attributed to its polysaccharide content (Whistler and BeMiller, 1993). Such extracts can be used as natural food-grade emulsifiers (Ndjouenkeu, *et al.*, 1997) or thickeners and emulsion stabilizers (Georgiadis *et al.*, 2011). This suggests that can be a promising source of texture modifiers for complex food matrices. Okra polysaccharide was also shown to have unusual lubricity properties and to be able to form a tenacious coating on the skin which is difficult to remove by washing (BeMiller *et al.*, 1993). The lubricity property is also an important property for a good food fat mimetic (Glicksman, 1991) and okra polysaccharides can be used as fat substitute in many products like chocolate bars and cookies. Many quality characteristics of such fat free cookies were comparable with those of full fat cookies (Romanchik-Cerpovicz *et al.*, 2002).

Likewise, okra polysaccharide has also been used as a milk-fat substitute in chocolate frozen dairy desserts where it could replace the milk-fat up to 70 % while the melting points of the products did not change, although the melting rate decreased slightly (Constantino and Romanchik-Cerpovicz, 2004). Okra polysaccharides also exhibit foam

(Baht and Tharathan, 1987) and emulsion stabilizing properties (BeMiller *et al.*, 1993). It behaves like egg white at higher concentrations which can form threads and stabilize foams, therefore, okra polysaccharides are also use as a dried egg white substitute (Woolfe *et al.*, 1977).

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CHAPTER THREE METHODOLOGY

3.1 Materials

The ingredients; cocoa butter, cocoa liquor, milk powder and sugar were obtained from Research and Development Department of Cocoa Processing Company Limited, Tema, Ghana. These were used in the chocolate formulations. The okra pectin used as an emulsifier in the milk chocolates was extracted at the Department of Food Science and Technology, Kwame Nkrumah University of Science and Technology.

3.1.1 Preparation of okra pectin

The method for preparation of okra pectin is a modification from Alba *et al.*, (2013).

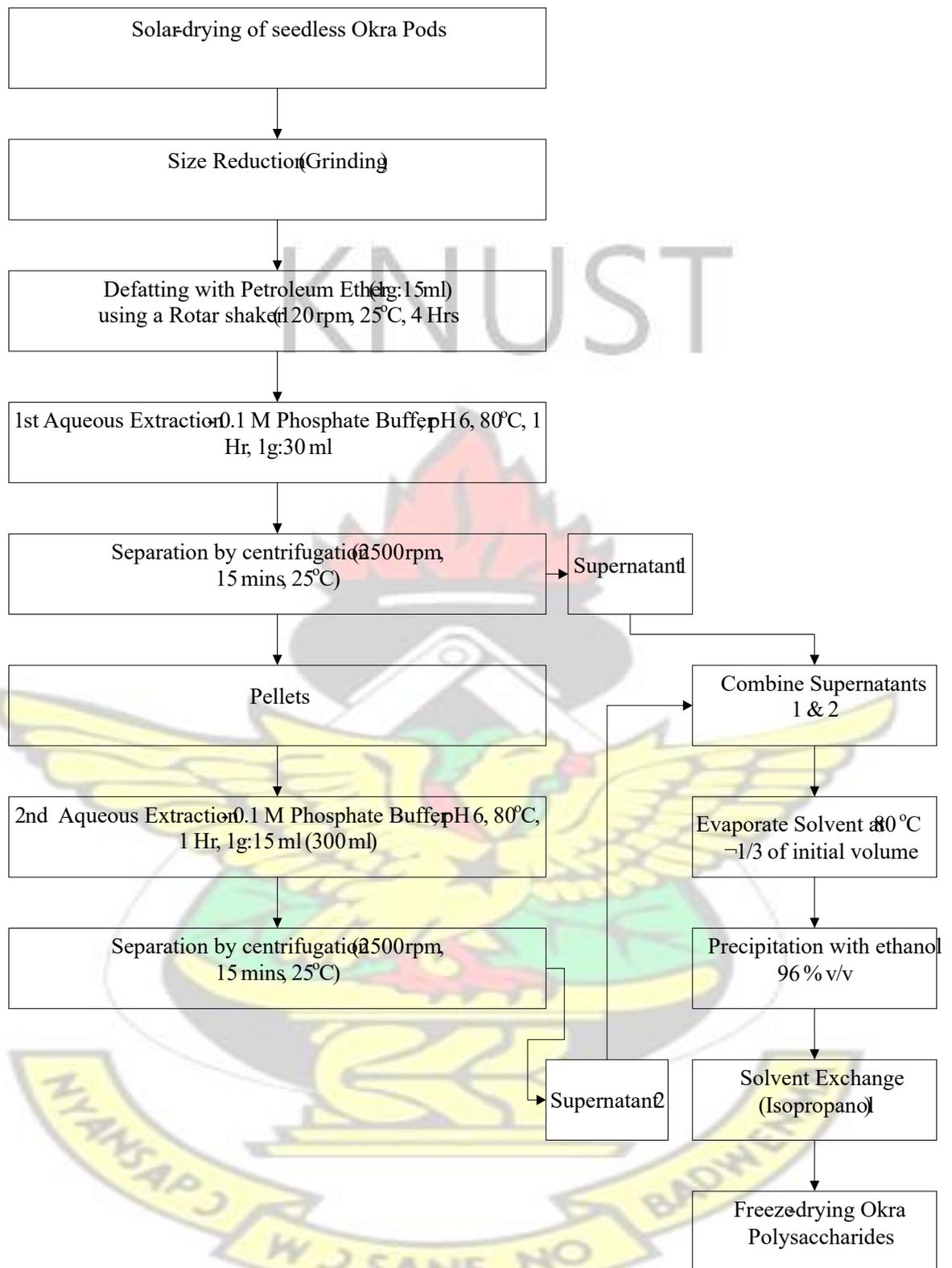
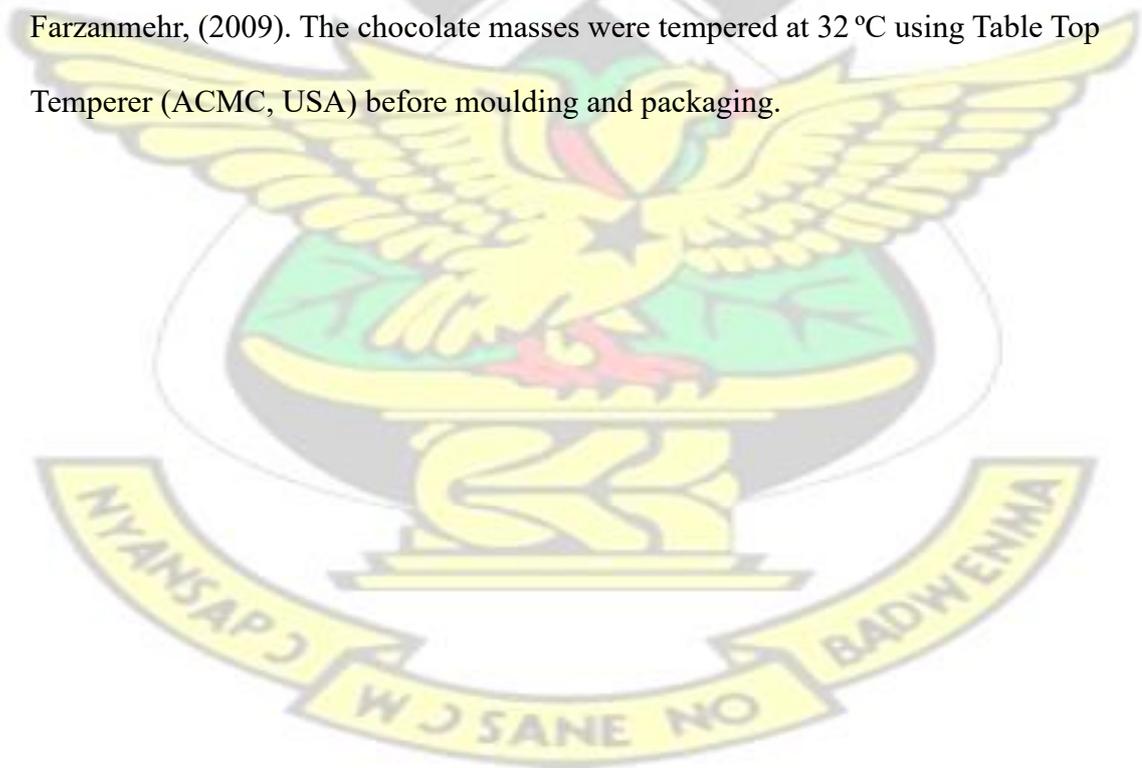


Figure 1: Okra extraction process

3.1.2 Milk chocolate preparation

For each experiment, a 2000 g batch of milk chocolate was produced using an attrition ball mill WIENEROTO (Wiener and co., The Netherlands). The chocolate production starts with the manual feeding of ingredients; cocoa butter, cocoa liquor, sugar and milk into the jacketed mill grinding chamber, that has been pre-heated to about 60 °C. The milling temperatures are control by thermostat so that the product does not suffer product damage. The products were allowed to mill for about 45 min after which the okra pectin was added. The milling was continued for another 15 min. After the established refining time, the milk chocolate was discharged in a stainless-steel container. The milled chocolate with a particle size of less than 20 microns was conched for about 4 h in a laboratory mini conche. The time was an adaptation from Abbasi and Farzanmehr, (2009). The chocolate masses were tempered at 32 °C using Table Top Temperer (ACMC, USA) before moulding and packaging.



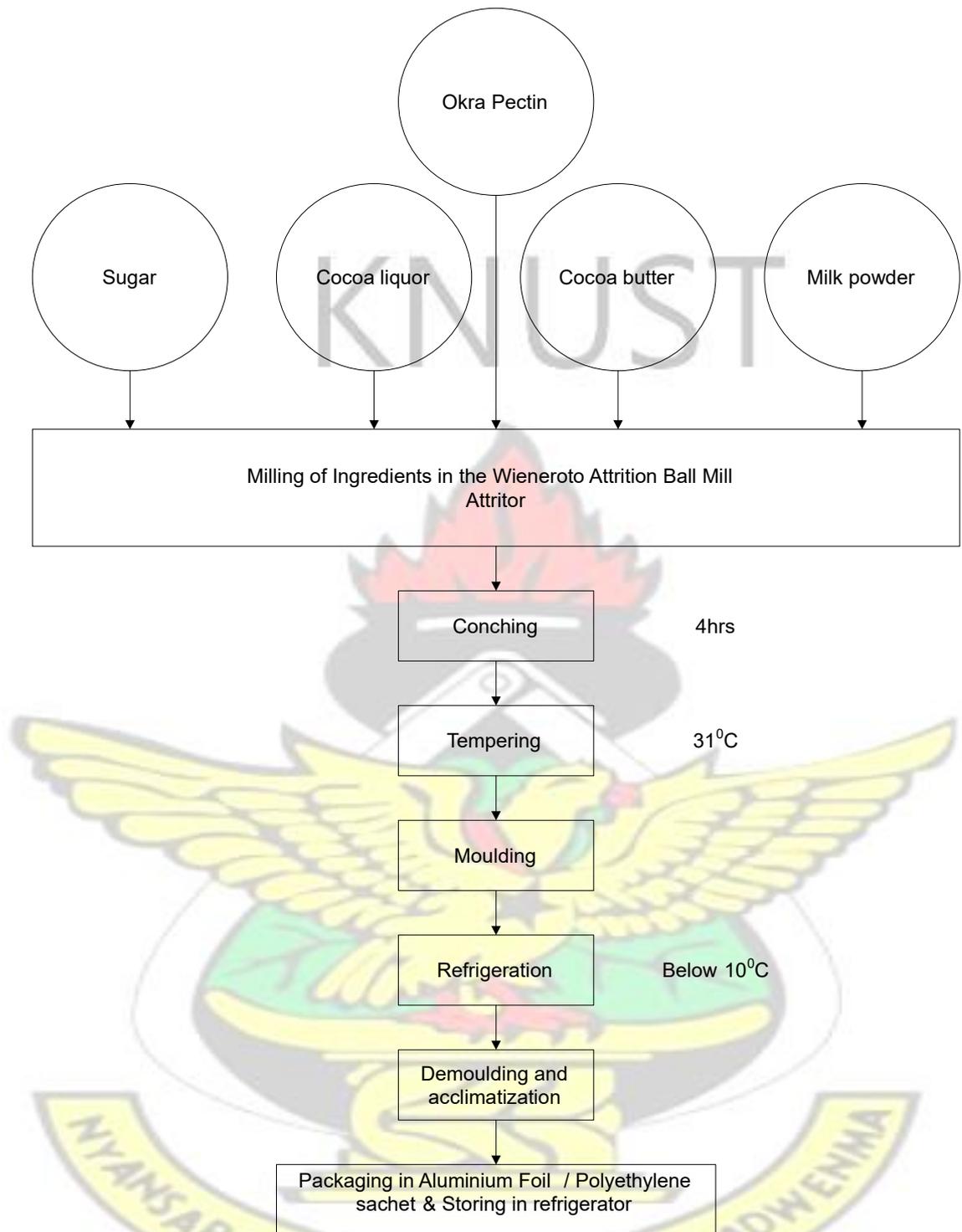


Figure 2: Flow diagram of chocolate process

3.2 Chocolate storage

Packaged samples were kept at a temperature controlled ($25\pm 5^{\circ}\text{C}$) storage room for a day before the sensory evaluation. For the validation evaluation, the samples were kept at the sample temperature for three (3) more days after the first sensory test. All samples were wrapped in an aluminum foil to prevent environmental effects.

3.3 Design of experiment

A four-component constrained mixture design (Simplex lattice) was used for the recipe formulations using MINITAB (version 17) Statistical Package. The components were sugar (X1), milk (X2), cocoa butter (X3) and cocoa liquor (X4). The lower and upper constraints of the component variables were determined from an existing milk chocolate recipe from the Cocoa Processing Company Limited, Tema, Ghana. The emulsifier, Okra pectin (0.145%) was held constant in all the formulations. This is because from Datsomor *et al*, (2016), okra pectin added at 0.145% of the total weight of the ingredients produced chocolate with similar flow and yield to standard milk chocolate produced with lecithin. The component proportions were expressed as fractions of the mixture, and the sum ($X1+X2+X3+X4$) of the proportions equaled 1 (or 100).

Table 1: Lower and upper bound constraints for each mixture component

COMPONENT/INGREDIENT	LOWER BOUND CONSTRAINT (%)	UPPER BOUND CONSTRAINT (%)
Sugar	30	44
Milk	18	32
Cocoa butter	20	34
Cocoa liquor	18	32

Source: Cocoa Processing Company Limited, Tema.

Table 2: Simplex lattice Design for the 15 formulations

StdOrder	RunOrder	PtType	Blocks	SUGAR	MILK	BUTTER	LIQUOR
3	1	2	1	37.00	18.00	27.00	18.00
15	2	-1	1	31.75	19.75	21.75	26.75
6	3	2	1	30.00	25.00	27.00	18.00
2	4	2	1	37.00	25.00	20.00	18.00
9	5	2	1	30.00	18.00	27.00	25.00
8	6	1	1	30.00	18.00	34.00	18.00
4	7	2	1	37.00	18.00	20.00	25.00
13	8	-1	1	31.75	26.75	21.75	19.75
5	9	1	1	30.00	32.00	20.00	18.00
7	10	2	1	30.00	25.00	20.00	25.00
1	11	1	1	44.00	18.00	20.00	18.00
14	12	-1	1	31.75	19.75	28.75	19.75
12	13	-1	1	38.75	19.75	21.75	19.75
11	14	0	1	33.50	21.50	23.50	21.50
10	15	1	1	30.00	18.00	20.00	32.00

NB: Constant in all the formulation is the okra pectin

3.4 Sensory Analysis

The formulated products were evaluated by trained judges for its sensory characteristics such as mouthfeel, aftertaste, flavour, taste, appearance and overall acceptability on a nine-point Hedonic scale. Chocolate quality may be defined by consumer tasting which evaluates the eating quality in terms of characteristics such as appearance, taste, mouthfeel, flavour and aftertaste (Afoakwa, 2008)

The sensory evaluations were done in a room illumined with fluorescent light. The temperature of the room was below 30°C. The judges were asked to allow a minute interval between tasting of samples and then score for the attributes of each sample based on the agreed numbers on the 9-point Hedonic scale (with 1=dislike extremely to 9=liked extremely). About three (3) pieces of 10g of each formulation were presented to each

judge in a disposal trays coded with three-digit random numbers. Panelists were provided with water and were allowed to reevaluate samples. To cope with fatigue of analyzing all these 15 formulated samples for each attribute, the Balanced Incomplete Block Design, Plan 11.24, Type I from Cochran and Cox, (1957), was used as

$$t=15, k=3, r = 7, b=35, \lambda=1, E=.71,$$

Where t =number of treatments, k =the number of experimental units per block, r = the number of replications of each treatment, b = the number of blocks λ =the number of blocks in which each pair of treatment occurred together E = efficiency factor.

3.5 Statistical analyses

Statistical analyses of the data were done using MINITAB17. Mixture regression analysis was used to fit the data to polynomial models as follows:

$$Y = \beta_1X_1+\beta_2X_2+\beta_3X_3+\beta_4X_4+\beta_{12}X_1X_2+\beta_{13}X_1X_3+\beta_{14}X_1X_4+\beta_{23}X_2X_3+\beta_{24}X_2X_4+\beta_{34}X_3X_4$$

where Y = a predicted response, and $\beta_1, \beta_2, \beta_3, \beta_4, \beta_{12}, \beta_{13}, \beta_{14}, \beta_{23}, \beta_{24}, \beta_{34}$ are the corresponding parameter estimates for each linear and cross-product term. Also, analyzed from the sensory evaluation were the cox response trace plot, and the overlaid contour plots.

CHAPTER FOUR RESULTS AND DISCUSSION

4.1 Mean ratings for the sensory attributes

A summary of the mean scores for the sensory attributes is shown in Table 3 on the Milk Chocolate prepared from mixtures containing 30-44 % sugar, 18-32% milk powder, 20-34% cocoa butter and 18-32% cocoa liquor (with okra pectin remaining constant in all the formulations). Ratings on appearance, mouthfeel, taste, aftertaste and overall acceptability were above 5.0 for fourteen out of the fifteen samples. However, flavour had a minimum statistic below 5.0 (4.17). These suggest, generally that the fifteen formulated samples were liked on the hedonic scale.

Table 3: Descriptive statistic for the sensory attributes

	Range	Minimum	Maximum	Mean	Std. Deviation	Variance
	Statistic	Statistic	Statistic	Statistic	Statistic	Statistic
appearance	1.71	5.29	7.00	6.2213	.12684	.49124
mouthfeel	3.00	5.00	8.00	6.6407	.20323	.78711
flavour	3.16	4.17	7.33	6.3180	.25381	.98299
taste	2.83	5.60	8.43	6.7427	.20560	.79627
aftertaste	3.23	5.20	8.43	6.7587	.23658	.91626
overall acceptability	1.50	5.66	7.16	6.5353	.12085	.46805

Table 4: Mean ratings of the sensory attributes

Formulation and mean ratings of sensory attributes						
	APPEARANCE	MOUTHFEEL	FLAVOUR	TASTE	AFTERTASTE	OVERALL ACCEPTABILITY
1	6.86±1.35	7.14±1.68	6.14±2.54	7.00±1.67	7.86±1.07	7.83±0.98
2	5.80±1.92	6.60±1.67	4.17±2.93	6.40±1.52	6.20±2.28	7.00±1.41

3	6.67±2.35	7.00±1.66	6.13±2.53	6.00±2.74	5.78±2.49	5.50±2.56
4	7.00±0.71	7.00±1.41	7.20±1.30	7.60±1.14	7.00±2.00	7.33±1.21
5	5.80±1.64	7.20±2.49	4.50±3.15	5.60±1.67	5.20±2.39	5.40±2.19
6	6.86±0.90	8.00±1.41	6.29±1.38	5.86±2.04	5.67±1.63	6.20±0.84
7	6.40±1.52	5.80±2.59	6.75±0.96	7.20±0.84	7.20±1.10	7.00±1.73
8	6.00±1.73	7.11±1.36	7.33±1.50	7.00±0.93	7.22±0.97	7.13±1.25
9	6.50±1.41	5.50±1.77	7.13±0.99	7.50±0.76	7.86±0.90	7.38±1.41
10	5.71±2.63	6.14±1.21	5.29±1.89	5.71±2.36	5.71±2.14	5.40±2.30
11	5.29±1.89	5.00±1.26	7.25±1.98	8.43±0.79	8.43±0.79	8.43±0.79
12	6.00±1.79	7.43±1.27	6.17±1.72	6.67±2.16	6.67±1.03	7.60±1.52
13	6.13±2.23	6.50±1.31	6.75±2.49	7.33±0.87	6.88±1.25	6.75±1.04
14	6.00±2.16	6.29±1.80	7.17±1.47	6.40±1.67	7.00±1.53	7.40±1.14
15	6.30±1.57	6.90±1.29	6.50±1.35	6.44±1.67	6.70±1.42	6.88±1.13

Table 5: Analysis of Variance (p-values)

Attributes	p-values*
Appearance	0.939536
Mouthfeel	0.092123
Flavour	0.099645
Between Groups	
Taste	0.112374
Aftertaste	0.01616
Overall acceptability	0.016501

*p < 0.05 means significantly different.

The table 5 shows the p-values for each attribute after the Analysis of Variance (between groups). The results indicated that at 5% significance level, aftertaste and overall acceptability were considered significant (p=0.016).

Mixture regression analysis (Table 6) was used to fit the response data (of sensory attributes) to quadratic polynomial models.

$$Y = \beta_1 X_1 + \beta_2 X_2 + \beta_3 X_3 + \beta_4 X_4 + \beta_{12} X_1 X_2 + \beta_{13} X_1 X_3 + \beta_{14} X_1 X_4 + \beta_{23} X_2 X_3 + \beta_{24} X_2 X_4 + \beta_{34} X_3 X_4$$

Table 6: Regression model for sensory attributes

Term	COEFFICIENT					
	APPEARANCE	MOUTHFEEL	FLAVOUR	TASTE	AFTERTASTE	OVERALL ACCEPTABILITY
SUGAR(X1)	-62.7	-58.36	-19.5	10.5	-6.6	-27.3
MILK (X2)	6.3	-87.4	10.9	65.5	119	22.8
BUTTER(X3)	31.4	11.54	61.5	10.1	-39.1	15.1
LIQUOR(X4)	64.8	64.98	91.4	46.1	68.9	67.2
SUGAR*MILK (X1)(X2)	175.9	352.77*	98.6	-47	-179.3	80.2
SUGAR*BUTTER (X1)(X3)	108.6	113.78	-42.2	3.7	217.1	80.2
SUGAR*LIQUOR (X1)(X4)	78.5	-48.11	17	-23.8	-34.1	-2.1
MILK*BUTTER (X2)(X3)	-58.6	42.97	-11.9	-94.4	-119.2	-48.3
MILK*LIQUOR (X2)(X4)	-189.9	-19.79	-247.9	-221.1*	-249.3	-185.6*
BUTTER*LIQUOR (X3)(X4)	-210.4*	-72.2	-333.3	-71.3	-132.8	-164
R ²	84.64%	92.42%	65.60%	93.07%	85.87%	87.62%
R ² (ADJUSTED)	56.98%	78.78%	3.67%	80.59%	60.43%	65.34%

*Significant coefficient at $p \leq 0.05$

The predictive models are summarized in Table 6. The R² values of the models ranged from 0.656 to 0.9307 with the adjusted R² ranging from 0.0367 to 0.8059. The R² value indicated the goodness of a developed model, with the value approaching one for the best model and approaching zero for the worst model. More generally, a higher value of R² means that the developed model can better predict the results.

The response trace plot or the cox plot (Figures 3-8) were applied to statistically analyze the effect of sugar, milk, butter and liquor (when the okra pectin which served as an emulsifier was held constant) on the sensory characteristics of milk chocolate in this study. Theoretically, a response trace plot can show the effect of each component on the corresponding response. Several response traces, which are a series of predictions from the fitted model, are plotted along a component direction. The trace curves indicate the

effect of changing the corresponding component along an imaginary line (direction). The points along a trace direction of a component are connected, thereby producing as many curves as there are components in the mixture. The Response trace plots was very useful for this study, because components in the mixture were more than three.

4.2 Effects of components on appearance

Appearance is an important attribute for chocolate. This is because it gives an indication on whether the chocolate was well tempered or there had been an initiation of fat or sugar bloom. The minimum and maximum statistical values for the appearance were 5.29 (neither liked nor disliked) to 7.00 (liked moderately).

The magnitudes of the coefficients for the four pure mixtures indicated that cocoa liquor (64.8) produced a milk chocolate with higher likeness for appearance than cocoa butter (31.4), milk (6.3) and sugar (-62.7). The regression model for appearance was reasonably accurate ($R^2 = 84.64\%$).

Positive coefficients for two-blend mixtures, Sugar*Milk (175.9), Sugar*Butter (108.6) and Sugar*Liquor (78.5) for appearance indicate that the two components acted synergistically or were complementary. On the other hand, negative coefficients for Milk*Butter (-58.6), Milk*Liquor (-189.9) and butter*liquor (-210.4) blend for appearance indicates that the two components are antagonistic towards one another. The butter by liquor mixture is the only two-blend mixture that can be judged as significant ($p=0.039$).

The Cox Response plot (Figure 3) suggests that an increase in the proportion of the milk had relatively no effect on the appearance of the milk chocolate, but as the proportion of

the butter increases there was an increase in the appearance. The proportion of liquor and sugar in the reference blend can be said to be near optimal.

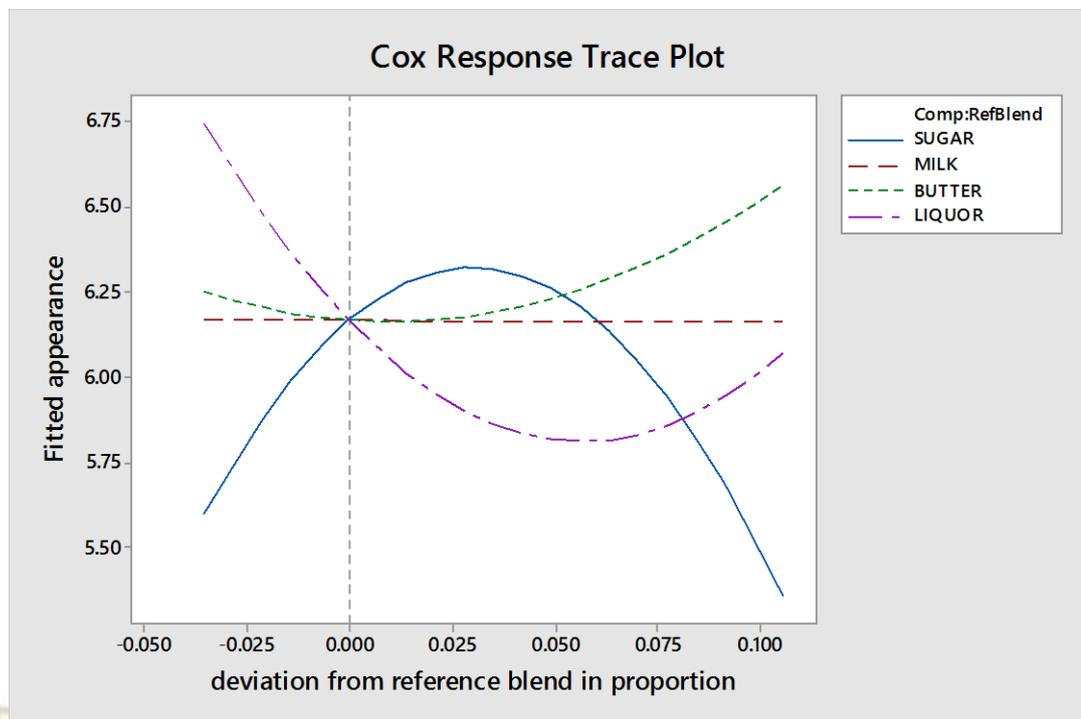


Figure 3: Cox Response Trace plot for Appearance

4.3 Effect of component on mouthfeel

Mouth feel is an important sensory attribute for consumer acceptability of chocolate. This because it measures the feel of smoothness, grainy, chalky, heat and coolness among others on the tongue when the product is in the mouth. (Folkenberg *et al.*, 1999).

The minimum and maximum statistical values for the mouthfeel were 5.0 (neither liked nor disliked) to 8.00 (liked very much). The coefficient in the pure mixture indicated that cocoa liquor (64.98) produced a milk chocolate with higher likeness for mouthfeel than cocoa butter (11.54), milk (-87.4) and sugar (-58.36). The regression model for mouth feel

was reasonably accurate ($R^2 = 92.48\%$). The only two-blend mixture that can be judged as significant ($p = 0.009$) was Sugar*Milk.

As the proposition of sugar in the blend increases the mouthfeel decreases. Same can be said for the milk. There was either an increase or decrease in mouthfeel as the liquor increases. However, there was a directly proportional increase in the mouthfeel when butter in the reference blend was increased. Chocolate has a unique mouthfeel because cocoa butter has a narrow melting point, very close to body temperature.

Smoothness may either be the absence of particles, lumps, bumps or any other noticeable texture in the product. The effect of the increase in the butter may also be because it lubricates further any unpleasant graininess that may be coming from either the sugar or milk. Only low quality chocolates are grainy. Fat brings about satiety in a food product thereby making the food feel smooth in the mouth.

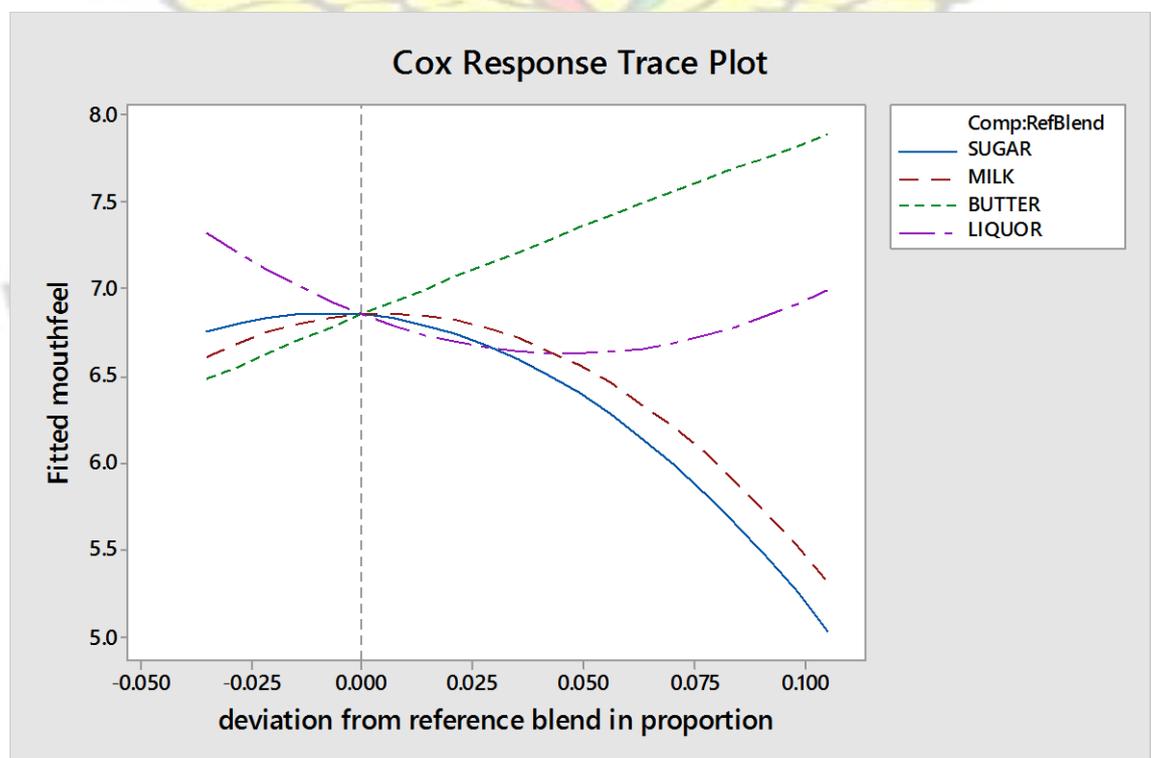


Figure 4: Cox Response Trace plot for mouthfeel

4.4 Effect of component on flavour

The flavor profile of chocolate has over 600 different volatile compounds that contribute to consumer perception of taste and aroma (Counet *et al.*, 2002).

The minimum and maximum statistical values for the flavour were 4.17 (disliked slightly) to 7.33 (liked moderately). The coefficient in the pure mixture indicated that cocoa liquor (91.4) produced a milk chocolate with higher likeness for flavour than the rest of the components. The coefficient two-blend mixture (98.6) for sugar and milk indicated a high likeness for flavour. The regression model for flavour was averagely accurate ($R^2 = 65.60\%$) compared to the other attributes. This did not fit very well with the model. None of the pure mixture or the two-blend mixtures can be judged as significant. The flavor matrix of chocolates is greatly dependent on concentration of volatile components and is influenced by the rate of release from chocolate which is dependent on temperature, molecular interactions, and partition coefficients of the particular compounds (Kinsella, 1990). The differences between chocolate flavors are usually based on the type of cocoa bean, how it was fermented and roasted. The liquor and butter used in the formulation were all from the same batch, hence a negligible effect on the flavour variation. The chocolate flavour is also affected by the conching process as well as the type and proportion of ingredients included in each chocolate formula. All the fifteen samples were subjected to the same conching and tempering processes, hence temperature may not have affected the flavour of the 15 samples.

From the Cox Response plot (Figure 5) As the proposition of milk in blend increases, there was a direct increase in the flavour. Milk chocolate flavour has a lot to do with the type of milk or cream product that is used, as well as the strength and taste of the cocoa liquor. Usually harsh cocoa solids may hide the milky creamy flavours, but once the cocoa

intensity is reduced as it can be seen in the Plot, the creaminess comes to the fore, thereby enhancing the flavor.

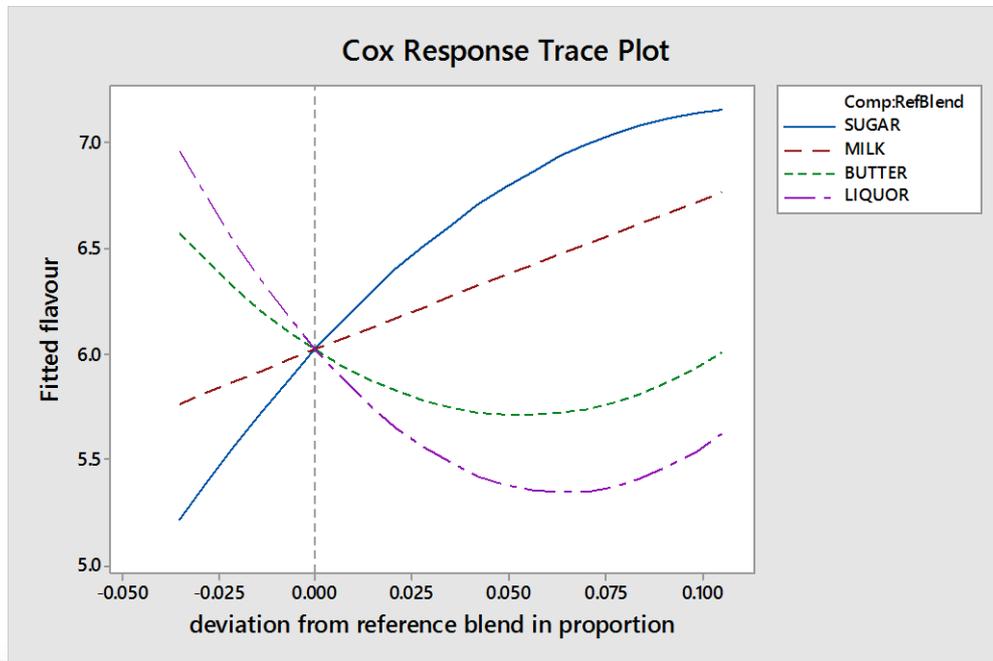


Figure 5: Cox Response Trace plot for flavour

4.5 Effect of component on taste

One of the key determinants of chocolate acceptance or rejection is its taste. The taste of chocolate products being sweet, bitter, sour or salty is used as a critical factor for quality, and dictates their preference and marketability by consumers.

From Table 3, the minimum and maximum statistical values for the taste were 5.6 (liked slightly) to 8.43 (liked very much). The least rating for taste was obtained for formulation 5 (5.6), while the highest mean score (8.43) was obtained for formulation 11 (table 4). The magnitudes of the coefficients for the four pure mixtures indicated that milk (65.5) produced a milk chocolate with higher likeness for taste than the other attributes. The regression model for taste was reasonably accurate ($R^2 = 93.07\%$). The milk by liquor mixture is the only two-blend mixture that can be judged as significant

($p=0.044$).

To a very large extent, ingredient composition has an influence on the Chocolate taste than processing technology. The Cox Response plot (Figure 6) suggests that as the proportion of the sugar increases there was an increase in the taste. Likewise, as the proportion of the butter decrease, there was a decrease in the taste of the product.

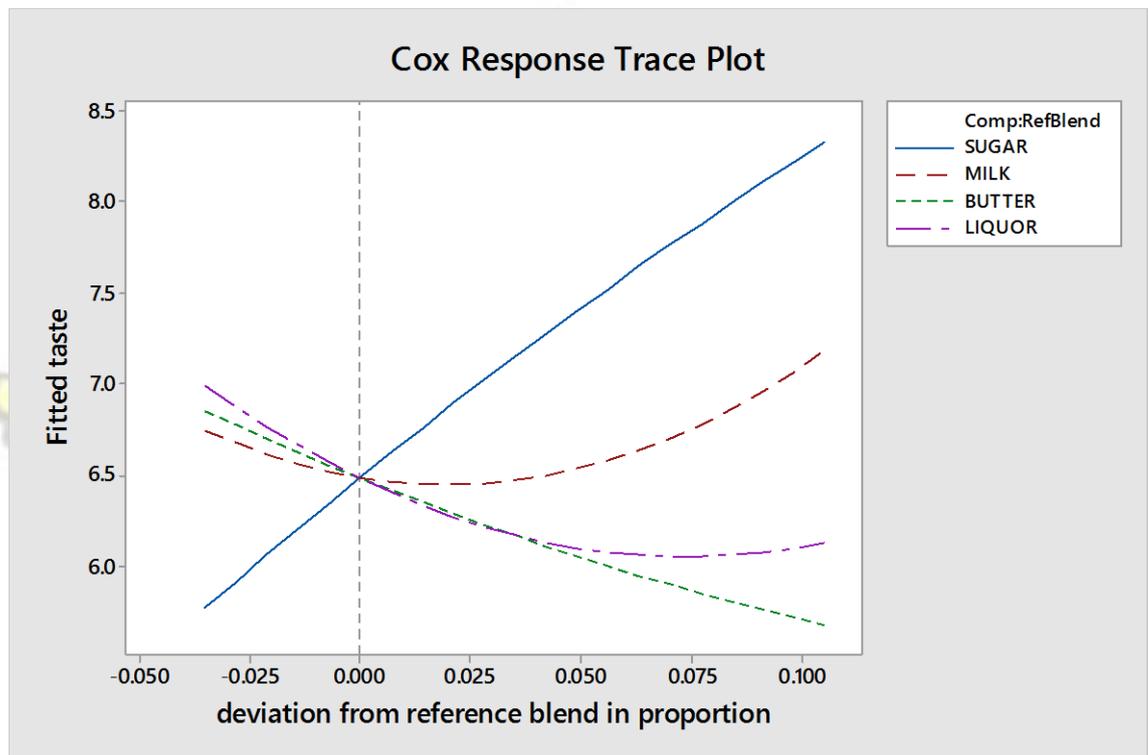


Figure 6: Cox Response Trace plot for taste

4.6 Effect of component on aftertaste

The lingering taste after eating foods is an important attribute for judging the acceptability of foods. In this study, high mean score of the recipe meant low lingering after-taste.

The minimum and maximum statistical values for the aftertaste were 5.2 (neither liked nor disliked) to 8.43 (liked very much). The magnitudes of the coefficients for the four pure mixtures indicated that milk (119) produced a milk chocolate with higher likeness

for lower lingering aftertaste than the other attributes. The regression model for the aftertaste was reasonably accurate ($R^2 = 85.87\%$). None of the pure or the two-blend mixture could be judged as significant.

The Cox Response plot (Figure 7) for aftertaste had a quite similar plot as the taste.

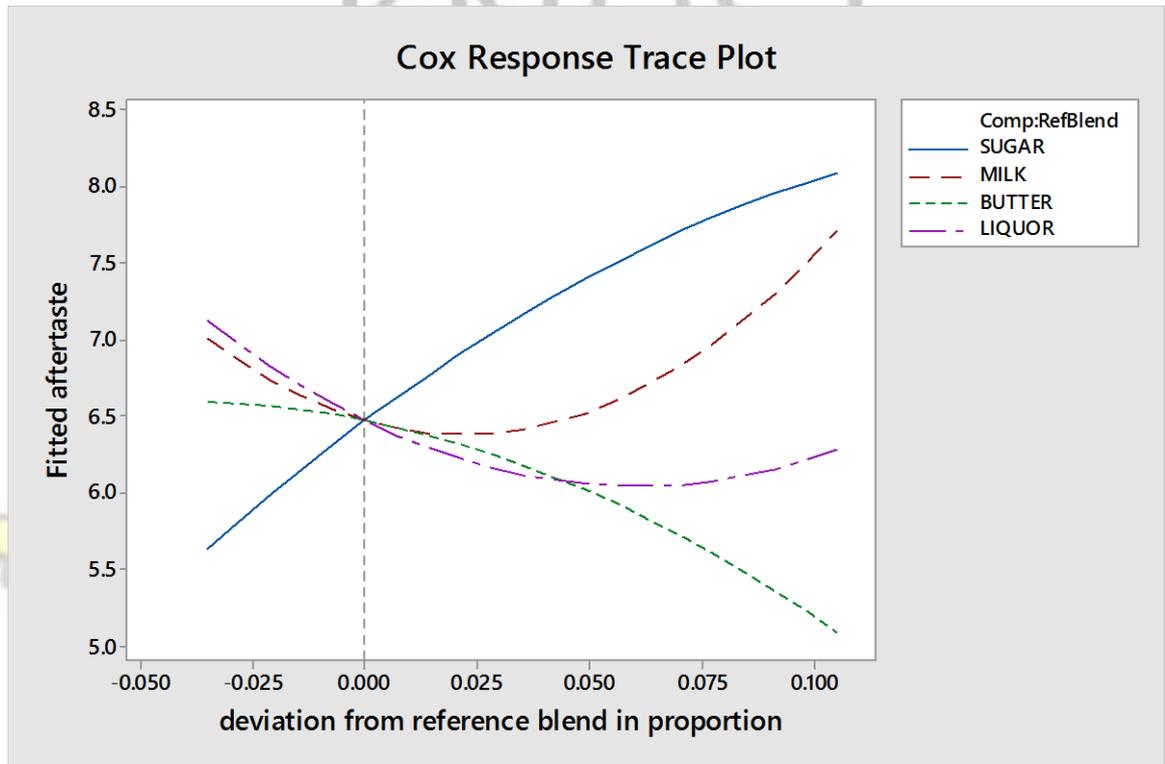


Figure 7: Cox Response Trace plot for aftertaste

4.7 Effect of component on overall acceptability

The minimum and maximum statistical values for the appearance were 5.66 (neither liked nor disliked) to 7.16 (liked moderately).

The magnitudes of the coefficients for the four pure mixtures indicated that cocoa liquor (67.2) produced a milk chocolate with higher likeness for overall acceptability than other compositions. The regression model for overall acceptability was reasonably accurate ($R^2 = 87.62\%$). The milk by liquor mixture is the only two-blend mixture that can be judged as significant ($p = 0.035$) factor influencing the overall acceptability (Table 3, Figure 8).

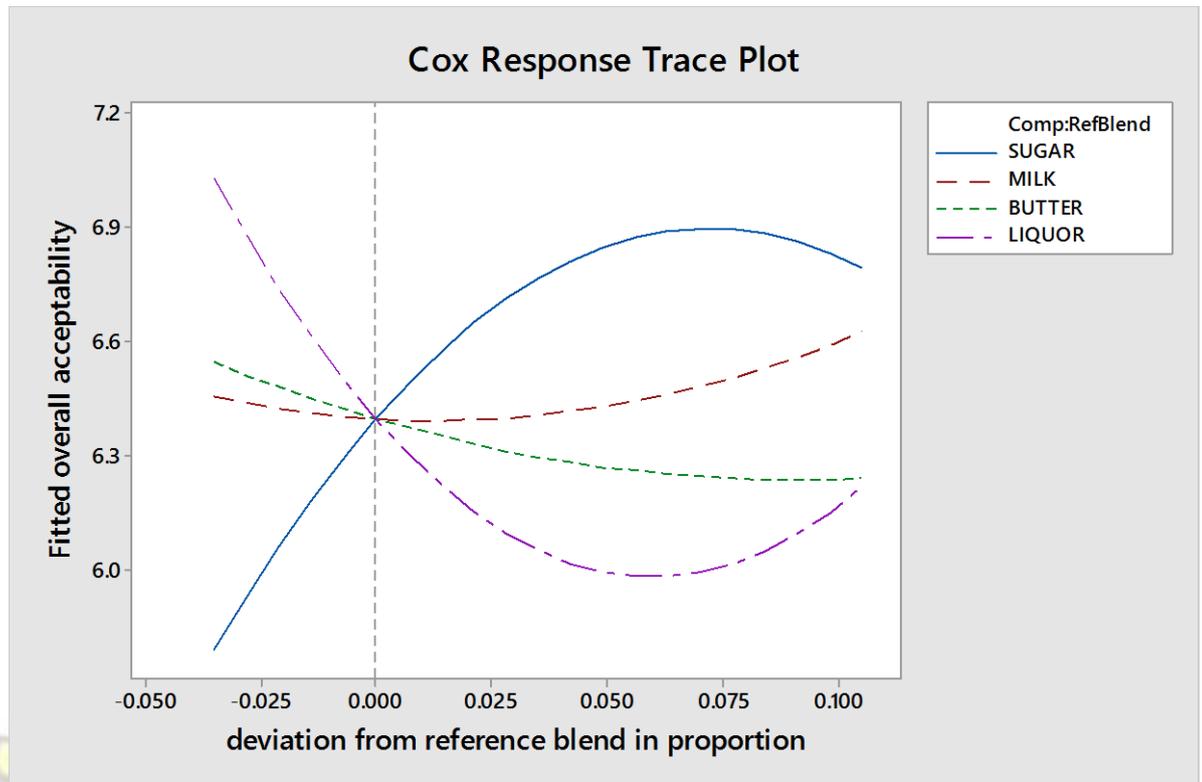


Figure 8: Cox Response Trace plot for Overall acceptability

4.8 Optimal formulation

Optimization in sensory evaluation is defined as a procedure for developing the best possible product in its class (Sidel and Stone, 1983). By holding one attribute constant, Contour plots of the other attributes were overlaid, using MINITAB (version17) and the optimum regions (of the component formulations) where the criteria for all the sensory attributes (appearance, mouthfeel, flavour, taste, aftertaste and overall acceptability) were satisfied. The feasible regions that satisfy optimum response variables settings are illustrated as the white area inside each plot. The shaded areas on the graphical optimization plot refer to responses which do not meet the selection criteria. By holding the milk powder at minimum proposition of 0.18, the optimum region is illustrated in the Figure 9.

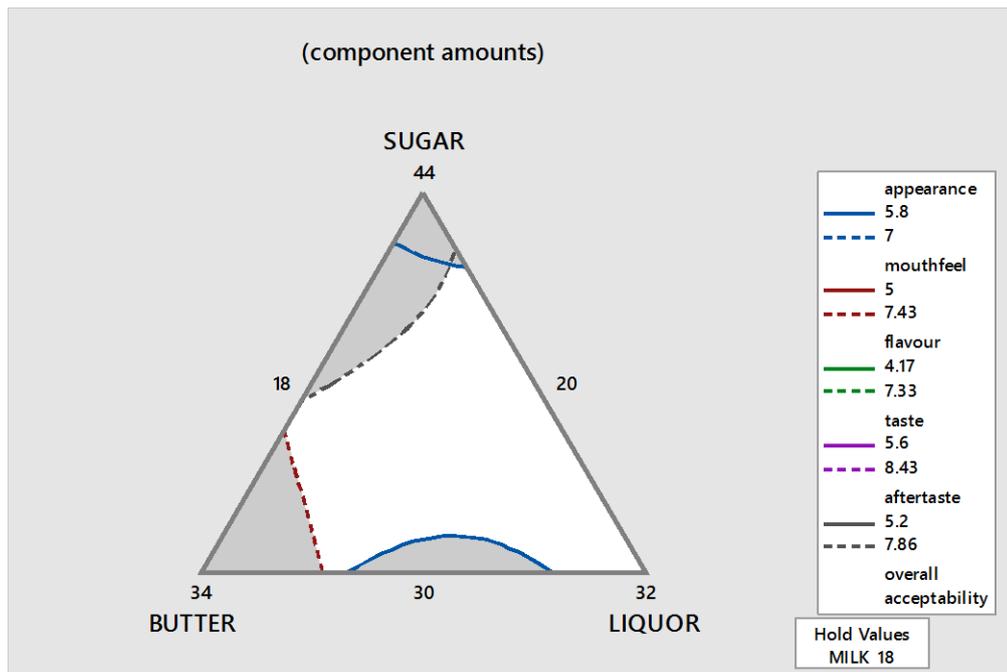


Figure 9: Overlaid contour plot when milk in addition to okra pectin are held constant

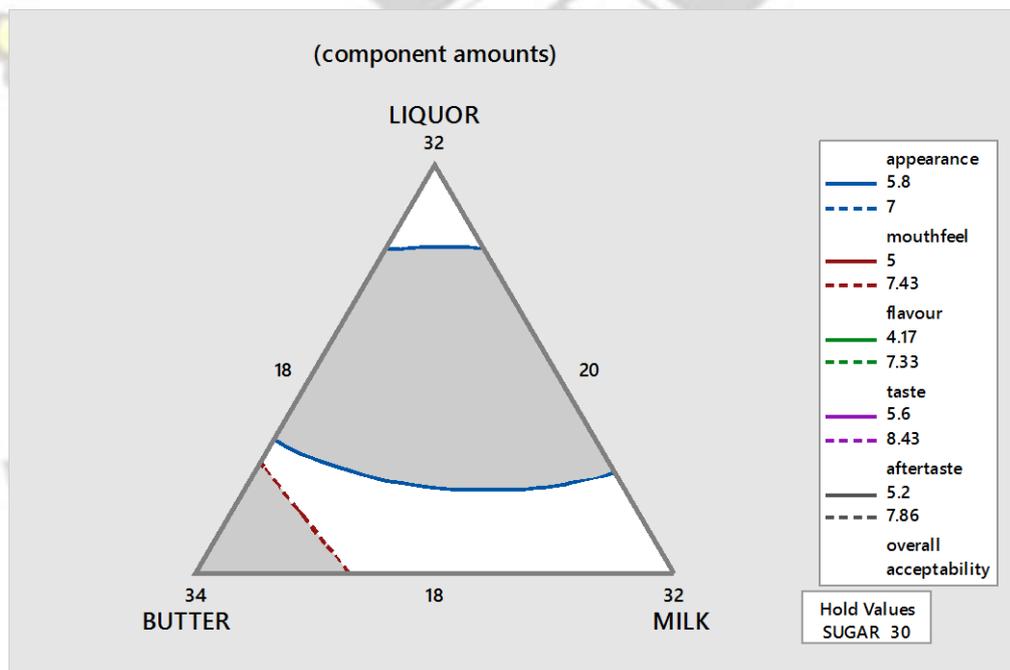


Figure 10: Overlaid contour plot when sugar in addition to okra pectin are held constant

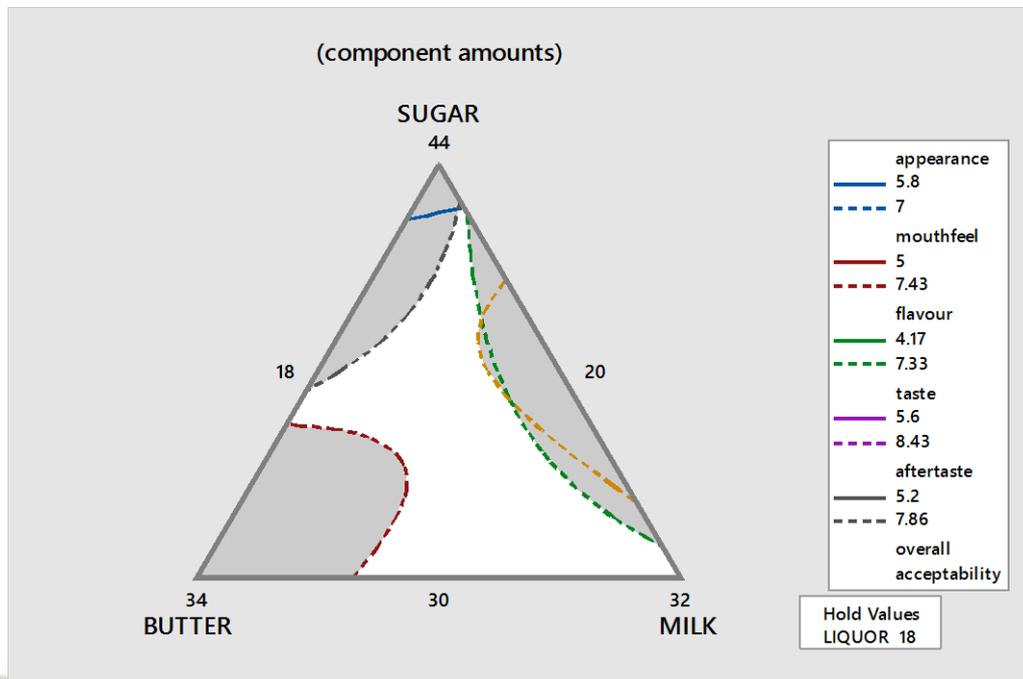


Figure 11: Overlaid contour plot when liquor in addition to okra pectin are held constant

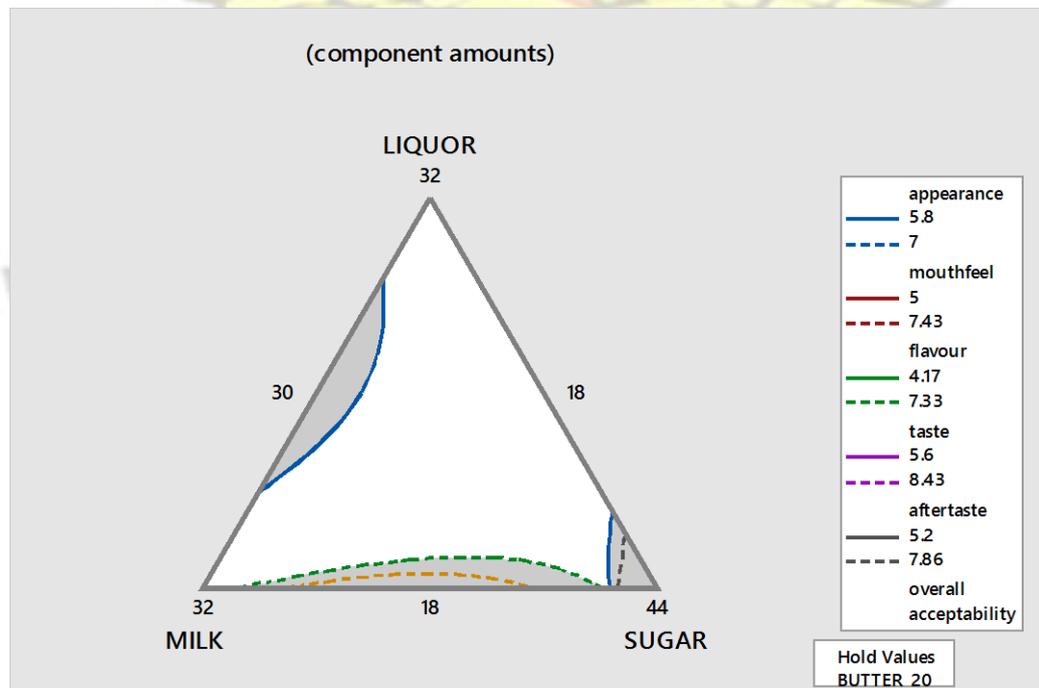


Figure 12: Overlaid contour plot when butter in addition to okra pectin are held constant

4.10 Response optimizer

Optimal settings of the design variables for one response may be far from optimal or even physically impossible for another response. Hence, the response optimization was used to allow for compromise among the various responses. It provided an optimal solution for the input variable combinations and the optimization plot. The optimization plot was interactive. This meant that one could adjust input variable settings on the plot in the Minitab software to search for more desirable solutions.

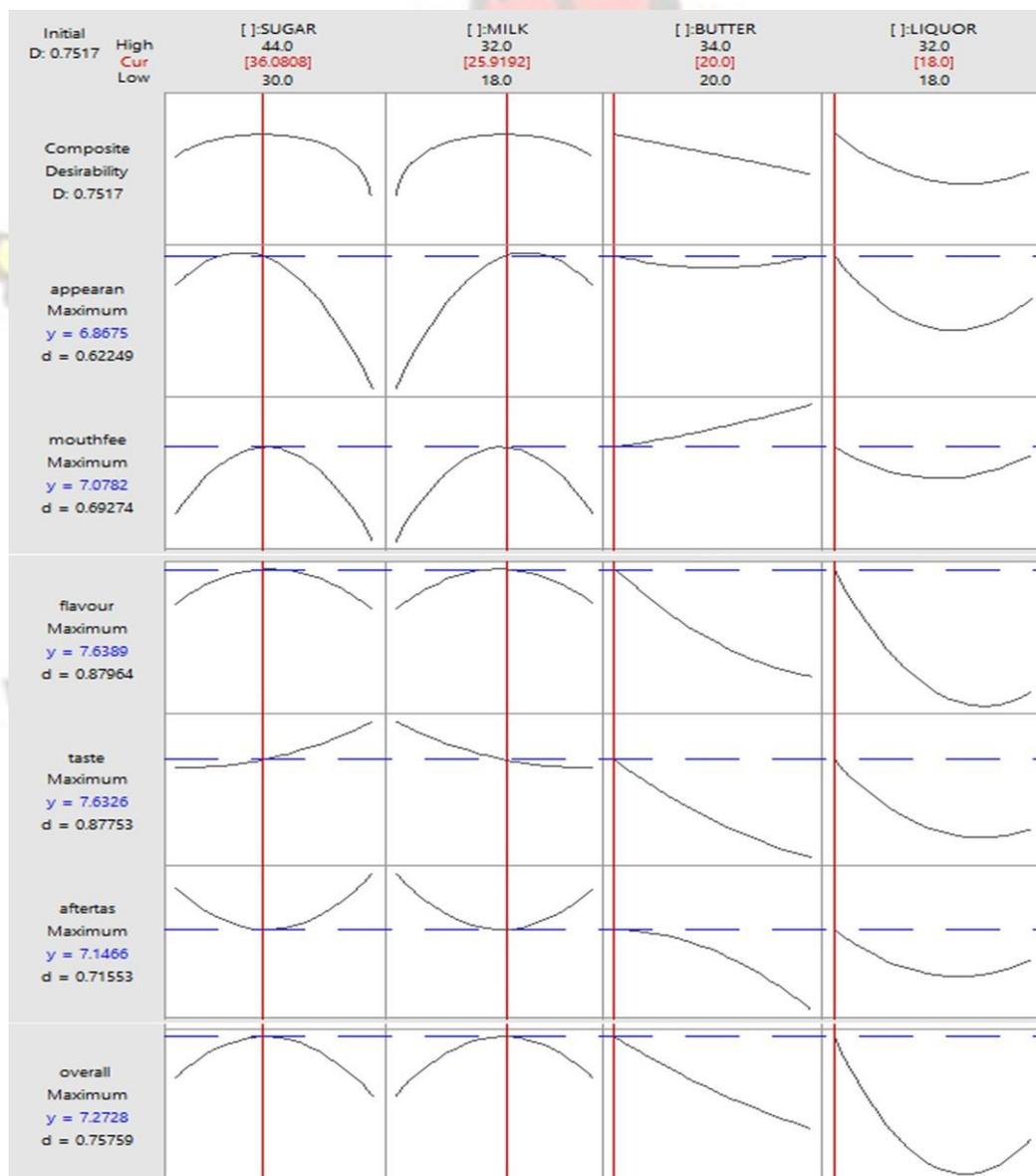


Figure 13: Optimization plot for sensory acceptability

The Minitab response optimizer analysis result is shown in Figure 13. Desirability function for milk chocolate maximizing appearance, mouthfeel, flavour, taste, aftertaste, and overall acceptability based on a 9-point Hedonic scale (1=extremely dislike, 9=like extremely). Lower boundary was set to 5 (neither like nor dislike) and maximum to 8 (like very much). Weight and importance of each Y was set to 1.

Overall desirability (D) was determined to be 0.7517 based on individual d values ranging from 0.62249 to 0.87753 (Figure 13). From these values, the optimal blend was found to be 36.08% sugar, 25.92% milk, 20.00% butter and 18.00% liquor.

The predicted responses at these optimum levels were liked moderately for appearance (6.87), mouthfeel (7.08), aftertaste (7.15), and overall acceptability. Liked very much for flavor (7.64) and taste (7.63).

Datsomor, (2016) indicated that okra pectin added at 0.145% of the total weight of the ingredients produced chocolate with similar flow and yield to standard milk chocolate produced when lecithin was used. The addition of lecithin reduces the viscosity of chocolate dramatically and this brings about the reductions in the fat content without changing the flow properties. It is possible therefore, to reduce the fat content with approximately 4% without changing the flow properties by adding 0.4% lecithin to the chocolate. It was suggested that at okra pectin at 0.145% with similar flow properties may have have similar effect. Any value above this may bring about thickening effect and off flavor.

Just by considering the sensory evaluation in this study, the acceptable chocolate developed had its butter at the minimum region of 20% (Figure 13). So was the cocoa liquor (18%), which contains more than half of butter.

Therefore, if manufactures can produce milk chocolate at these low levels of butter and liquor using the okra pectin, and still be acceptable in terms of appearance taste and smoothness, then this will go a long way to increase the marketability of the product, hence greatly enhancing the profit margin.

4.11 Model Validation

To determine whether the predictive models obtained were sufficient and adequate, two formulations that fell within all the optimum regions and two formulations outside the region were selected for the validation process.

Table 7: Formulations for validation experiments

SAMPLES	SUGAR	MILK	BUTTER	LIQUOR
OR1	30.00	32.00	20.00	18.00
OR2	30.00	18.00	20.00	32.00
OOR1	30.00	18.00	34.00	18.00
OOR2	44.00	18.00	20.00	18.00

OR1 and OR2 are samples in the Optimum Region

OOR1 and OOR2 are samples Outside the Optimum Region

Okra pectin=constant

Formulations in Table 7 were subjected to sensory evaluation by the same panel of judges.

Results from the validation study (Table 8) indicated that the ratings for Appearance,

mouthfeel, flavor, taste, aftertaste and overall acceptability compared well with the predicted ratings. The validation results indicate a good agreement between the observed and predicted ratings

Table 8: Predicted and validated ratings for sensory attributes within and outside optimum region

	Appearance	Mouthfeel	Flavour	Taste	Aftertaste	Overall Acceptability
Predicted Ratings	5.29-7.00	5.00-8.00	4.17-7.77	5.6-8.43	5.20-8.43	5.66-7.16
Optimum Region 1	7.0	7.52	7.30	8.00	7.45	7.03
Optimum Region 2	6.64	7.12	7.43	7.67	6.98	7.00
Outside Optimum Region 1	4.9	5.00	6.00	5.60	6.40	7.00
Outside Optimum Region 2	5.1	6.45	4.97	5.55	5.87	6.09

CHAPTER FIVE

CONCLUSION AND RECOMMENDATION

5.1 Conclusion

The optimal proportion of the ingredients in the milk chocolate based on sensory characteristics were found to be 36.08% sugar, 25.92% milk, 20.00% butter and 18.00% liquor at okra pectin level of 0.145%. At these levels the chocolate may be liked moderately in terms of its overall acceptability.

5.2 Recommendation

- Future research should be conducted to determine the influence of the okra pectin on the optimization of processing parameters of the milk chocolate when the ball mill is used. The following could be studied from the process; moisture, particle size, yield flow and casson viscosity.
- To help in the commercialization of the okra pectin in chocolate, future research should focus on the ease of extraction and purification of the pectin.
- Determine the rheological properties of chocolate developed with okra pectin as an emulsifier.
- The optimum recipe should be subjected to an accelerated shelf-life, where the chocolate will be subjected to temperature cycling regimes. This will help in the study of fat bloom during storage.

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APPENDICES

APPENDIX 1: QUESTIONNAIRE FOR MILK CHOCOLATE

NAME.....

DATE:.....

You have received three (3) different samples of milk chocolate. Please evaluate them from left to right in the order as indicated. Rinse your mouth with some of the water provided and wait for 1 minute before evaluating the next sample.

On the scale of 1 to 9 (see below for key), rate the samples according to the attributes specified.

Samples Order:

1 Appearance:

1	2	3	4	5	6	7	8	9
---	---	---	---	---	---	---	---	---

2: Mouth feel:

1	2	3	4	5	6	7	8	9
---	---	---	---	---	---	---	---	---

3: Flavor:

1	2	3	4	5	6	7	8	9
---	---	---	---	---	---	---	---	---

4: Taste:

1	2	3	4	5	6	7	8	9
---	---	---	---	---	---	---	---	---

5: After-Taste:

1	2	3	4	5	6	7	8	9
---	---	---	---	---	---	---	---	---

6: Overall Acceptability:

1	2	3	4	5	6	7	8	9
---	---	---	---	---	---	---	---	---

Comments.....
.....

KEY

- | | |
|------------------------|------------------------------|
| 1 – Dislike extremely | 5 – Neither like nor dislike |
| 2 – Dislike very much | 6 – Like slightly |
| 3 – Dislike moderately | 7 – Like moderately |
| 4 – Dislike slightly | 8 – Like very much |
| | 9 – Like extremely |

APPENDIX 2: BALANCED INCOMPLETE BLOCK DESIGN

block	Rep 1			block	Rep 2			block	Rep 3		
(1)	<u>1</u>	<u>2</u>	<u>3</u>	(6)	<u>1</u>	<u>4</u>	<u>5</u>	(11)	<u>1</u>	<u>6</u>	<u>7</u>
(2)	<u>4</u>	<u>8</u>	<u>12</u>	(7)	<u>2</u>	<u>8</u>	<u>10</u>	(12)	<u>2</u>	<u>9</u>	<u>11</u>
(3)	<u>5</u>	<u>10</u>	<u>15</u>	(8)	<u>3</u>	<u>13</u>	<u>14</u>	(13)	<u>3</u>	<u>12</u>	<u>15</u>
(4)	<u>6</u>	<u>11</u>	<u>13</u>	(9)	<u>6</u>	<u>9</u>	<u>15</u>	(14)	<u>4</u>	<u>10</u>	<u>14</u>
(5)	<u>7</u>	<u>9</u>	<u>14</u>	(10)	<u>7</u>	<u>11</u>	<u>12</u>	(15)	<u>5</u>	<u>8</u>	<u>13</u>

block	Rep 4			block	Rep 5			block	Rep 6		
(16)	<u>1</u>	<u>8</u>	<u>9</u>	(21)	<u>1</u>	<u>10</u>	<u>11</u>	(26)	<u>1</u>	<u>12</u>	<u>13</u>
(17)	<u>2</u>	<u>13</u>	<u>15</u>	(22)	<u>2</u>	<u>12</u>	<u>14</u>	(27)	<u>2</u>	<u>5</u>	<u>7</u>
(18)	<u>3</u>	<u>4</u>	<u>7</u>	(23)	<u>3</u>	<u>5</u>	<u>6</u>	(28)	<u>3</u>	<u>9</u>	<u>10</u>
(19)	<u>5</u>	<u>11</u>	<u>14</u>	(24)	<u>4</u>	<u>9</u>	<u>13</u>	(29)	<u>4</u>	<u>11</u>	<u>15</u>
(20)	<u>6</u>	<u>10</u>	<u>12</u>	(25)	<u>7</u>	<u>8</u>	<u>15</u>	(30)	<u>6</u>	<u>8</u>	<u>14</u>

block	Rep 7		
(31)	<u>1</u>	<u>14</u>	<u>15</u>
(32)	<u>2</u>	<u>4</u>	<u>6</u>
(33)	<u>3</u>	<u>8</u>	<u>11</u>
(34)	<u>5</u>	<u>9</u>	<u>12</u>
(35)	<u>7</u>	<u>10</u>	<u>13</u>

APPENDIX 3: SIMPLEX LATTICE DESIGN

Components: 4 Design points: 15
 Process variables: 0 Lattice degree: 2

Mixture total: 100.00000

Number of Boundaries for Each Dimension

Point Type 1 2 3 0
 Dimension 0 1 2 3
 Number 4 6 4 1

KNUST

Number of Design Points for Each Type

Point Type 1 2 3 4 0 -1
 Distinct 4 6 0 0 1 4
 Replicates 1 1 0 0 1 1
 Total number 4 6 0 0 1 4

Bounds of Mixture Components

Comp	Amount		Proportion		Pseudocomponent	
	Lower	Upper	Lower	Upper	Lower	Upper
A	30.000	44.000	0.30000	0.44000	0.00000	1.00000
B	18.000	32.000	0.18000	0.32000	0.00000	1.00000
C	20.000	34.000	0.20000	0.34000	0.00000	1.00000
D	18.000	32.000	0.18000	0.32000	0.00000	1.00000

APPENDIX 4: ANALYSIS OF VARIANCE

appearance

Source of Variation	SS	df	MS	F	P-value	F crit
Between Groups	22.16786	14	1.583418	0.477658	0.939536	1.803206
Within Groups	298.3464	90	3.31496			
Total	320.5143	104				

mouthfeel

Source of Variation	SS	df	MS	F	P-value	F crit
Between Groups	57.8873	14	4.134807	1.607859	0.092123	1.803206

Within Groups	231.446	90	2.571623
<u>Total</u>	<u>289.3333</u>	<u>104</u>	

flavour

<i>Source of Variation</i>	<i>SS</i>	<i>df</i>	<i>MS</i>	<i>F</i>	<i>P-value</i>	<i>F crit</i>
Between Groups	87.73333	14	6.266667	1.581984	0.099645	1.803206
Within Groups	356.5143	90	3.96127			
<u>Total</u>	<u>444.2476</u>	<u>104</u>				

Total

taste

<i>Source of Variation</i>	<i>SS</i>	<i>df</i>	<i>MS</i>	<i>F</i>	<i>P-value</i>	<i>F crit</i>
Between Groups	59.65633	14	4.261166	1.545232	0.112374	1.808472
Within Groups	237.1556	86	2.757623			
<u>Total</u>	<u>296.8119</u>	<u>100</u>				

aftertaste

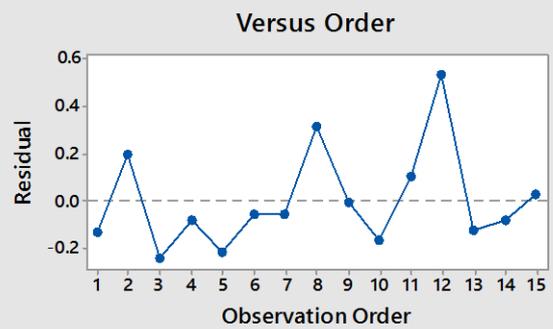
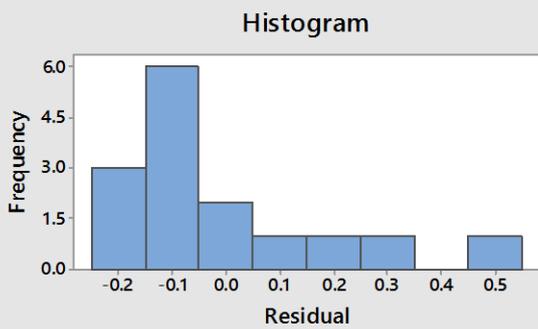
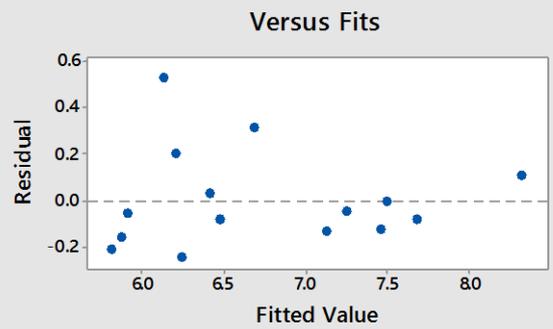
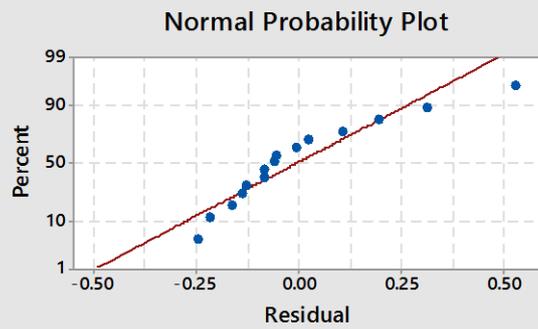
<i>Source of Variation</i>	<i>SS</i>	<i>df</i>	<i>MS</i>	<i>F</i>	<i>P-value</i>	<i>F crit</i>
Between Groups	77.29105	14	5.520789	2.149594	0.01616	1.805778
Within Groups	226.0099	88	2.568295			
<u>Total</u>	<u>303.301</u>	<u>102</u>				

overall acceptability

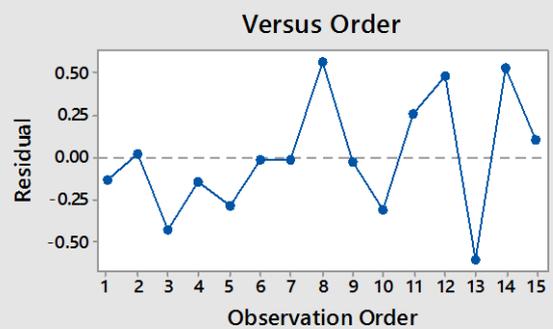
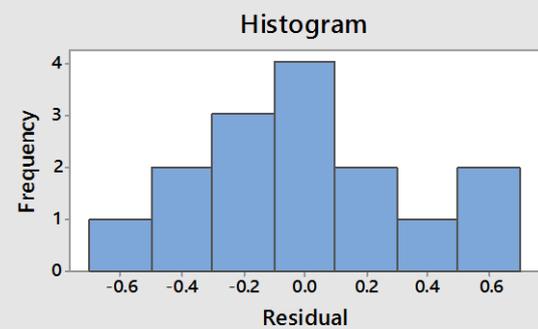
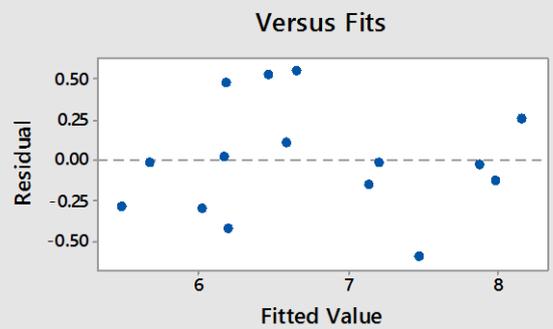
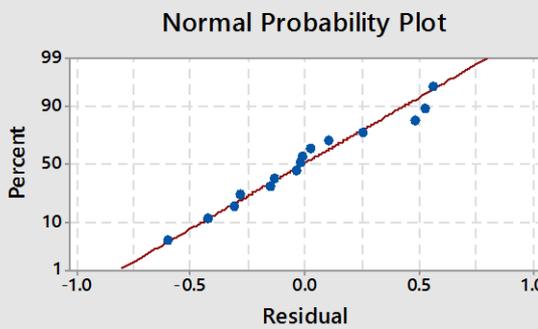
<i>Source of Variation</i>	<i>SS</i>	<i>df</i>	<i>MS</i>	<i>F</i>	<i>P-value</i>	<i>F crit</i>
Between Groups	69.48394	14	4.963138	2.178287	0.016501	1.827755
Within Groups	168.606	74	2.278459			
<u>Total</u>	<u>238.0899</u>	<u>88</u>				

APPENDIX 5: RESIDUE PLOTS

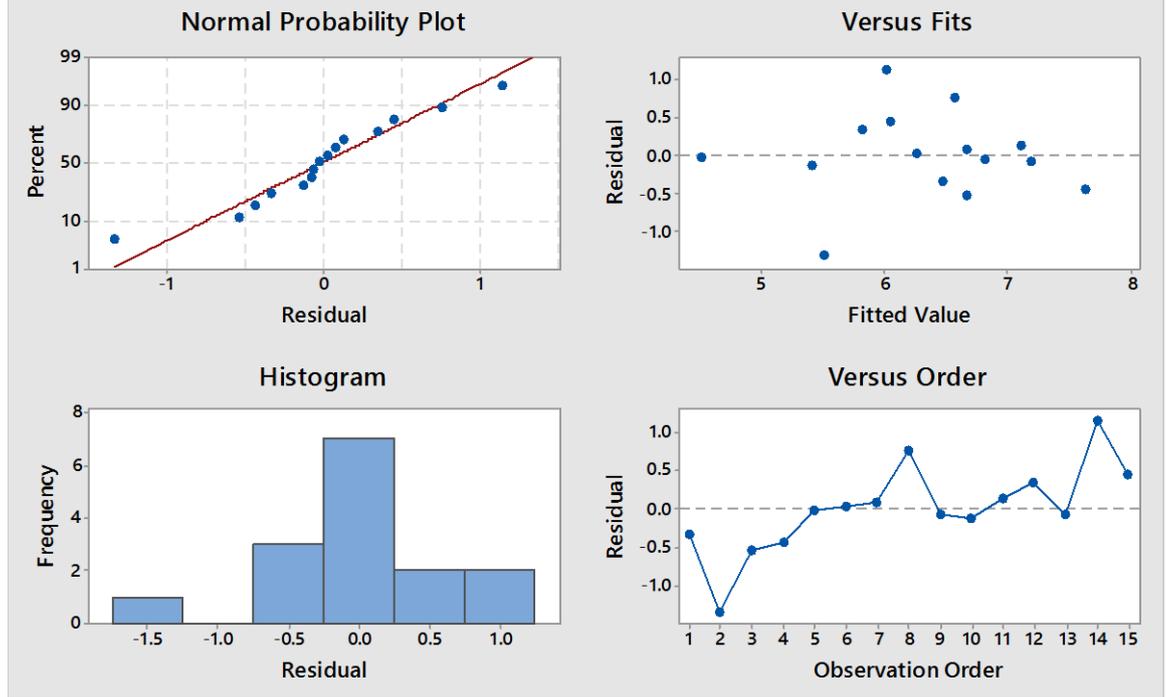
Residual Plots for taste



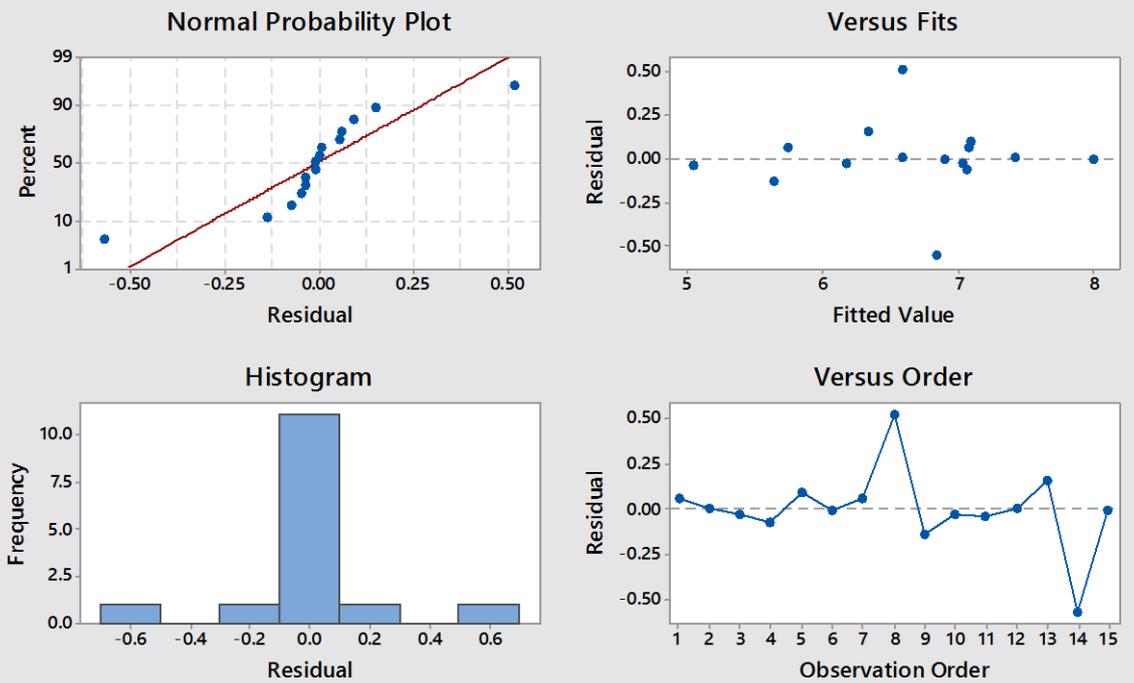
Residual Plots for aftertaste



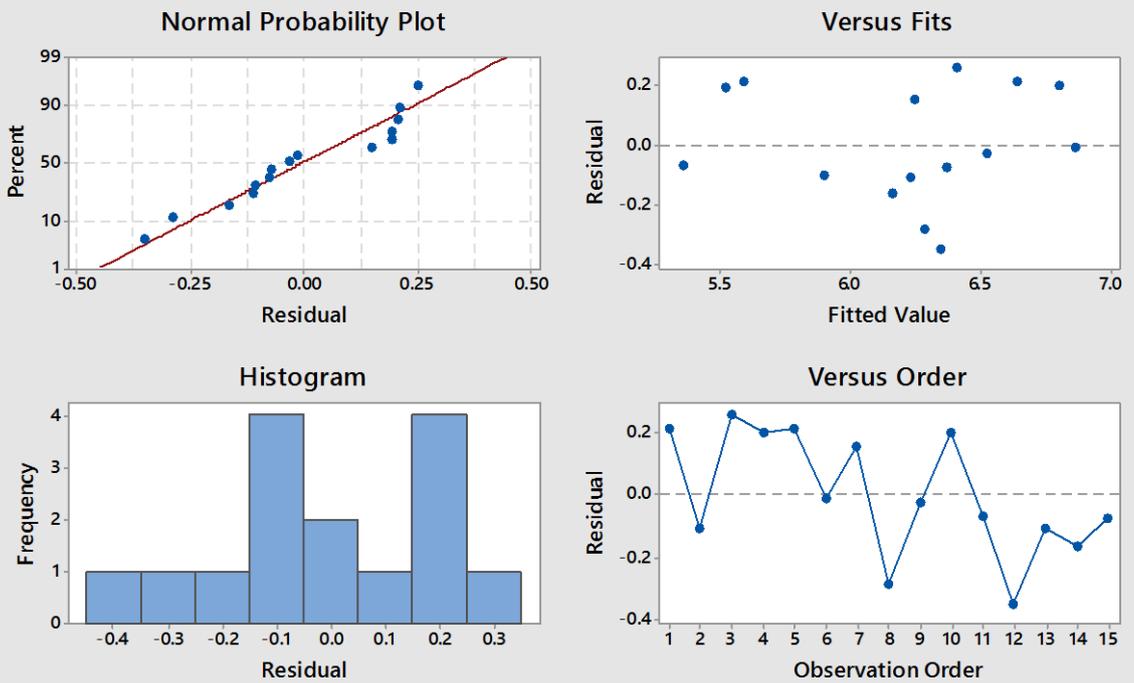
Residual Plots for flavour

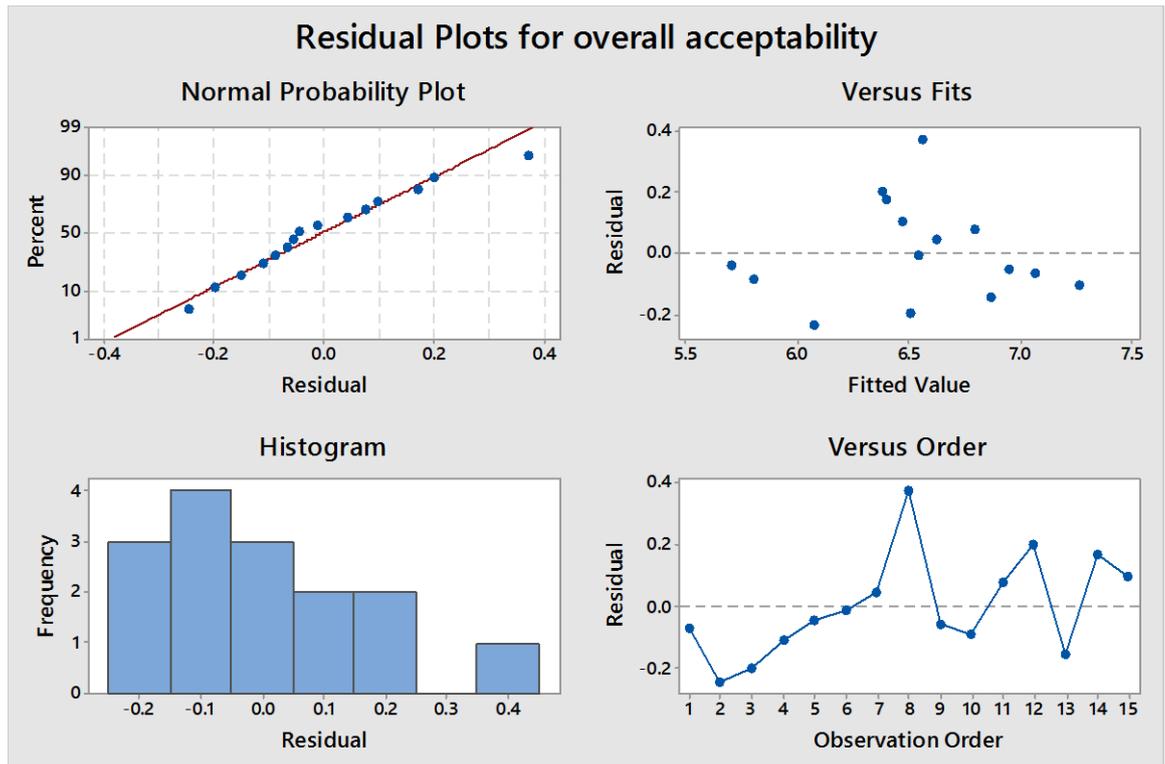


Residual Plots for mouthfeel



Residual Plots for appearance





APPENDIX 6: REGRESSION FOR MIXTURES

Appendix 6a: appearance versus SUGAR, MILK, BUTTER, LIQUOR

Estimated Regression Coefficients for appearance (component proportions)

Term	Coef	SE Coef	T	P	VIF
SUGAR	-62.7	19.97	*	*	6574
MILK	6.3	28.61	*	*	5680
BUTTER	31.4	27.22	*	*	6105
LIQUOR	64.8	28.61	*	*	5680
SUGAR*MILK	175.9	76.04	2.31	0.069	4409
SUGAR*BUTTER	108.6	76.04	1.43	0.212	5254
SUGAR*LIQUOR	78.5	76.04	1.03	0.349	4409
MILK*BUTTER	-58.6	76.04	-0.77	0.475	2168
MILK*LIQUOR	-189.9	76.04	-2.50	0.055	1816
BUTTER*LIQUOR	-210.4	76.04	-2.77	0.039	2168

S = 0.321933 PRESS = 17.5040
R-Sq = 84.64% R-Sq(pred) = 0.00% R-Sq(adj) = 56.98%

Analysis of Variance for appearance (component proportions)

Source	DF	Seq SS	Adj SS	Adj MS	F	P
Regression	9	2.85447	2.85447	0.317163	3.06	0.115

Linear	3	0.67548	2.08565	0.695217	6.71	0.033
Quadratic	6	2.17899	2.17899	0.363165	3.50	0.095
SUGAR*MILK	1	0.44473	0.55430	0.554296	5.35	0.069
SUGAR*BUTTER	1	0.18277	0.21157	0.211572	2.04	0.212
SUGAR*LIQUOR	1	0.19695	0.11036	0.110362	1.06	0.349
MILK*BUTTER	1	0.01645	0.06162	0.061625	0.59	0.475
MILK*LIQUOR	1	0.54430	0.64626	0.646256	6.24	0.055
BUTTER*LIQUOR	1	0.79380	0.79380	0.793796	7.66	0.039
Residual Error	5	0.51820	0.51820	0.103641		
Total	14	3.37267				

Estimated Regression Coefficients for appearance (component amounts)

Term	Coef
SUGAR	-0.627050
MILK	0.0630996
BUTTER	0.314392
LIQUOR	0.647847
SUGAR*MILK	0.0175854
SUGAR*BUTTER	0.0108645
SUGAR*LIQUOR	0.00784676
MILK*BUTTER	-0.00586353
MILK*LIQUOR	-0.0189882
BUTTER*LIQUOR	-0.0210443

Unusual Observations for appearance

Obs	StdOrder	appearance	Fit	SE Fit	Residual	St Resid
3	3	6.667	6.412	0.298	0.254	2.10R

R denotes an observation with a large standardized residual.

Appendix 6b: mouthfeel versus SUGAR, MILK, BUTTER, LIQUOR

Estimated Regression Coefficients for mouthfeel (component proportions)

Term	Coef	SE Coef	T	P	VIF
SUGAR	-58.36	22.49	*	*	6574
MILK	-87.40	32.23	*	*	5680
BUTTER	11.54	30.66	*	*	6105
LIQUOR	64.98	32.23	*	*	5680
SUGAR*MILK	352.77	85.66	4.12	0.009	4409
SUGAR*BUTTER	113.78	85.66	1.33	0.241	5254
SUGAR*LIQUOR	-48.11	85.66	-0.56	0.599	4409
MILK*BUTTER	42.97	85.66	0.50	0.637	2168
MILK*LIQUOR	-19.79	85.66	-0.23	0.826	1816
BUTTER*LIQUOR	-72.20	85.66	-0.84	0.438	2168

S = 0.362642 PRESS = 10.2843
R-Sq = 92.42% R-Sq(pred) = 0.00% R-Sq(adj) = 78.78%

Analysis of Variance for mouthfeel (component proportions)

Source	DF	Seq SS	Adj SS	Adj MS	F	P
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Regression	9	8.01777	8.01777	0.89086	6.77	0.024
Linear	3	5.49042	2.00106	0.66702	5.07	0.056
Quadratic	6	2.52735	2.52735	0.42123	3.20	0.111
SUGAR*MILK	1	2.10843	2.23065	2.23065	16.96	0.009
SUGAR*BUTTER	1	0.25360	0.23203	0.23203	1.76	0.241
SUGAR*LIQUOR	1	0.02378	0.04148	0.04148	0.32	0.599
MILK*BUTTER	1	0.04444	0.03309	0.03309	0.25	0.637
MILK*LIQUOR	1	0.00366	0.00702	0.00702	0.05	0.826
BUTTER*LIQUOR	1	0.09343	0.09343	0.09343	0.71	0.438
Error	5	0.65755	0.65755	0.13151		
Total	14	8.67532				

Estimated Regression Coefficients for mouthfeel (component amounts)

Term	Coef
SUGAR	-0.583558
MILK	-0.873977
BUTTER	0.115387
LIQUOR	0.649821
SUGAR*MILK	0.0352774
SUGAR*BUTTER	0.0113776
SUGAR*LIQUOR	-0.00481087
MILK*BUTTER	0.00429693
MILK*LIQUOR	-0.00197896
BUTTER*LIQUOR	-0.00721996

Appendix 6c: flavour versus SUGAR, MILK, BUTTER, LIQUOR

Estimated Regression Coefficients for flavour (component proportions)

Term	Coef	SE Coef	T	P	VIF
SUGAR	-19.5	59.88	*	*	6574
MILK	10.9	85.80	*	*	5680
BUTTER	61.5	81.62	*	*	6105
LIQUOR	91.4	85.80	*	*	5680
SUGAR*MILK	98.6	228.03	0.43	0.684	4409
SUGAR*BUTTER	-42.2	228.03	-0.18	0.861	5254
SUGAR*LIQUOR	17.0	228.03	0.07	0.943	4409
MILK*BUTTER	-11.9	228.03	-0.05	0.960	2168
MILK*LIQUOR	-247.9	228.03	-1.09	0.327	1816
BUTTER*LIQUOR	-333.3	228.03	-1.46	0.204	2168

S = 0.965425 PRESS = 126.278
R-Sq = 65.60% R-Sq(pred) = 0.00% R-Sq(adj) = 3.67%

Analysis of Variance for flavour (component proportions)

Source	DF	Seq SS	Adj SS	Adj MS	F	P
Regression	9	8.8859	8.88589	0.98732	1.06	0.502
Linear	3	5.7550	1.50121	0.50040	0.54	0.677
Quadratic	6	3.1309	3.13093	0.52182	0.56	0.750
SUGAR*MILK	1	0.1197	0.17418	0.17418	0.19	0.684
SUGAR*BUTTER	1	0.0430	0.03189	0.03189	0.03	0.861
SUGAR*LIQUOR	1	0.0698	0.00517	0.00517	0.01	0.943
MILK*BUTTER	1	0.0157	0.00254	0.00254	0.00	0.960

MILK*LIQUOR	1	0.8916	1.10174	1.10174	1.18	0.327
BUTTER*LIQUOR	1	1.9911	1.99107	1.99107	2.14	0.204
Residual Error	5	4.6602	4.66022	0.93204		
Total	14	13.5461				

Estimated Regression Coefficients for flavour (component amounts)

Term	Coef
SUGAR	-0.195022
MILK	0.108871
BUTTER	0.615020
LIQUOR	0.914291
SUGAR*MILK	0.00985789
SUGAR*BUTTER	-0.00421812
SUGAR*LIQUOR	0.00169910
MILK*BUTTER	-0.00119078
MILK*LIQUOR	-0.0247925
BUTTER*LIQUOR	-0.0333292

Unusual Observations for flavour

Obs	StdOrder	flavour	Fit	SE Fit	Residual	St Resid
15	15	6.500	6.050	0.941	0.450	2.07R

R denotes an observation with a large standardized residual.

Appendix 6d: taste versus SUGAR, MILK, BUTTER, LIQUOR

Estimated Regression Coefficients for taste (component proportions)

Term	Coef	SE Coef	T	P	VIF
SUGAR	10.5	21.75	*	*	6574
MILK	65.5	31.17	*	*	5680
BUTTER	10.1	29.65	*	*	6105
LIQUOR	46.1	31.17	*	*	5680
SUGAR*MILK	-47.0	82.83	-0.57	0.595	4409
SUGAR*BUTTER	3.7	82.83	0.04	0.966	5254
SUGAR*LIQUOR	-23.8	82.83	-0.29	0.785	4409
MILK*BUTTER	-94.4	82.83	-1.14	0.306	2168
MILK*LIQUOR	-221.1	82.83	-2.67	0.044	1816
BUTTER*LIQUOR	-71.3	82.83	-0.86	0.429	2168

S = 0.350669 PRESS = 14.7764
R-Sq = 93.07% R-Sq(pred) = 0.00% R-Sq(adj) = 80.59%

Analysis of Variance for taste (component proportions)

Source	DF	Seq SS	Adj SS	Adj MS	F	P
Regression	9	8.25500	8.25500	0.917222	7.46	0.020
Linear	3	7.20900	0.35813	0.119375	0.97	0.476
Quadratic	6	1.04600	1.04600	0.174334	1.42	0.359

SUGAR*MILK	1	0.01504	0.03956	0.039558	0.32	0.595
SUGAR*BUTTER	1	0.00090	0.00025	0.000247	0.00	0.966
SUGAR*LIQUOR	1	0.00233	0.01017	0.010175	0.08	0.785
MILK*BUTTER	1	0.09823	0.15988	0.159877	1.30	0.306
MILK*LIQUOR	1	0.83840	0.87626	0.876258	7.13	0.044
BUTTER*LIQUOR	1	0.09110	0.09110	0.091098	0.74	0.429
Residual Error	5	0.61484	0.61484	0.122969		
Total	14	8.86984				

Estimated Regression Coefficients for taste (component amounts)

Term	Coef
SUGAR	0.105340
MILK	0.654620
BUTTER	0.100505
LIQUOR	0.461410
SUGAR*MILK	-0.00469785
SUGAR*BUTTER	0.000370948
SUGAR*LIQUOR	-0.00238253
MILK*BUTTER	-0.00944441
MILK*LIQUOR	-0.0221104
BUTTER*LIQUOR	-0.00712909

Appendix 6e: aftertaste versus SUGAR, MILK, BUTTER, LIQUOR

Estimated Regression Coefficients for aftertaste (component proportions)

Term	Coef	SE Coef	T	P	VIF
SUGAR	-6.6	35.73	*	*	6574
MILK	119.0	51.20	*	*	5680
BUTTER	-39.1	48.70	*	*	6105
LIQUOR	68.9	51.20	*	*	5680
SUGAR*MILK	-179.3	136.07	-1.32	0.245	4409
SUGAR*BUTTER	217.1	136.07	1.60	0.171	5254
SUGAR*LIQUOR	-34.1	136.07	-0.25	0.812	4409
MILK*BUTTER	-119.2	136.07	-0.88	0.421	2168
MILK*LIQUOR	-249.3	136.07	-1.83	0.126	1816
BUTTER*LIQUOR	-132.8	136.07	-0.98	0.374	2168

S = 0.576083 PRESS = 52.6217
R-Sq = 85.87% R-Sq(pred) = 0.00% R-Sq(adj) = 60.43%

Analysis of Variance for aftertaste (component proportions)

Source	DF	Seq SS	Adj SS	Adj MS	F	P
Regression	9	10.0810	10.08098	1.12011	3.38	0.097
Linear	3	7.1455	2.15125	0.71708	2.16	0.211
Quadratic	6	2.9355	2.93552	0.48925	1.47	0.343
SUGAR*MILK	1	0.6084	0.57620	0.57620	1.74	0.245
SUGAR*BUTTER	1	0.8209	0.84511	0.84511	2.55	0.171
SUGAR*LIQUOR	1	0.0055	0.02086	0.02086	0.06	0.812
MILK*BUTTER	1	0.1535	0.25484	0.25484	0.77	0.421
MILK*LIQUOR	1	1.0313	1.11437	1.11437	3.36	0.126
BUTTER*LIQUOR	1	0.3161	0.31607	0.31607	0.95	0.374
Residual Error	5	1.6594	1.65936	0.33187		

Total 14 11.7403

Estimated Regression Coefficients for aftertaste (component amounts)

Term	Coef
SUGAR	-0.0655198
MILK	1.18974
BUTTER	-0.391063
LIQUOR	0.689071
SUGAR*MILK	-0.0179294
SUGAR*BUTTER	0.0217139
SUGAR*LIQUOR	-0.00341185
MILK*BUTTER	-0.0119238
MILK*LIQUOR	-0.0249342
BUTTER*LIQUOR	-0.0132792

Unusual Observations for aftertaste

Obs	StdOrder	aftertaste	Fit	SE Fit	Residual	St Resid
11	11	8.429	8.169	0.561	0.260	2.01R

R denotes an observation with a large standardized residual.

Appendix 6f: overall acceptability versus SUGAR, MILK, BUTTER, LIQUOR

Estimated Regression Coefficients for overall acceptability (component proportions)

Term	Coef	SE Coef	T	P	VIF
SUGAR	-27.3	17.05	*	*	6574
MILK	22.8	24.43	*	*	5680
BUTTER	15.1	23.23	*	*	6105
LIQUOR	67.2	24.43	*	*	5680
SUGAR*MILK	80.2	64.91	1.24	0.272	4409
SUGAR*BUTTER	80.2	64.91	1.24	0.271	5254
SUGAR*LIQUOR	-2.1	64.91	-0.03	0.975	4409
MILK*BUTTER	-48.3	64.91	-0.74	0.491	2168
MILK*LIQUOR	-185.6	64.91	-2.86	0.035	1816
BUTTER*LIQUOR	-164.0	64.91	-2.53	0.053	2168

S = 0.274825 PRESS = 11.2102
 R-Sq = 87.62% R-Sq(pred) = 0.00% R-Sq(adj) = 65.34%

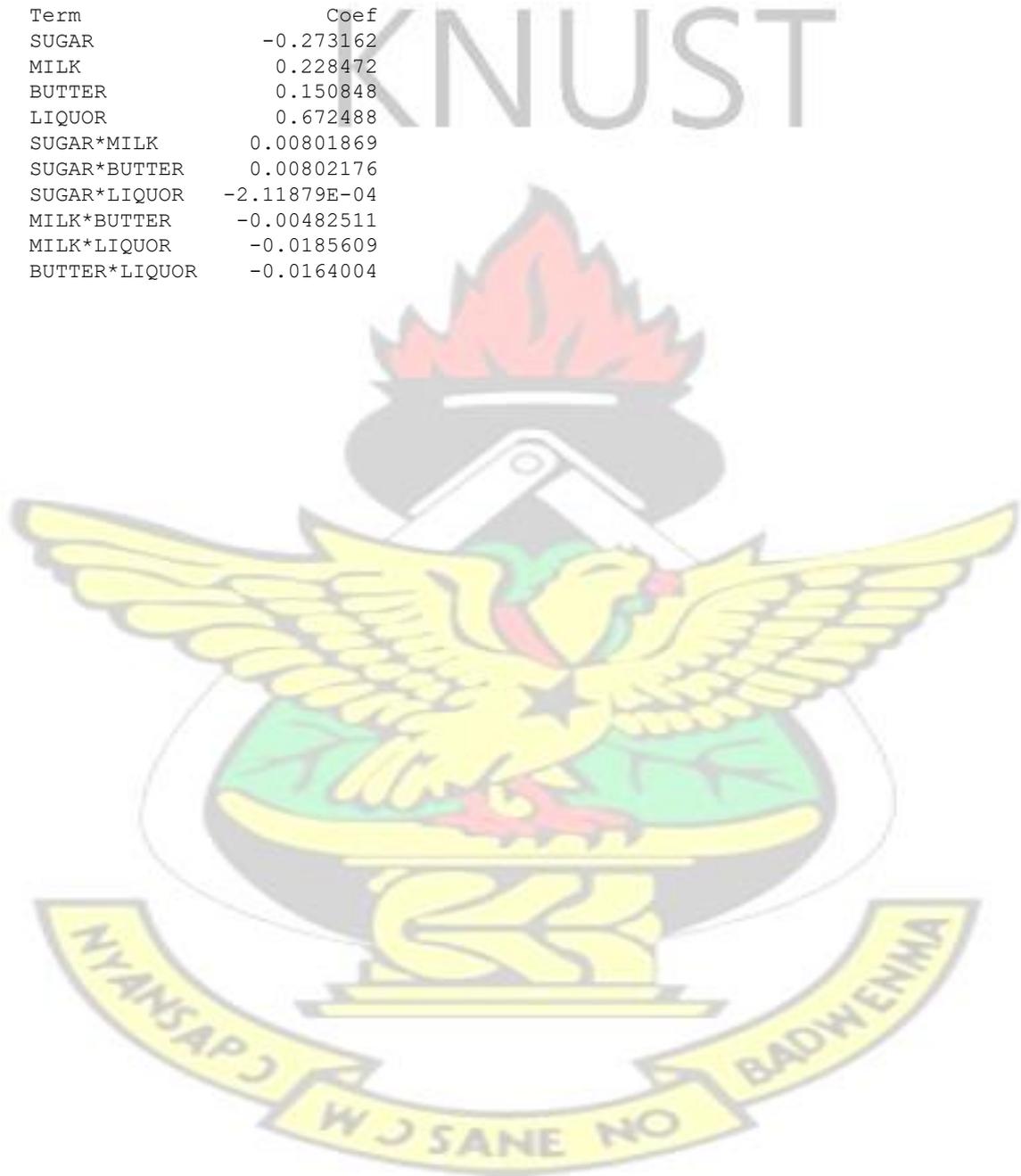
Analysis of Variance for overall acceptability (component proportions)

Source	DF	Seq SS	Adj SS	Adj MS	F	P
Regression	9	2.67317	2.67317	0.297019	3.93	0.073
Linear	3	1.43387	0.94576	0.315255	4.17	0.079
Quadratic	6	1.23930	1.23930	0.206551	2.73	0.145
SUGAR*MILK	1	0.09764	0.11525	0.115251	1.53	0.272
SUGAR*BUTTER	1	0.10238	0.11534	0.115339	1.53	0.271
SUGAR*LIQUOR	1	0.00745	0.00008	0.000080	0.00	0.975
MILK*BUTTER	1	0.00989	0.04173	0.041730	0.55	0.491

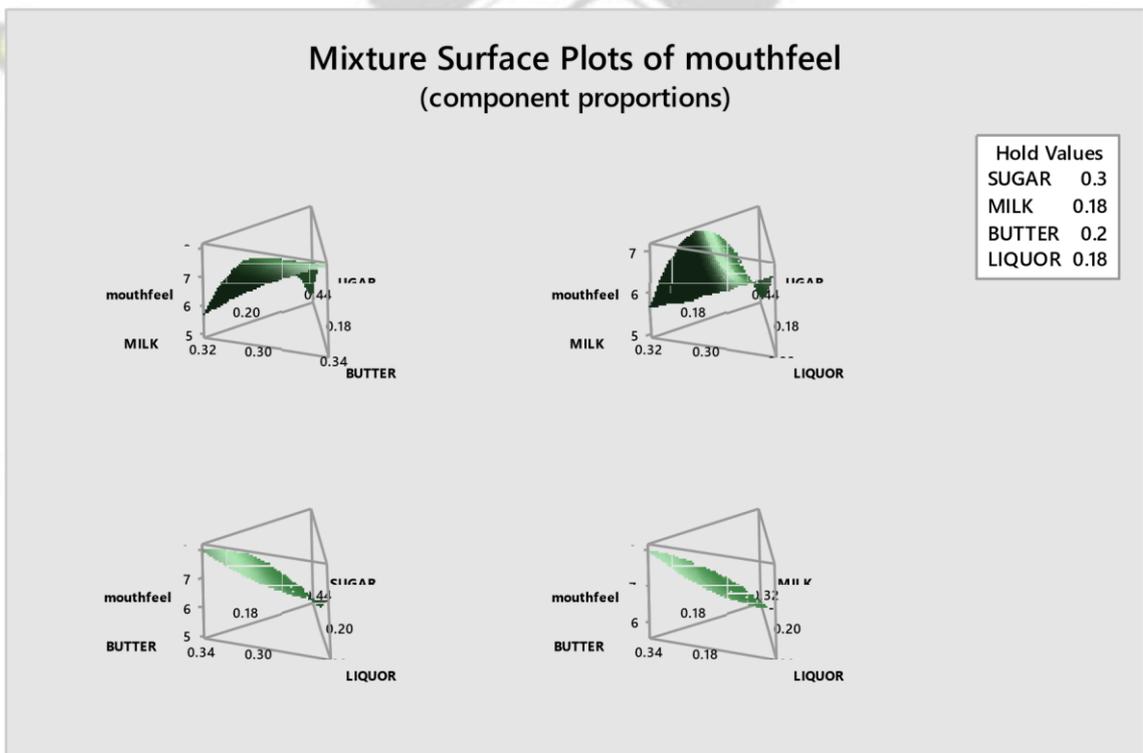
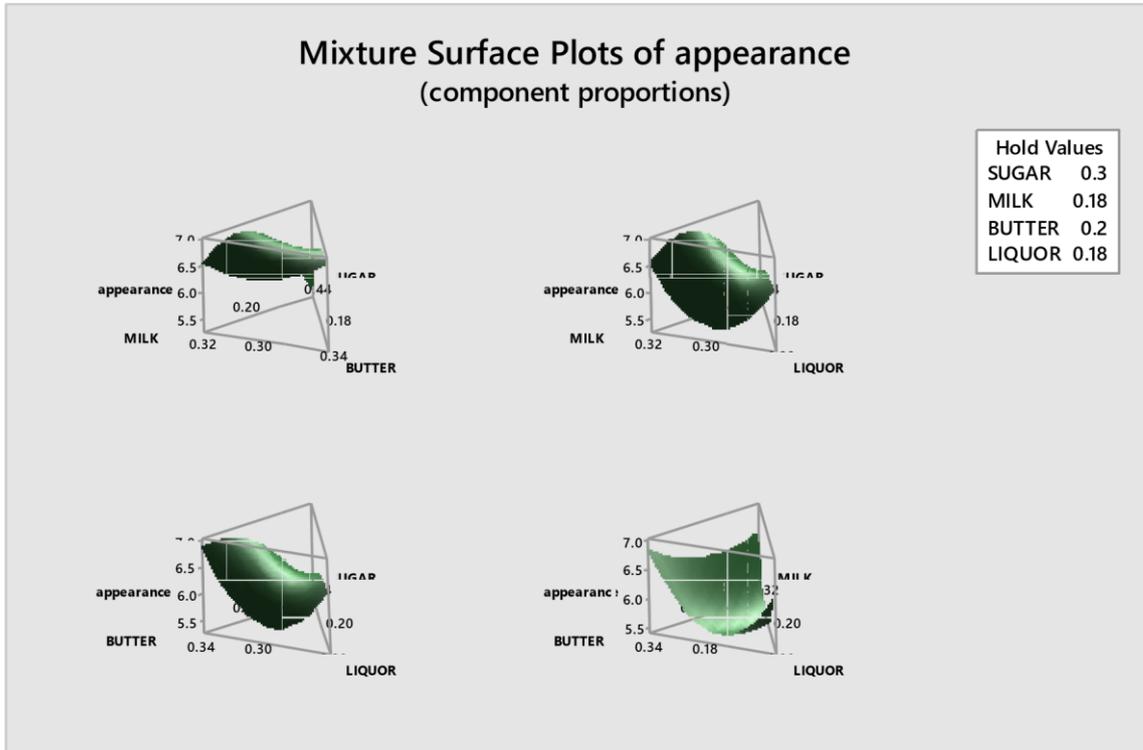
MILK*LIQUOR	1	0.53984	0.61750	0.617496	8.18	0.035
BUTTER*LIQUOR	1	0.48211	0.48211	0.482108	6.38	0.053
Residual Error	5	0.37764	0.37764	0.075529		
Total	14	3.05082				

Estimated Regression Coefficients for overall acceptability (component amounts)

Term	Coef
SUGAR	-0.273162
MILK	0.228472
BUTTER	0.150848
LIQUOR	0.672488
SUGAR*MILK	0.00801869
SUGAR*BUTTER	0.00802176
SUGAR*LIQUOR	-2.11879E-04
MILK*BUTTER	-0.00482511
MILK*LIQUOR	-0.0185609
BUTTER*LIQUOR	-0.0164004

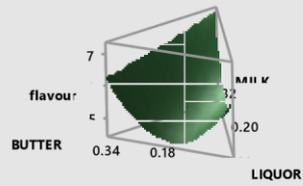
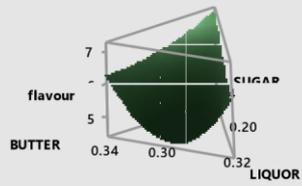
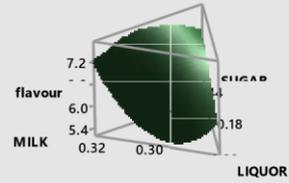
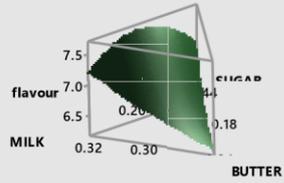


APPENDIX 7: MIXTURE SURFACE PLOTS



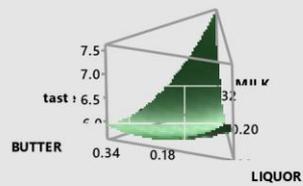
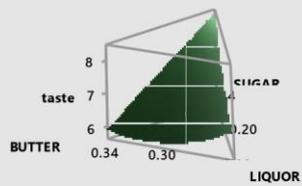
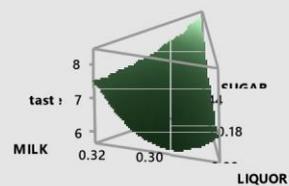
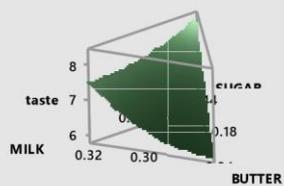
Mixture Surface Plots of flavour (component proportions)

Hold Values	
SUGAR	0.3
MILK	0.18
BUTTER	0.2
LIQUOR	0.18



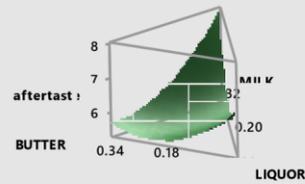
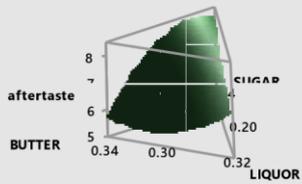
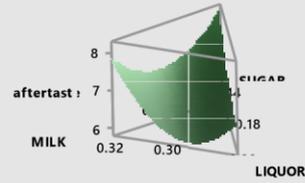
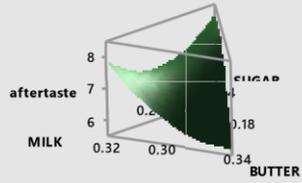
Mixture Surface Plots of taste (component proportions)

Hold Values	
SUGAR	0.3
MILK	0.18
BUTTER	0.2
LIQUOR	0.18



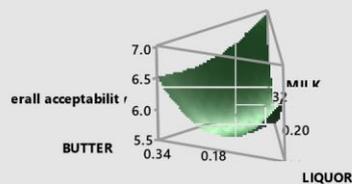
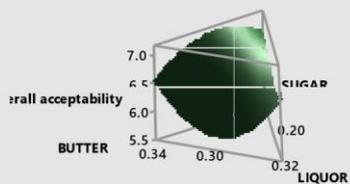
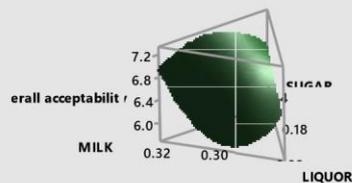
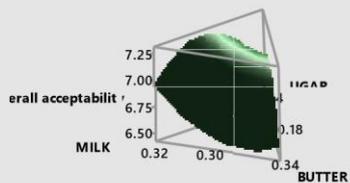
Mixture Surface Plots of aftertaste (component proportions)

Hold Values	
SUGAR	0.3
MILK	0.18
BUTTER	0.2
LIQUOR	0.18

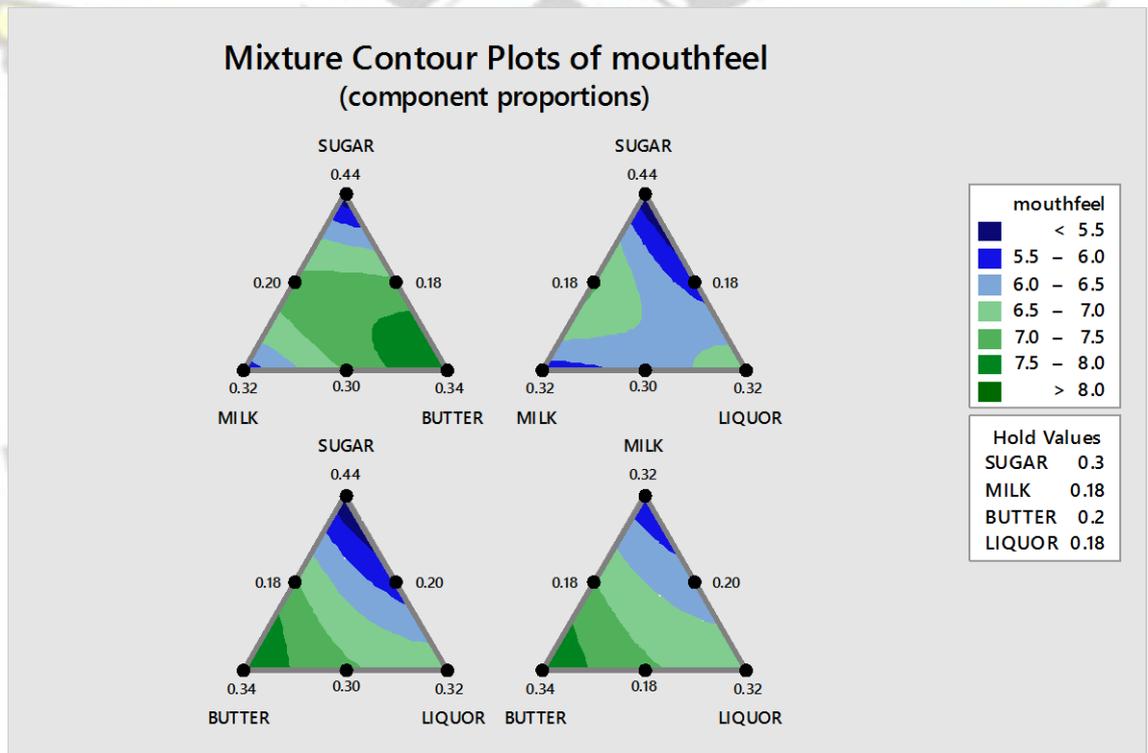
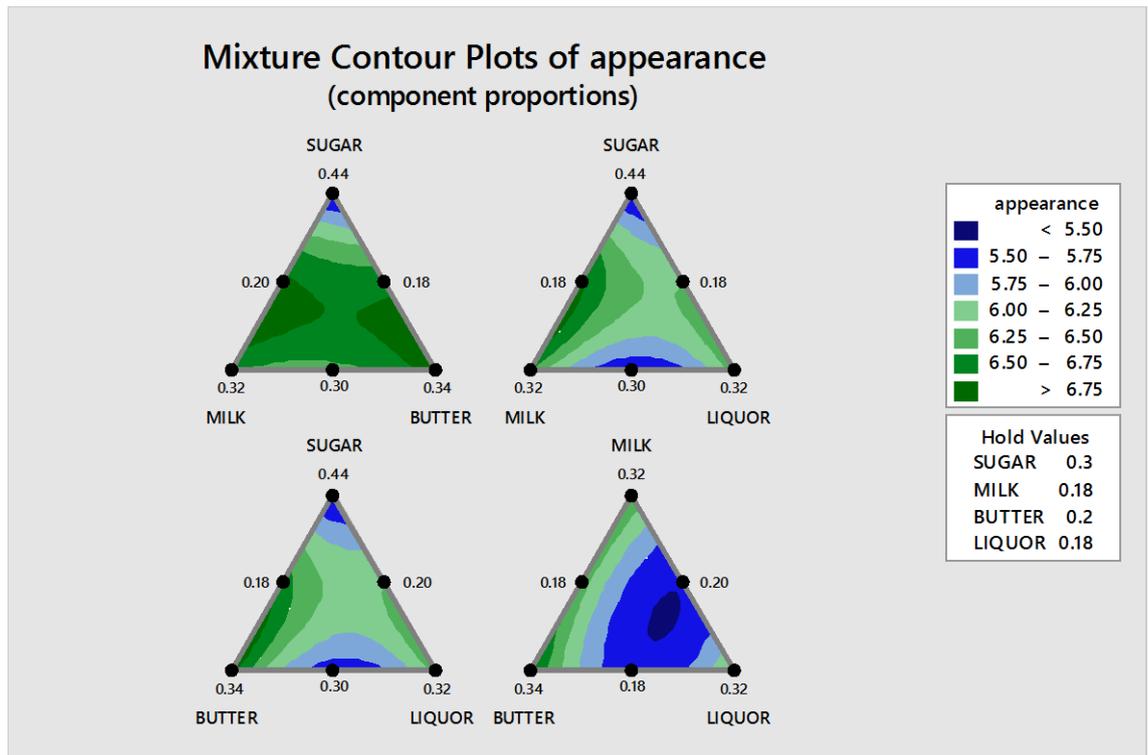


Mixture Surface Plots of overall acceptability (component proportions)

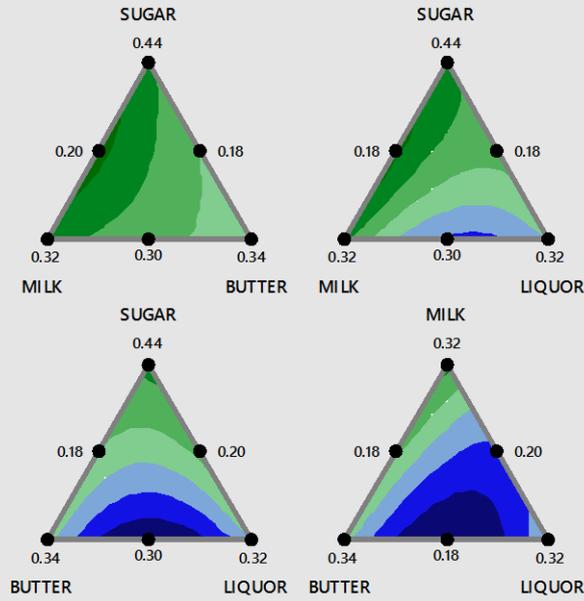
Hold Values	
SUGAR	0.3
MILK	0.18
BUTTER	0.2
LIQUOR	0.18



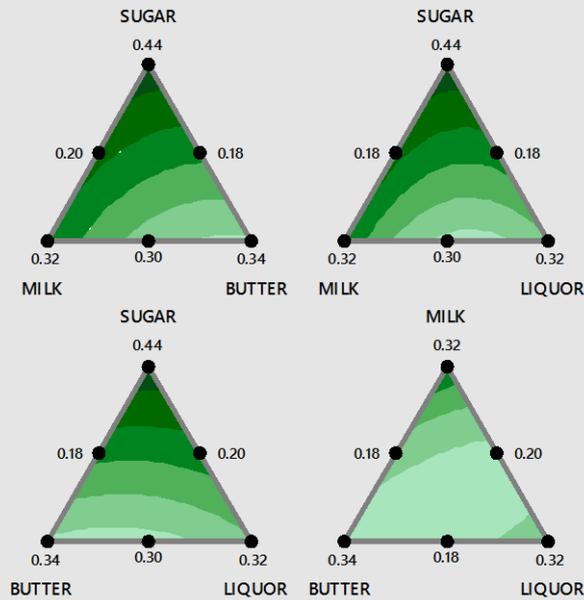
APPENDIX 8: COUNTOUR PLOT



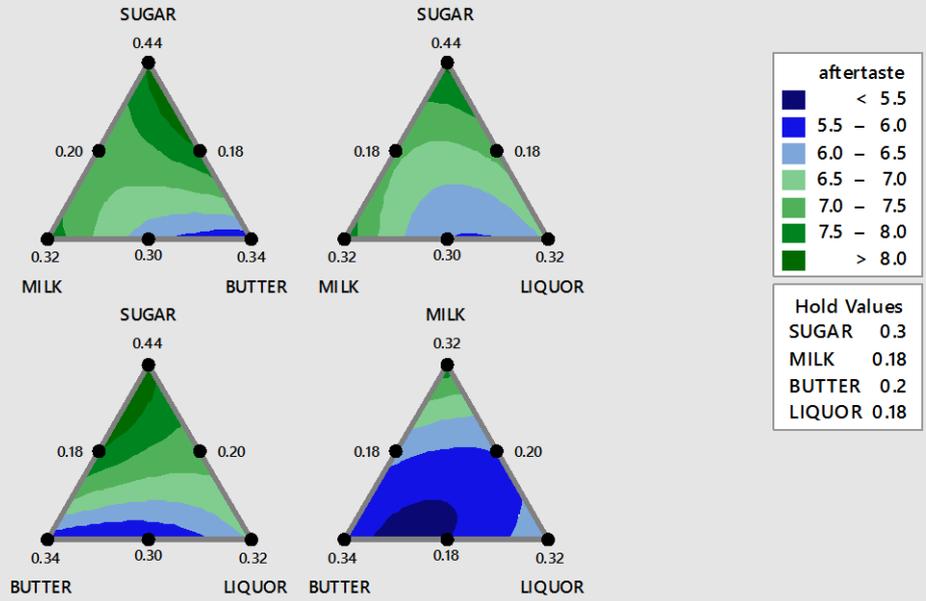
Mixture Contour Plots of flavour (component proportions)



Mixture Contour Plots of taste (component proportions)



Mixture Contour Plots of aftertaste (component proportions)



Mixture Contour Plots of overall acceptability (component proportions)

