

# Optimization Of Buffalo Milk Pastillas De Leche Using Sensory Acceptability Criteria By Plackett-Burman And Central Composite Designs

Ava Apellado-Buenaventura, Jose Subtiniente Valmorida

**Abstract:** Small-scale dairy farming in the community is growing fast with the aid of the Philippine Carabao Center by upgrading the local buffaloes. This study aimed to optimize pastillas de leche made from Murrah buffalo milk with the sensory acceptability criteria. A Plackett-Burman Design was used to screen seven ingredient and process variables which include cornstarch, all-purpose flour, liquid glucose, refined sugar, skimmed milk, temperature and time. Results revealed that the levels of cornstarch, temperature, and refined sugar significantly affect the product. These variables were used in the subsequent optimization using Central Composite Design. The acceptability of the products from the treatments were significantly higher than the control in terms of color and taste. On the other hand, the acceptability of the products in terms of aroma, texture, mouthfeel and general acceptability was not significantly higher than the control. Chemical analysis revealed a higher acceptability on products with lower moisture and higher total solids contents. Nine (9) treatments have water activity values lower than 0.85 and thus compliant with the Philippine National Standards regulation. Optimum regions of the variables were identified using the minimum acceptability rating which was uniform to all sensory attributes used. The optimum levels per liter buffalo milk for cornstarch were found in two regions, <17g and >33.44g. For refined sugar, the optimum level is found within the range of 203.72g to 221.74g. Temperature optimum levels were found in two regions also, < 78.32 and > 81.51 degrees Celsius.

**Index Terms:** Buffalo milk, Central Composite Design, Food processing, Pastillas de leche, Plackett-Burman Design, Product optimization, Sensory acceptability

## 1 INTRODUCTION

MILK is an important food and is considered a good source of calcium [1]. This commodity is present in the tables of almost every household. Thus, it helps build a healthy society. Milk can be an instrument for rural development and employment, thereby, slowing down the migration of the rural population [2]. The National Dairy Authority administrator, Salvacion Bulatao, emphasized that the Philippines is a huge market for milk and its products. In the last three years, small dairy farming communities has been growing strongly [3]. To this effect, the Philippine Carabao Center (PCC) is active in upgrading local buffaloes through its gene pool and extension work. The daily production of buffalo milk in PCC at Central Mindanao University (PCC at CMU) could reach more than 200 liters. More liters of milk are produced daily by farmers engaged in cooperatives through the efforts of the center. Milk has high amount of nutrients fit for all ages, a quality that makes it desirable for processing into different products [4]. The dairy industry is maintaining its competitiveness in developing new products and processes [5]. This results in an increase in the utilization of locally produced buffalo's milk [6]. The high nutritional value and its functional properties makes buffalo milk suitable for processing various dairy products [7]. One example is pastillas de leche, a milk-based confectionery using milk and sugar as the main ingredients. Parducho [8] reviewed that pastillas de leche originated from a home-based production. The farmers made the product for the primary purpose of lengthening the shelf-life of surplus milk.

From then on, pastillas-making grew into a small-scale industry in dairy production areas like Muñoz, Nueva Ecija, San Miguel, Bulacan and Maramag, Bukidnon. Today, several small-scale industries consider making pastillas de leche using buffalo milk. However, the studies on dairy products using buffalo milk are considerably lesser than products using milk of other animals<sup>9</sup> such as the cow's milk. The most commonly studied buffalo milk products are yogurt and cheese. Published literature on pastillas de leche as a milk product is scarce. This study aimed at giving a scientific insight to process a good quality pastillas de leche from buffalo milk.

## 2 MATERIALS AND METHODS

Fresh raw buffalo milk was obtained from the Philippine Carabao Center at CMU. All-purpose flour (Cream Brand), cornstarch (Cream Brand), refined sugar (Gaisano repacked), powdered skimmed milk (Golden Boy Brand) and glucose (Lucid Brand) were purchased in Valencia City, Bukidnon. The fresh milk was strained through layers of cheese-cloth to remove traces of animal feed, soil, insects, and other foreign materials acquired from the milking process. Other ingredients were weighed according to the amount required for each treatment. Cooking was done using a heavy skillet with constant stirring under different processing conditions required for each treatment. The cooked mixture was allowed to cool before being molded into serving pieces for individual wrapping with glassine (primary wrapper) and water cellophane (outer wrapper). Fig. 1 illustrates the process in producing pastillas de leche.

- Ava Apellado-Buenaventura is an instructor at Central Mindanao University. E-mail: [ava.buenaventura@cmu.edu.ph](mailto:ava.buenaventura@cmu.edu.ph)
- Jose Subtiniente Valmorida is a professor at the College of Human Ecology, Central Mindanao University. E-mail: [jvalmorida@cmu.edu.ph](mailto:jvalmorida@cmu.edu.ph)

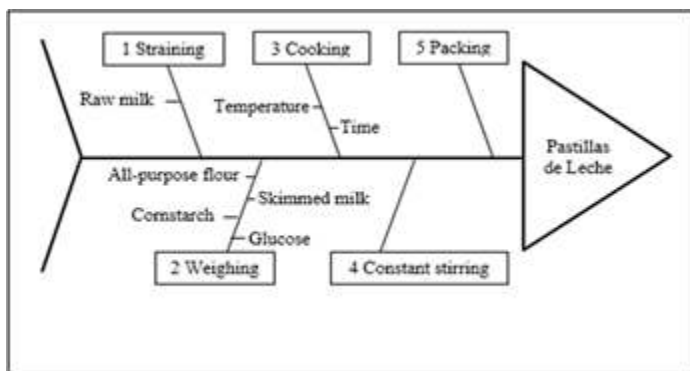


Fig.1. Ishikawa diagram for making pastillas de leche

A two-level Plackett-Burman (PB) design was employed in the screening to identify variables that would significantly affect pastillas de leche. The design generated eight runs for seven variables and then translated to eight treatments with a maximum and minimum level of each variable as shown in Table 1.

TABLE 1  
TWO-LEVEL PLACKETT-BURMAN 8-RUN MATRIX

TREATMENT	VARIABLES						
	CORN STARCH (g)	AP FLOUR (g)	LIQUID GLUCOSE (g)	REFINED SUGAR (g)	SKIM MILK (g)	TEMPERATURE (°C)	TIME (mins)
1	25	25	150	0	25	70	95
2	0	25	150	200	0	80	95
3	0	0	150	200	25	70	105
4	25	0	0	200	25	80	95
5	0	25	0	0	25	80	105
6	25	0	150	0	0	80	105
7	25	25	0	200	0	70	105
8	0	0	0	0	0	70	95

The samples were evaluated by panelists using the Hedonic Scale to obtain results for each treatment's attribute acceptability. A Central Composite Design (CCD) was subsequently employed in determining the optimum region of the three variables which significantly affected pastillas de leche. Table 2 shows the design matrix of a three-variable CCD. A maximum, a middle, and a minimum level of each variable was identified.

TABLE 2  
DESIGN MATRIX OF A THREE-VARIABLE CENTRAL COMPOSITE DESIGN

TREATMENTS	VARIABLES		
	Cornstarch (g)	Refined Sugar (g)	Temperature (°C)
1	15	250	75
2	15	150	75
3	15	150	85
4	15	250	85
5	15	200	80
6	25	200	75
7	25	150	80

8	25	200	85
9	25	250	80
10	25	200	80
11	35	250	75
12	35	150	75
13	35	150	85
14	35	250	85
15	35	200	80

The sensory evaluation for the optimization phase utilized the Balanced Incomplete Block design. Table 3 shows Cochran and Cox's Plan 13.9 Incomplete Block design for fifteen treatments including the control. Sixteen blocks (panelists) were employed and the design was repeated six times to make six replicates.

TABLE 3  
PLAN 13.9 INCOMPLETE BLOCK DESIGN FOR FIFTEEN TREATMENTS WITH CONTROL

TREATMENTS	BLOCKS (PANELISTS)															
	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16
1	X	X	X	X	X	X										
2	X	X					X	X	X	X						
3	X		X				X				X	X	X			
4	X			X				X			X			X	X	
5	X				X				X			X		X	X	X
6	X					X				X			X	X	X	X
7		X	X				X						X	X	X	X
8		X		X				X				X	X	X		X
9		X			X				X		X	X	X		X	
10		X				X				X	X	X		X		
11			X	X				X	X	X						X
12			X		X			X	X		X				X	
13			X			X		X	X				X	X		
14				X	X		X		X			X	X			
15				X		X	X		X		X				X	
Control					X	X	X	X			X					X

Laboratory panelists were semi-trained to evaluate the products using the principles of the Quantitative Descriptive Analysis (QDA). The panelists were recruited from the faculty of the Department of Food Science, Department of Nutrition and Dietetics, and Department of H.E. Education and Family Life, College of Home Economics in Central Mindanao University. In QDA, a lexicon of sensory attributes for pastillas de leche were formulated by the group composed of the panelists. The discussion was facilitated by a group leader. Commercially available pastillas was the basis for generating the lexicon. The products from the treatments were then evaluated by the panelists using the Hedonic Rating Scales for both descriptive and acceptability evaluations. Table 4 illustrates the Hedonic scales for the descriptive evaluation using the lexicon for each attribute generated by the group and the scales for the corresponding acceptability.

**TABLE 4**  
**LEXICON OF DESCRIPTIONS FOR PASTILLAS DE LECHE AND THE HEDONIC SCALE**

COLOR	AROMA	TASTE	TEXTURE	MOUThFEEL	ACCEPTABILITY
5-intense brown	5-strong milky aroma	5-very sweet	5-hard	5-extremely grainy	9-like extremely
4-dark brown	4-rich milky aroma	4-moderately sweet	4-slightly hard	4-moderately grainy	8-like very much
3-brown	3-milky aroma	3-sweet	3-soft	3-grainy	7-like moderately
2-light brown	2-slight milky aroma	2-slightly sweet	2-slightly soft	2-slightly fine	6-like slightly
1-pale brown	1-weak milky aroma	1-bland	1-very soft	1-fine	5-neither like nor dislike 4-dislike slightly 3-dislike moderately 2-dislike very much 1-dislike extremely

The scale from 1 to 5 was used in the evaluation to describe the intensity of each attribute. The group also agreed on a method of assessment for each attribute to achieve uniformity. The color was defined by its brownness and the scale ranges from "pale brown" to "intense brown". Pastillas aroma was defined by its "milky" and was evaluated in a scale from "weak milky aroma" to "strong milky aroma." The taste of pastillas was evaluated for its sweetness with a scale ranging from "bland" to "very sweet". The texture was defined by its hardness and was evaluated in a range from "very soft" to "hard". The mouthfeel was evaluated by the samples' "graininess" in a range from "fine" to "extremely grainy". Moisture, total solids and water activity were the chemical characteristics of pastillas sought. Samples were submitted to SPAL, Department of Soil Science at Central Mindanao University for moisture analysis by Oven Drying method. The total solids content was just calculated from the moisture content of the samples. More samples were submitted to the DOST-RSTL in Cagayan de Oro City for water activity analysis by the Dew Point method. The screening of variables from the Plackett-Burman Design was analyzed using the software Statistica Version 12 (Statsoft Inc., USA). ANOVA results was generated and the significant variables were identified at  $p = 0.05$  level of significance. The optimum region from the Central Composite Design was identified with the aid of the software SAS Version 6.12 (SAS Institute Inc., USA). Significant differences between the response variables (chemical and sensory) were analyzed using SPSS 20 with Scheffe's test for post hoc analysis.

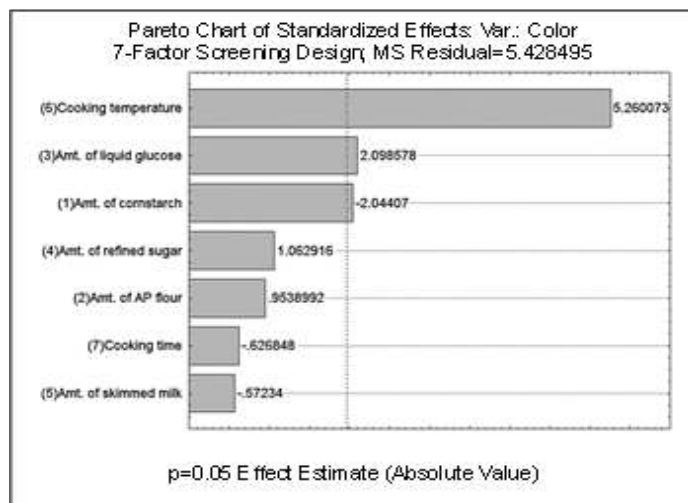
### 3 RESULTS AND DISCUSSION

The data from the sensory evaluation were analyzed to screen the independent variables and Statistica 12 was used for this purpose. The Plackett-Burman 7x8 analysis generated the parameter estimates and analysis of variances showing the variables that significantly affect the product attributes at  $p=0.05$ .

#### 3.1 SCREENING

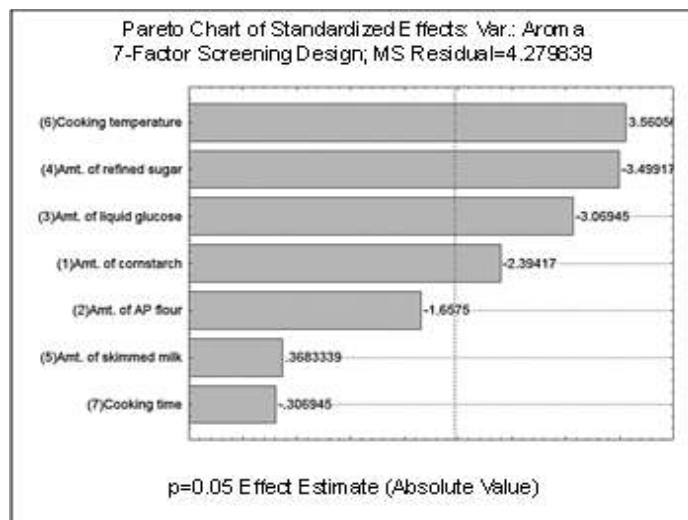
In Fig. 2, cooking temperature, liquid glucose and cornstarch affect pastillas' color acceptability. Increasing the level of cooking temperature and liquid glucose will be more

preferred by the respondents in contrast to cornstarch ( $t = -2.04$ ).



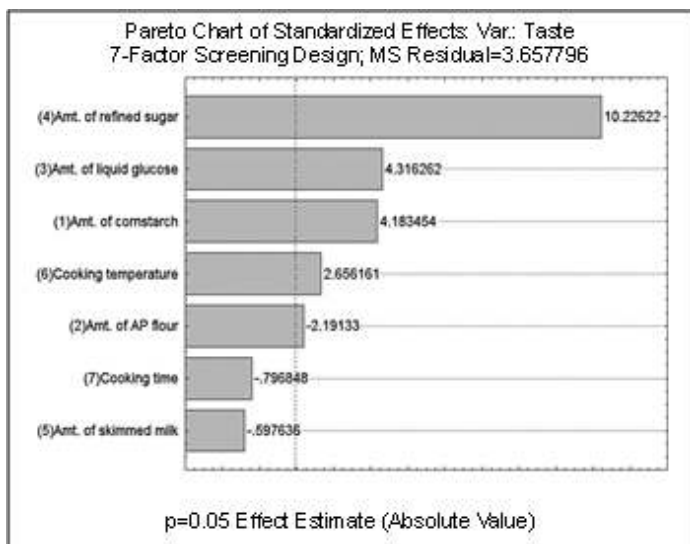
**Fig. 2. Chart showing variables significantly affecting color**

Fig. 3 below shows that cooking temperature ( $t = 3.56$ ), refined sugar ( $t = -3.49$ ), liquid glucose ( $t = -3.06$ ) and cornstarch ( $t = -2.39$ ) significantly affect the aroma with cooking temperature as the only variable with a positive  $t$ -value. The higher temperature level used produced a more acceptable aroma due to the reaction rate of Maillard reactions in the food matrix consisting of reducing sugars and alpha-amino acids [12]. Maillard reactions produces color and flavor compounds. This explains the significance of cooking temperature, liquid glucose, refined sugar, and cornstarch in affecting color and aroma acceptability.



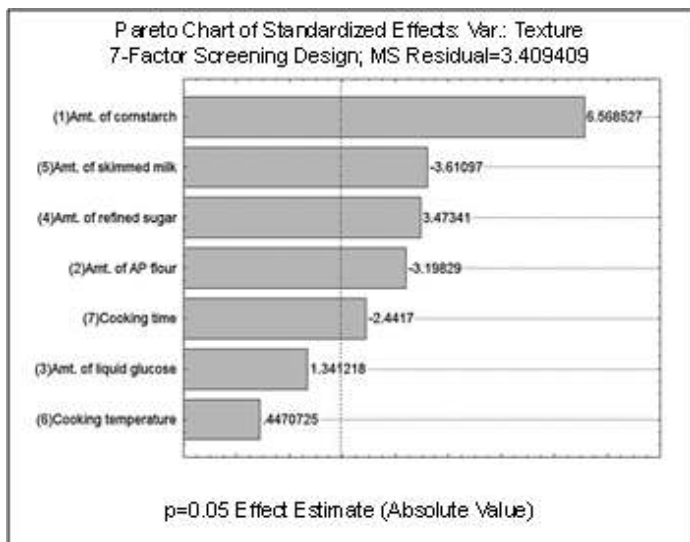
**Fig. 3. Chart showing variables significantly affecting aroma**

Fig. 4 shows significant factors affecting taste. Positive  $t$ -values are shown for refined sugar ( $t = 10.22$ ), liquid glucose ( $t = 4.31$ ), cornstarch ( $t = 4.18$ ) and cooking temperature ( $t = 2.65$ ) while a negative  $t$ -value for all-purpose flour ( $t=-2.19$ ). However, this observation did not agree with the finding of Parducho and Alfuerio [13] in which all-purpose flour was added as pastillas stabilizer and was favored by the panelists.



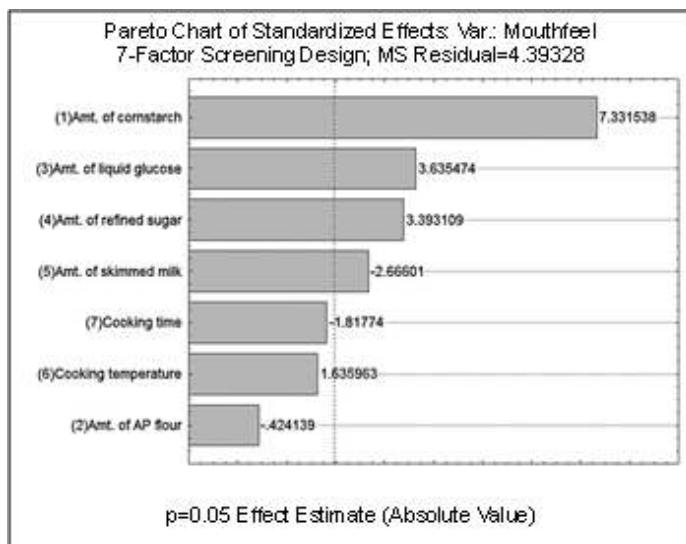
**Fig. 4.** Chart showing variables significantly affecting taste

Fig. 5 shows that cornstarch, skimmed milk, refined sugar, all-purpose flour and cooking time significantly affect texture. The high t-value ( $t=6.57$ ) for cornstarch support the findings of Athar [14] and Ayub and Siddiq [15] that cornstarch used as a stabilizer gives better rheology in yogurt.



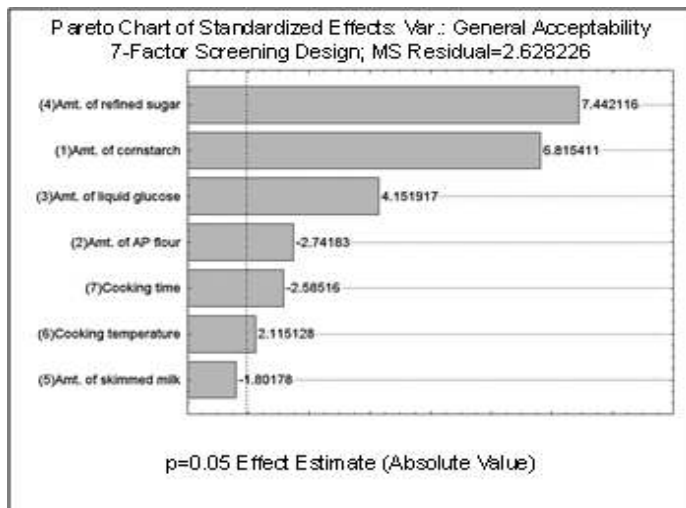
**Fig. 5.** Chart showing variables significantly affecting texture

Fig. 6 shows the variables affecting mouthfeel. Similar to texture, cornstarch primarily affects the mouthfeel of pastillas ( $t=7.33$ ), which is followed by liquid glucose ( $t=3.63$ ), refined sugar ( $t=3.39$ ) and skimmed milk ( $t = -2.66$ ). These findings agree with the investigation by Djurdjević and Jovanovic [16] on powdered skimmed milk proportionally affecting total solids and viscosity of yogurt.



**Fig. 6.** Chart showing variables significantly affecting mouthfeel

Fig. 7 shows the significant variables affecting general acceptability. Among the seven variables, skimmed milk is the only insignificant variable. Positive t-values for refined sugar ( $t = 7.44$ ), cornstarch ( $t = 6.81$ ), liquid glucose ( $t = 4.15$ ) and cooking temperature ( $t = 2.11$ ) indicates a positive relationship with general acceptability while negative t-values of all-purpose flour and cooking time ( $t = -2.74$  and  $t = -2.58$ , respectively) indicates a negative relationship. Increasing the level of the variable with positive t- values will gain higher acceptability rating.



**Fig. 7.** Chart showing variables significantly affecting General Acceptability

Based on the figures above, three variables were consistent on being the top significant variables affecting the attributes. The significant ingredient variables were cornstarch and refined sugar. The significant process variable is the temperature of cooking. From all sensory attributes, the three variables have a greater number of high t-scores compared to the other variables. Table 5 shows the summary of the significant variables and the corresponding ranks. After being identified, these variables were manipulated in the optimization phase.

**TABLE 5**  
SUMMARY OF THE SIGNIFICANT VARIABLES AND THEIR  
CORRESPONDING RANKS

VARIABLE	COLOR	AROMA	TASTE	TEXTURE	MOUTH FEEL	GENERAL ACCEPTABILITY	TOP 3
Cornstarch	3 (-)	4 (-)	3	1	1	2	X
All-purpose flour			5 (-)	4 (-)		4 (-)	
Liquid glucose	2	3 (-)	2		2	3	
Refined sugar		2 (-)	1	3	3	1	X
Skimmed milk				2 (-)	4 (-)		
Cooking temp	1	1	4			6	X
Cooking time				5 (-)		5 (-)	

### 3.2 OPTIMIZATION

The three significant variables, cornstarch, refined sugar and temperature of cooking, were used as the independent variables for the optimization phase. The four remaining variables were retained in the formulations at their minimum levels throughout the treatments. The data were analyzed using the Statistical Analysis Software (SAS) which identified the linear, quadratic and interaction effects to the acceptability of each attribute. The analysis also identified estimated optimum values for each variable with a predicted acceptability rating. Table 6 shows the ANOVA for the color attribute. Cornstarch and cooking temperature significantly ( $p=0.01$ ) affect the color individually. Refined sugar's linear effect is not significant ( $p=0.05$ ). The linear negative estimate of cornstarch indicates that the lower levels give better color acceptability. Cooking temperature, on the other hand, has higher color acceptability at higher levels used as shown in its positive estimate. The quadratic effect of cornstarch ( $p=0.05$ ), refined sugar ( $p=0.05$ ) and cooking temperature ( $p=0.01$ ) significantly affect color acceptability. Cornstarch has a positive estimate which means that doubling cornstarch levels increases color acceptability. Both refined sugar and temperature quadratic effects have negative estimates indicating reduced acceptability when both levels are doubled. The interactions of cornstarch and refined sugar with temperature are highly significant ( $p=0.01$ ) with positive estimates that show higher acceptability when their levels are increased together. This supports the theory that high temperature hastens Maillard reactions involving reducing sugars and protein in the food matrix [12] to produce color compounds. Furthermore, Mačej [17] found denaturation of milk proteins at high temperatures affecting the technological properties.

**TABLE 6**  
ANOVA FOR COLOR

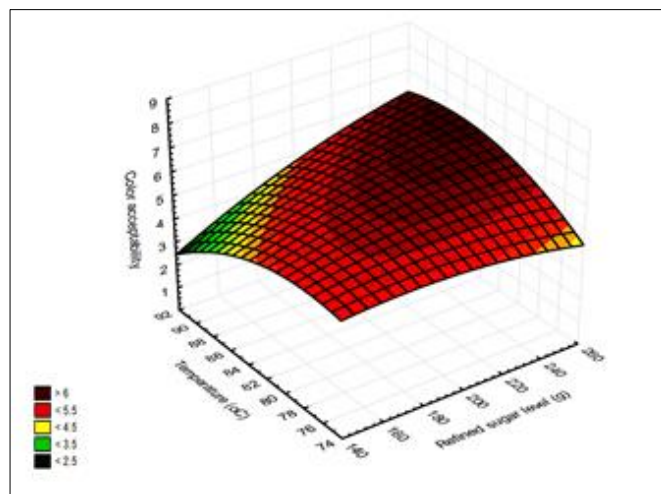
SOURCE OF VARIANCE	DF	SS	MS
Total	566	268.49	
Linear regression: estimates	3		
Cornstarch	1	95.08	31.69
Ref. sugar	1		
Temperature	1		
Quadratic:	3		
Cornstarch* <i>Cornstarch</i>	1	74.88	24.96
<i>Rsugar</i> * <i>Rsugar</i>	1		
Temp*Temp	1		
Crossproduct:	3		
Cornstarch* <i>Rsugar</i>	1	98.54	32.85
Cornstarch*Temp	1		
<i>Rsugar</i> *Temp	1		
Total error		2021.05	3.57

\* - significant at  $p=0.05$  level

\*\* - significant at  $p=0.01$  level

ns – not significant

The following figures show the surface plots of these interactions illustrating the color acceptability at different variable levels. Figure 8 shows the reaction of temperature and refined sugar when the cornstarch variable is set as a constant. The highest achievable acceptability ( $>6$ ) is located at a region with the temperature level 84-90°C and refined sugar level above 240g. Lower acceptability ratings ( $<4.5$ ) are achieved when the sugar level is below 180g even at temperature levels above 84°C.



**Fig. 8.** Color acceptability at varying temperature and sugar level keeping cornstarch at a constant

Fig. 9 shows the interaction of cornstarch and refined sugar at constant temperature. Fig. 10 shows the interaction of cornstarch and temperature at constant sugar level. Both plots exhibit a "saddle" where peaks with highest acceptability scores ( $>6$ ) are found in two separate regions. In Fig. 9, color acceptability increases with increasing sugar level having a peak at the 200g to 250g. But beyond the 250g sugar level, the acceptability decreases. Two levels of cornstarch have high acceptability and both levels are found to be beyond the levels used in this study. This "saddle effect" may be due to the closeness of cornstarch levels used that its effect on color is undetectable to the panelists.

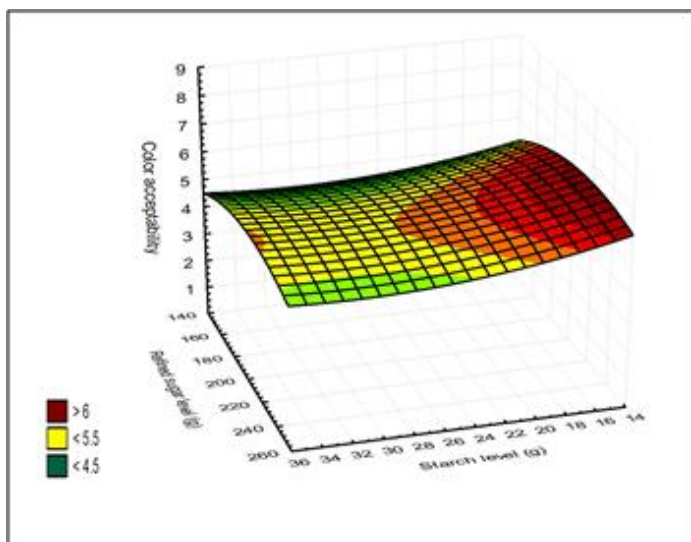


Fig. 9. Color acceptability at constant temperature

Fig. 10 shows a similar plot with Fig. 9 where a saddle effect was observed. Temperature levels at 76°C and 84°C show a region of high acceptability (>6). Two levels of cornstarch have high acceptability values. One peak shows high acceptability at cornstarch levels of 15g to 21g and the other peak is located at a region with cornstarch levels beyond the levels used in this study.

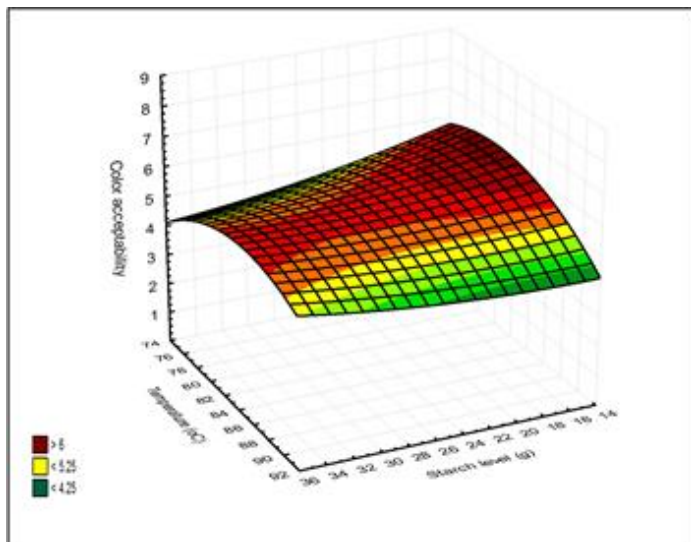


Fig. 10. Surface plot of color acceptability at constant refined sugar

Apparently, cornstarch does not influence the color of dairy products significantly. This is supported by the findings of Radi, Niakousari and Amiri [18] on yogurt made with different starch concentrations where the color of yogurt at different treatments did not differ significantly.

Table 7 shows the ANOVA for aroma. Linear regression for aroma shows that the individual effect of factors is not significant. The quadratic effects of sugar (p=0.01) and temperature (p=0.05) are significant and has a negative estimate which indicate that too much of each factor may be unacceptable.

TABLE 7  
ANOVA FOR AROMA

SOURCE OF VARIANCE		DF	SS	MS
Total		566	147.71	
Linear regression:	Parameter	3	6.88	2.29
estimates		1		
Cornstarch	-0.88ns	1		
Ref. sugar	0.0069ns	1		
Temperature	1.00ns	1		
Quadratic:		3	59.03	19.68
Cornstarch* Cornstarch	0.0023ns	1		
Rsugar* Rsugar	-0.00025**	1		
Temp* Temp	-0.0089*	1		
Crossproduct:		3	81.81	27.27
Cornstarch* Rsugar	-0.00028ns	1		
Cornstarch* Temp	0.0087**	1		
Rsugar* Temp	0.0011*	1		
Total error			1930.07	3.41

\* - significant at p=0.05 level

\*\* -significant at p=0.01 level

ns – not significant

The interactions of cornstarch with temperature (p=0.01) and sugar with temperature (p=0.05) are significant with positive estimates. These interactions positively affect the acceptability of pastillas aroma. Similar to color, the development of aroma compounds results from the reaction of reducing sugars, proteins and other compounds present in the food. This reaction is hastened with high temperatures. Fig. 11 shows a region with high acceptability (>6) at a sugar level of around 200g and a temperature level of around 80°C.

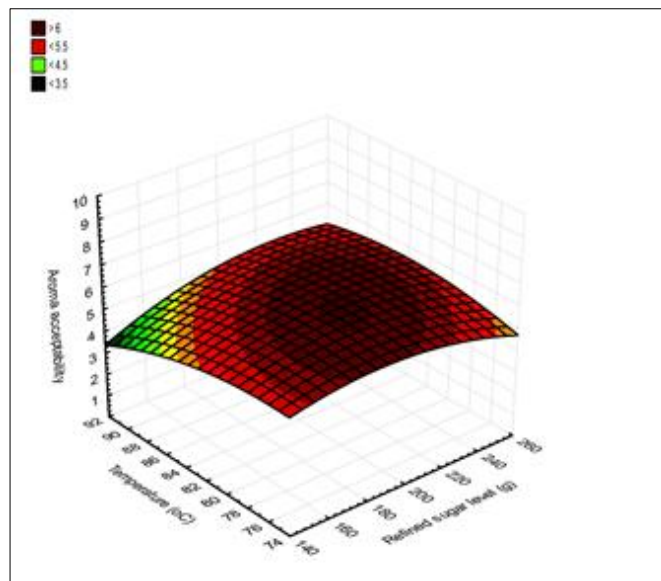


Fig. 11. Aroma acceptability at constant starch level

Fig. 12 shows a “saddle effect” with two regions for cornstarch levels with high acceptability. The lower level at approximately 18g and below and the upper level is beyond the levels used in the study. Refined sugar levels show high acceptability at 180g to 220g.

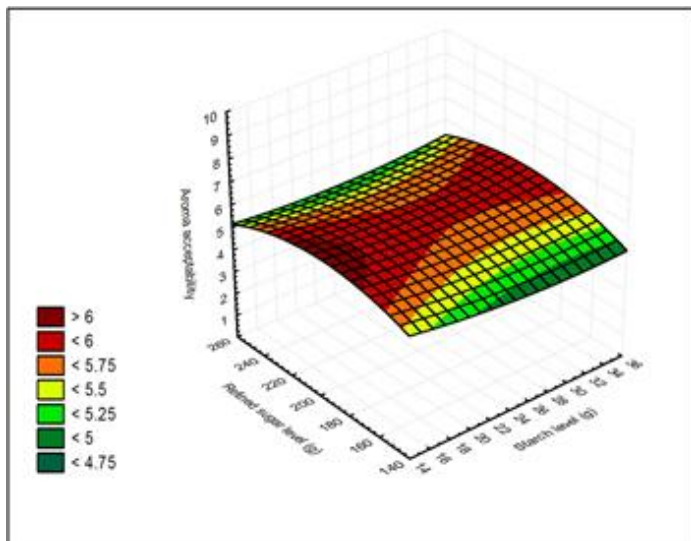


Fig. 12. Aroma acceptability at constant temperature

Fig. 13 also shows a saddle effect with the cornstarch and temperature levels. One peaks at a temperature level of 74°C to 80°C and a starch level of below 22g. The other peak is at 82°C to above 90°C with a starch level of above 30g. The “saddle effect” may be attributed to the formation of complexes by starch with a wide variety of molecules including those responsible for flavor as explained by Yildiz [12]. Some flavor molecules become unavailable contributing to difficulty by the panelists to perceive aroma.

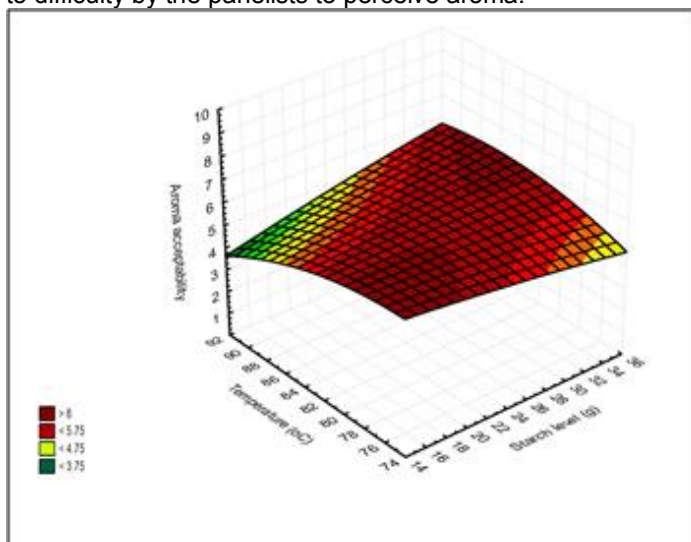


Fig. 13. Aroma acceptability at constant refined sugar level

Table 8 shows the ANOVA for taste. Refined sugar does not affect the taste significantly as an individual factor. This is contrary to one of its main function as a sweetener. This may be due to the presence of liquid glucose in all treatments that may substitute in contributing to the sweetness of pastillas. Cornstarch affects the taste similar to how it affects aroma wherein it forms complexes with flavor molecules [12]. The quadratic and cross-product effects of sugar, however, affect the taste significantly ( $p=0.05$ ). Temperature significantly affects taste acceptability in the linear ( $p=0.05$ ), quadratic ( $p=0.01$ ), and cross-product ( $p=0.01$ ) main effects. Interaction between factors positively and significantly affects taste acceptability.

TABLE 8  
ANOVA FOR TASTE

SOURCE OF VARIANCE	DF	SS	MS
Total	566	248.27	
Linear regression:			
Parameter	3	109.49	36.50
estimates	1		
Cornstarch	-0.87**	1	
Ref. sugar	-0.021ns	1	
Temperature	1.41*	1	
Quadratic:	3	47.63	15.88
Cornstarch* <i>Cornstarch</i>	0.0010ns	1	
<i>Rsugar*Rsugar</i>	-0.00015*	1	
Temp*Temp	-0.012**	1	
Crossproduct:	3	91.15	30.38
Cornstarch* <i>Rsugar</i>	0.00051*	1	
Cornstarch*Temp	0.0087**	1	
<i>Rsugar*Temp</i>	0.0087**	1	
Total error		1639.96	2.90

\* - significant at  $p=0.05$  level

\*\* -significant at  $p=0.01$  level

ns – not significant

In Fig. 14, high acceptability (>6) was achieved at a sugar level of around 230g with a temperature level of around 78°C. Increasing or decreasing the levels of both factors will decrease taste acceptability.

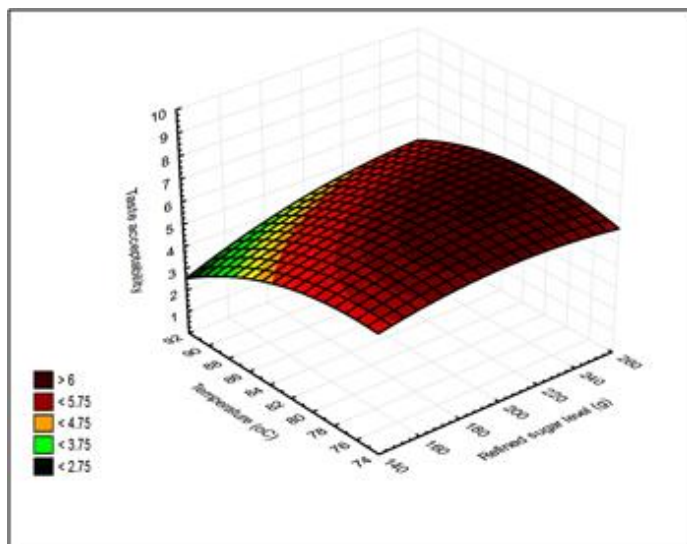


Fig. 14. Taste acceptability at constant starch level

This interaction is highly significant ( $p=0.01$ ) as seen in Table 8, a finding that supports the theory on Maillard reactions for the development of desirable flavor compounds [12].

Fig. 15 shows the interaction of refined sugar and starch that affects the taste acceptability. Refined sugar has a longer level-range of 170g to 250g at a lower cornstarch level (below 22g). A shorter range for sugar level was observed (220g to 250g) at the higher cornstarch level of 30g and above.

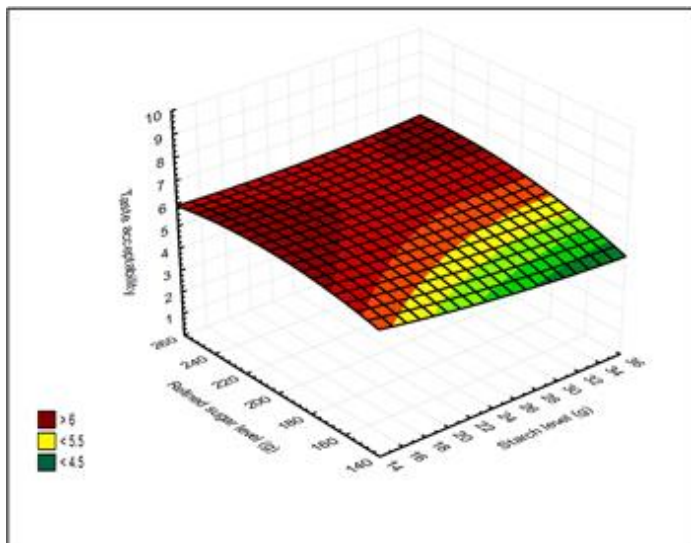


Fig. 15. Taste acceptability at constant temperature

Fig. 16 shows the interaction of temperature with starch. A high acceptability (>6) was observed at lower cornstarch and temperature levels (15-18g and 75-78°C, respectively).

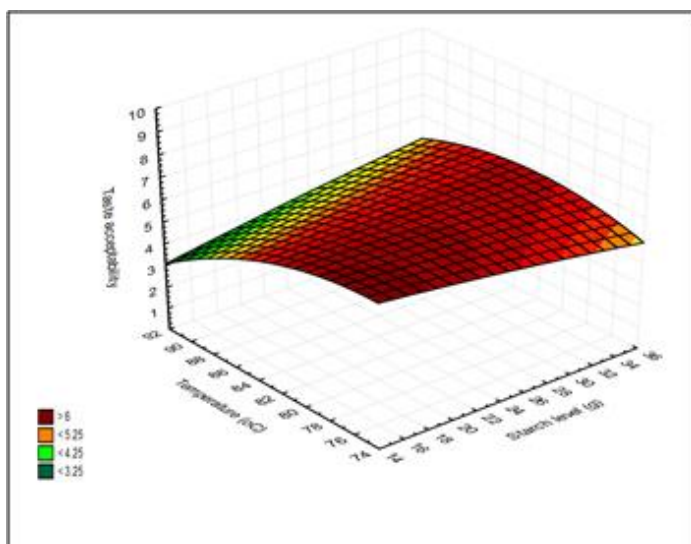


Fig. 16. Taste acceptability at constant refined sugar level

Table 9 shows the ANOVA for texture and only cornstarch and temperature have remarkable effects. Cornstarch is highly significant ( $p=0.01$ ) both in its linear effect and cross-product effect with temperature. This supports the findings of Athar, Khan, and Shah [14] and Ayub and Siddiq [15] that cornstarch affects the rheology of yogurt. Tomasik, Sady, Grega, and Najgebauer [19] explained that cornstarch forms polymeric complexes with milk casein.

TABLE 9  
ANOVA FOR TEXTURE

SOURCE OF VARIANCE		DF	SS	MS
Total		566	99.97	
Linear regression: estimates		3	32.92	10.97
	Parameter	1		
	Cornstarch	-0.68**	1	
	Ref. sugar	0.0077ns	1	
	Temperature	0.64ns	1	
Quadratic:		3	20.25	6.75
	Cornstarch* Cornstarch	0.00092ns	1	
	Rsugar* Rsugar	-0.00013ns	1	
	Temp* Temp	-0.0058ns	1	
Crossproduct:		3	46.81	15.6
	Cornstarch* Rsugar	0.00020ns	1	
	Cornstarch* Temp	0.0072**	1	
	Rsugar* Temp	0.00058ns	1	
Total error			1576.35	2.78

\* - significant at  $p=0.05$  level

\*\* - significant at  $p=0.01$  level

ns – not significant

Fig. 17 shows that there is high texture acceptability at sugar levels 220g to 240g and at temperature levels 80 to 84°C. Both Fig. 18 and Fig. 19 show a saddle in acceptability due to starch level and to starch and temperature levels, respectively.

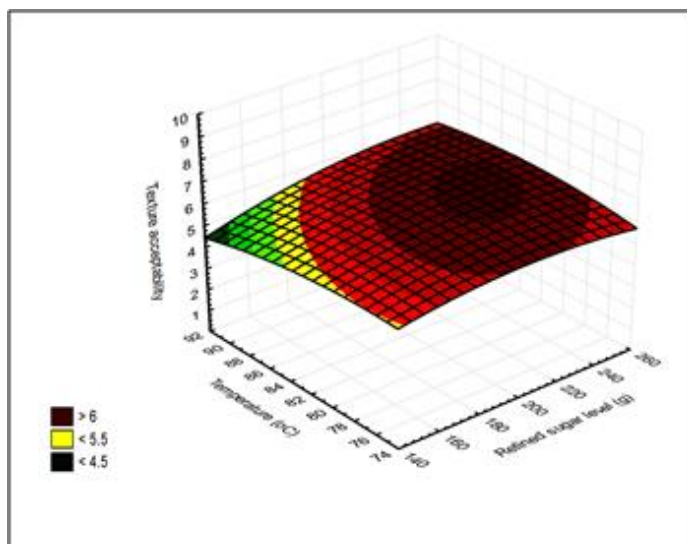


Fig. 17. Texture acceptability at constant starch level



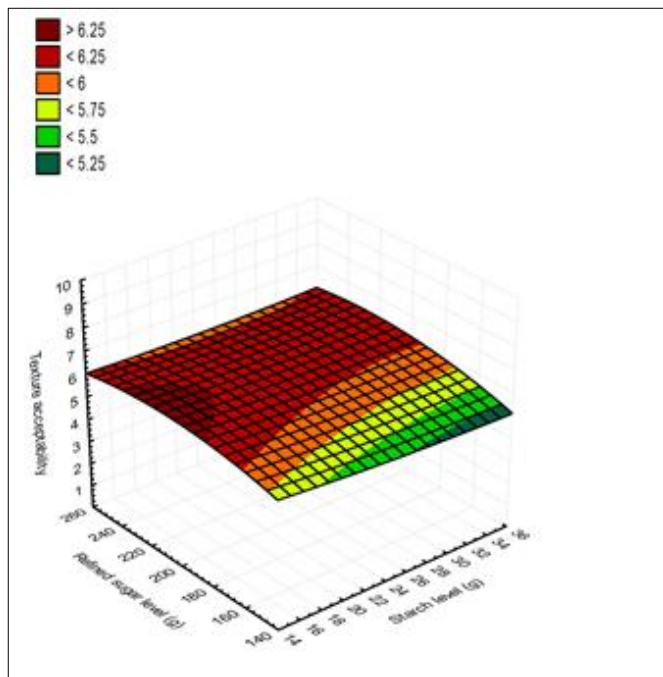


Fig. 18. Texture acceptability at constant temperature level

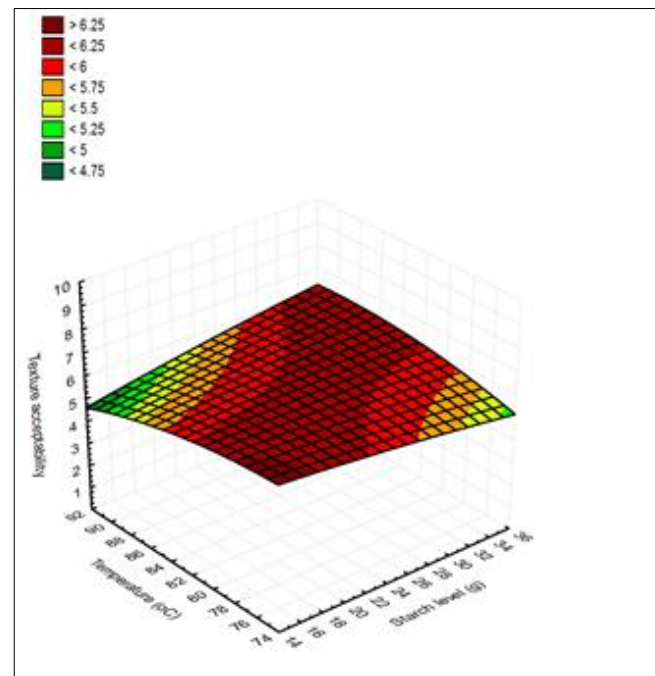


Fig. 19. Texture acceptability at constant refined sugar level

Table 10 shows the ANOVA for mouthfeel. Similar to the ANOVA analysis in texture, only cornstarch has a significant linear effect ( $p=0.01$ ). The quadratic effects of sugar ( $p=0.01$ ) and temperature ( $p=0.05$ ) are also significant. Refined sugar form crystal lattices in the food matrix that affects the mouthfeel. Vaclavik and Christian [20] further explains that sugar concentration is a factor influencing the degree of crystallization and candy type. The interaction effects of the three factors are all significant ( $p=0.05$ ). Furthermore, the temperature of starch gelatinization was found to be influenced by the presence of sucrose [21].

TABLE 10  
ANOVA FOR MOUTHFEEL

SOURCE OF VARIANCE	DF	SS	MS
Total	566	223.99	
Linear regression:	Parameter estimates	3	
Cornstarch	-0.94**	1	39.16
Ref. sugar	-0.043 <sup>ns</sup>	1	
Temperature	0.68 <sup>ns</sup>	1	10.97
Quadratic:		3	
Cornstarch* Cornstarch	0.0012 <sup>ns</sup>	1	53.30
Rsugar* Rsugar	-0.00024**	1	6.75
Temp* Temp	-0.0079	1	
Crossproduct:		3	
Cornstarch* Rsugar	0.00046*	1	131.53
Cornstarch* Temp	0.0097**	1	15.6
Rsugar* Temp	0.0016**	1	
Total error		2111.76	3.73

\* - significant at  $p=0.05$  level

\*\* - significant at  $p=0.01$  level

ns – not significant

Fig. 20 to Fig. 22 show the surface plots for mouthfeel acceptability that illustrates the ANOVA results.

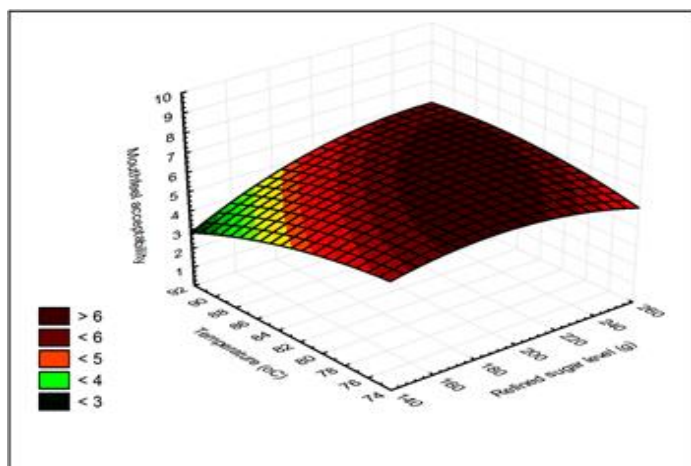


Fig. 20. Mouthfeel acceptability at constant starch level

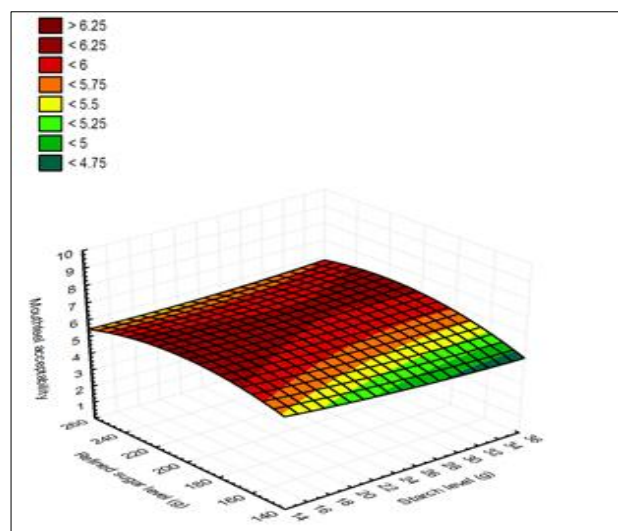
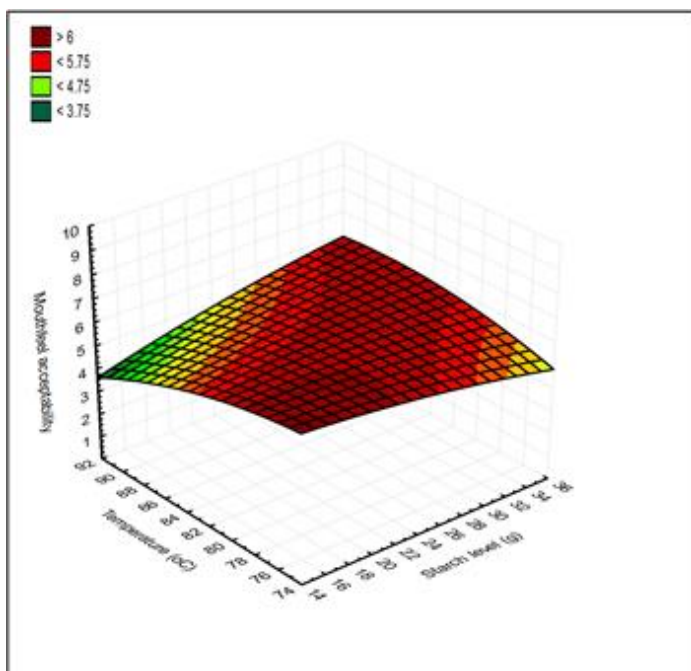
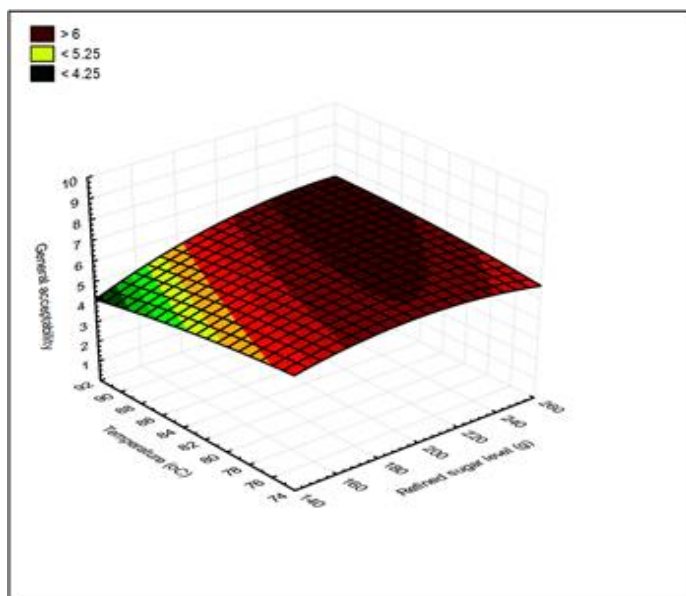


Fig. 21. Mouthfeel acceptability at constant temperature level



**Fig. 22.** Mouthfeel acceptability at constant refined sugar level



**Fig. 23.** General acceptability at constant starch level

Table 11 shows the ANOVA for general acceptability. The linear effect of cornstarch is highly significant ( $p=0.01$ ) with a negative estimate which indicates that increasing its level will decrease the general acceptability of pastillas. Refined sugar is the only factor with a significant quadratic effect ( $p=0.01$ ). It has a negative estimate indicating a decrease in product general acceptability when levels are further increased. The interaction effects of temperature with cornstarch and temperature with refined sugar are highly significant ( $p=0.01$ ) with a positive estimate indicating increased acceptability when the level of interaction increases.

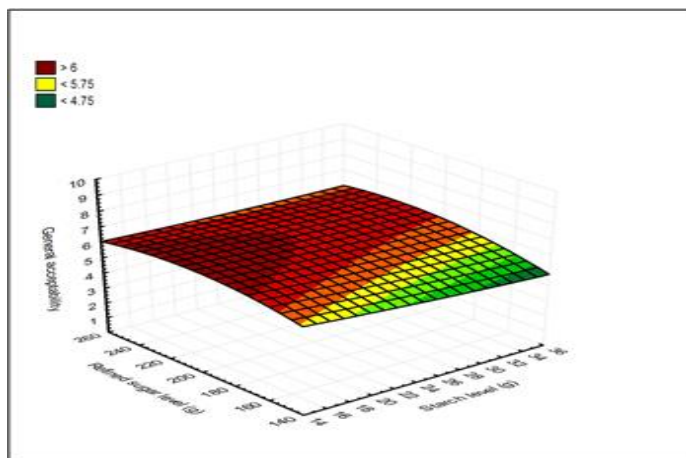
**TABLE 11**  
ANOVA FOR GENERAL ACCEPTABILITY

SOURCE OF VARIANCE		DF	SS	MS
Total		566	141.68	
Linear regression:				
	Parameter estimates	3	61.36	20.45
	Cornstarch	1		
	Ref. sugar	1		
	Temperature	1		
Quadratic:				
	Parameter estimates	3	25.00	8.33
	Cornstarch* Cornstarch	1		
	Rsugar* Rsugar	1		
	Temp* Temp	1		
Crossproduct:				
	Parameter estimates	3	55.32	18.44
	Cornstarch* Rsugar	1		
	Cornstarch* Temp	1		
	Rsugar* Temp	1		
Total error			1153.49	2.04

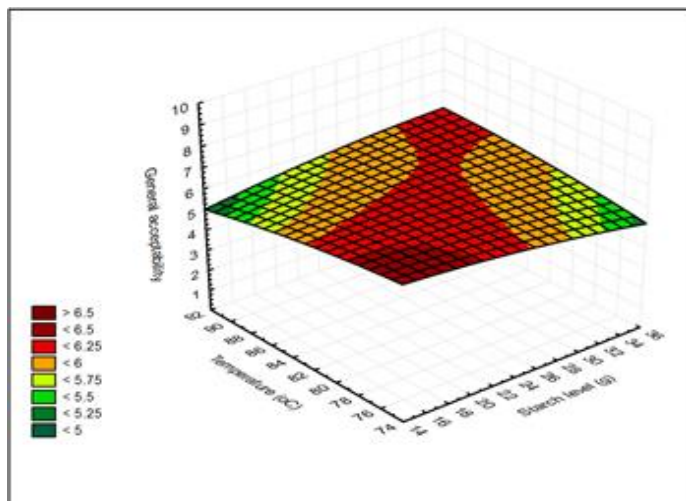
\* - significant at  $p=0.05$  level

\*\* -significant at  $p=0.01$  level

ns – not significant



**Fig. 24.** General acceptability at constant temperature level



**Fig. 25.** General acceptability at constant refined sugar level

Fig. 23 to Fig. 25 illustrates the general acceptability exhibiting the same trend as the other quality attributes where Fig. 24 and Fig. 25 with constant temperature and refined sugar respectively, show a saddle effect.

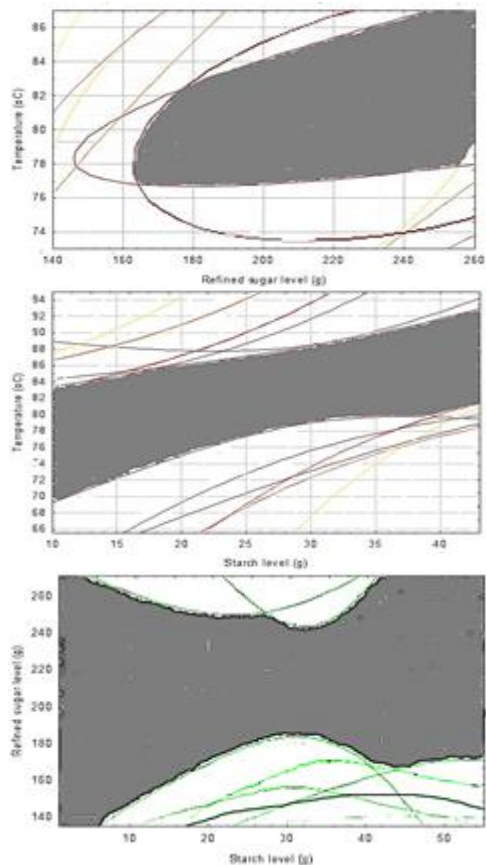
Table 12 shows the estimated optimum levels for maximum acceptability by SAS. The mean responses for all attributes range from the 5 to 6 ratings and are in between the “neither like nor dislike” and “like slightly” description. To achieve the

predicted maximum acceptability ratings that range from 6 to 7 (“like slightly” to “like moderately”), the estimated factor levels should be followed. For instance, general acceptability is predicted to have a rating of 6.59 if the cornstarch level is near the minimum (16.73g), the refined sugar near the middle (203.72g) and temperature near the middle (78.32°C).

**TABLE 12**  
ESTIMATED OPTIMUM FACTOR LEVELS FOR MAXIMUM ACCEPTABILITY BY SAS

	COLOR	AROMA	TASTE	TEXTURE	MOUTHFEEL	GENERAL ACCEPTABILITY
Mean Response	5.39	5.52	5.70	5.92	5.69	5.92
Predicted Response	6.49	6.32	6.53	6.49	6.52	6.59
Estimated Optimum						
Cornstarch (g)	15.46	34.54	17.00	33.44	33.56	16.73
Ref. sugar (g)	213.4	210.27	204.00	221.35	221.74	203.72
Temperature(°C)	81.51	84.13	78.04	84.93	84.61	78.32

Fig. 26a-26c shows the estimated optimum regions of variables based on the contour plots generated. The shaded regions correspond to the levels of variables at which the acceptability of the product is more than 5.25. This rating corresponds to “neither like nor dislike” and “like slightly” descriptors.



**Fig. 26.** Estimated optimum regions of variables at constant a) starch, b) sugar, and c) temperature

Analysis of variance on the chemical and sensory properties were done to identify significant differences between treatments including the control using Scheffe’s post hoc tests. Table 13 is the ANOVA of chemical properties generated from IBM SPSS Version 20. It shows that the treatments are significantly different (p=0.05) in their moisture, total solids content, and water activity.

**TABLE 13**  
ANOVA OF CHEMICAL PROPERTIES

		SUM OF SQUARES	DF	MEAN SQUARE	F	SIG.
Moisture	Between Groups	738.216	15	49.214	61.313	.000
	Within Groups	25.686	32	.803		
	Total	763.902	47			
Total solids	Between Groups	738.216	15	49.214	61.313	.000
	Within Groups	25.686	32	.803		
	Total	763.902	47			
Water activity	Between Groups	.123	15	.008	215.083	.000
	Within Groups	.001	32	.000		
	Total	.124	47			

Table 14 shows the mean acceptability and chemical properties of each treatment. The color acceptability of control was significantly different (p=0.05) from 13 treatments but similar to 2 treatments. The taste acceptability also showed that the control is significantly different (p=0.05) from all treatments. This implies that the formulations and process levels included in the scope of this study produced a pastillas with higher acceptability. The moisture content and total solids content of the control is different (p=0.05) from 14 treatments and similar to only one treatment. The control has a higher moisture content and lower total solids. This was reflected in its low acceptability which means that pastillas with lower moisture (< 15%) and with higher total solids content (> 85%) is more acceptable. Water activity is an indicator of a product’s shelf-life. The control has significantly higher (p=0.05) water activity compared to most treatments. The regulated water activity for pastillas according to the Philippine National Standards should be below 0.85 [22]. Six treatments and the control did not meet this requirement.

**TABLE 14**  
MEAN ACCEPTABILITY AND CHEMICAL VALUES BY SCHEFFE’S POST HOC ANALYSIS

TREATMENTS	INDEPENDENT VARIABLES	DEPENDENT VARIABLES								
		Sensory mean scores					Mean chemical values			
		Color	Aroma	Taste	Texture	Mouth-feel	General Acceptability	Moisture Content	Total Solids	Water activity
1	Cornstarch (g)	5.33 <sup>ab</sup>	5.47	6.25 <sup>ab</sup>	6.06	5.72	6.17	19.41 <sup>e</sup>	80.59 <sup>b</sup>	0.78 <sup>b</sup>
15	Ref Sugar (g)									
75	Temperature (°C)									

2	3	4	5	6	7	8	9	10	11	12	13	14	15	Ctrl
15	15	15	15	25	25	25	25	25	35	35	35	35	35	25
150	150	250	200	200	150	200	250	200	250	150	150	250	200	200
75	85	85	80	75	80	85	80	80	75	75	85	85	80	90
6.03 <sup>ab</sup>	4.75 <sup>ab</sup>	6.67 <sup>b</sup>	6.42 <sup>ab</sup>	5.39 <sup>ab</sup>	4.44 <sup>a</sup>	6.36 <sup>ab</sup>	5.67 <sup>ab</sup>	5.44 <sup>ab</sup>	4.22 <sup>a</sup>	4.56 <sup>a</sup>	5.11 <sup>ab</sup>	6.14 <sup>ab</sup>	5.33 <sup>ab</sup>	4.44 <sup>a</sup>
6.17	4.86	5.14	6.44	6.00	5.19	6.19	5.44	5.47	4.69	4.94	5.28	6.22	5.89	4.89
6.47 <sup>b</sup>	4.75 <sup>ab</sup>	5.53 <sup>ab</sup>	6.58 <sup>b</sup>	6.08 <sup>ab</sup>	5.19 <sup>ab</sup>	6.42 <sup>ab</sup>	6.17 <sup>ab</sup>	5.78 <sup>ab</sup>	5.61 <sup>ab</sup>	4.78 <sup>a</sup>	4.83 <sup>ab</sup>	6.61 <sup>b</sup>	5.75 <sup>ab</sup>	4.44 <sup>a</sup>
6.22	5.33	6.11	6.33	6.56	5.25	6.47	6.14	5.92	5.56	4.94	5.86	6.58	5.92	5.36
6.64	4.72	5.44	6.42	6.42	4.86	6.81	5.94	5.64	5.03	5.03	5.06	6.69	5.81	4.78
6.61	5.25	6.19	6.44	6.39	5.19	6.67	6.17	5.97	5.53	5.00	5.25	6.47	5.92	5.47
15.63 <sup>abcd</sup>	12.69 <sup>a</sup>	9.41 <sup>a</sup>	13.31 <sup>a</sup>	17.73 <sup>d</sup>	19.00 <sup>e</sup>	14.76 <sup>abcd</sup>	18.31 <sup>d</sup>	20.88 <sup>f</sup>	16.59 <sup>abcd</sup>	24.5 <sup>g</sup>	20.59 <sup>f</sup>	17.23 <sup>abcd</sup>	18.82 <sup>e</sup>	24.5 <sup>g</sup>
84.37 <sup>cd</sup>	87.31 <sup>f</sup>	90.59 <sup>g</sup>	86.69 <sup>e</sup>	82.22 <sup>b</sup>	81.00 <sup>b</sup>	85.24 <sup>def</sup>	81.69 <sup>b</sup>	79.12 <sup>e</sup>	83.41 <sup>cd</sup>	75.4 <sup>7a</sup>	79.41 <sup>a</sup>	82.77 <sup>abcd</sup>	81.18 <sup>b</sup>	75.4 <sup>3a</sup>
0.85 <sup>gh</sup>	0.81 <sup>cd</sup>	0.71 <sup>a</sup>	0.80 <sup>bcd</sup>	0.83 <sup>def</sup>	0.89 <sup>ij</sup>	0.82 <sup>def</sup>	0.86 <sup>gh</sup>	0.89 <sup>ij</sup>	0.84 <sup>efg</sup>	0.91 <sup>i</sup>	0.88 <sup>hi</sup>	0.84 <sup>efg</sup>	0.86 <sup>gh</sup>	0.90 <sup>i</sup>

## 7 END SECTIONS

### 7.1 Conflict of Interest

The author does not find any conflicting interest with this study. However, the results of this study could be endorsed to the Philippine Carabao Center at Central Mindanao University for their product improvement.

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### 7.3 References

- [1] Soliman, G.Z.A. Comparison of chemical and mineral content of milk from human, cow, buffalo, camel and goat in Egypt. The Egyptian Journal of Hospital Medicine. 2005; 21: 116-130.
- [2] Sarwar, M., Khan M.A. and Mahr-Un-Nisa Z.I. Dairy industry In Pakistan: A scenario. International Journal of Agriculture and Biology. 2002; 4: 420-428.
- [3] Bulatao, S. The Philippine dairy industry: Situation, competitiveness, and outlook. 2013; Retrieved from [www.fao.org/ag/againfo/programmes/en/pplpi/docarc/LLAX14.pdf](http://www.fao.org/ag/againfo/programmes/en/pplpi/docarc/LLAX14.pdf).
- [4] Haque, M.A., Rashid M.H., Kajar M.F.I. and Istiak M.S. Comparison of chamcham manufactured from cow milk and buffalo milk. Journal of Bangladesh Agricultural University. 2012;10 (2): 255-260.
- [5] Fernandes, S.A.A., Ramos A., Ramos E.M., Veras D., Pinheiro F.R., Neto S., Requião L., Amui T., Carneiro J. and Matarazzo S. Sensory evaluation of buffalo butter. Italian Journal of Animal Science. 2007; 6 (2): 1140-1142.
- [6] Aplado, O., Oliveros M.C., Sarmago I., Magpantay V. and Lapitan R. Chemical composition, sensory quality and acceptability of cream cheese from pure buffalo's milk added with malunggay (Moringa Oleifera L.) leaf powder. Philippine Journal of Veterinary and Animal Science. 2013; 39 (1): 91-98.
- [7] Khedkar, C., Khedkar G., Patil M. and Kalyankar S.. Encyclopedia of food sciences and nutrition 2nd Edition. Maryland: Academic Press. 2003.
- [8] Parducho, R.R. Pastillas de leche from cow's and carabao's milk. Science Diliman. 1982; 81-89.
- [9] Mihaylova, G. and Peeva T. Trans-fatty acids and conjugated

**Note:** Treatments with different superscripts are significantly different ( $p=0.05$ )

Analysis revealed that there are two optimum levels for cornstarch, <17g for the minimum level and >33.44g for the upper level. Temperature has two optimum levels. The lower level is < 78.32°C and the upper level is > 81.51°C. The optimum level for sugar is close to the middle level used in the CCD. The level ranges from 203.72g to 221.74g. For further improvement, it is recommended that the levels of cornstarch and temperature used during the optimization should have a wider range where difference could be easily detectable by semi-trained panelists. "Saddle effects" may be avoided in determining the optimum region. A verification run should be done using the optimum levels identified. Since none of the treatments fit-in with the optimum regions, a new run is recommended to verify the predicted acceptability.

- linoleic acid in the buffalo milk. *Italian Journal of Animal Science*. 2007; 6 (2): 1056-1059.
- [10] Gacula, M. Design and analysis of sensory optimization. Connecticut, USA: Food and Nutrition Press; 1993.
- [11] Cochran, W. and Cox G. *Experimental Designs* 2nd Edition. New York: John Wiley & Sons, Inc; 1957.
- [12] Yildiz, F. *Advances in Food Biochemistry*. USA: Taylor and Francis Group, LLC; 2010.
- [13] Parducho, R.R. and Alfuerio D.G. Pastillas de leche: Effect of stabilizers and the kind of milk used. *Philippine Journal of Veterinary and Animal Science*. 1981; Retrieved from [agris.fao.org/agrissearch/search.do?recordID=PH8511557](http://agris.fao.org/agrissearch/search.do?recordID=PH8511557).
- [14] Athar, I.H., Khan U.N. and Shah M.A. Effect of various stabilizers on whey separation (syneresis) and quality of yogurt. *Pakistan Journal of Biological Sciences*. 2000.
- [15] Ayub, M. and Siddiq M. Effect of different types of milk and stabilizers on the chemical composition of quality yogurt. *Sarhad Journal of Agriculture*. 2003.
- [16] Djurdjević, J., Maćej O. and Jovanović S. The influence of dry matter, applied heat treatment and storage period on the viscosity of stirred yogurt. *Journal of Agricultural Sciences*. 2002; 47 (2):189-204.
- [17] Maćej, O., Jovanović S., and Djurdjević J.D. The influence of high temperatures on milk proteins. *Hem. Ind.* 2002; 56 (3): 123-132.
- [18] Radi, M., Niakousari M., and Amiri S. Physicochemical, textural and sensory properties of low-fat yogurt produced by using modified wheat starch as a fat replacer. *Journal of Applied Sciences*. 2009; 9 (11): 2194-2197.
- [19] Tomasik, P., Sady M., Grega T. and Najgebauer D. Polymeric complexes of cornstarch and waxy cornstarch phosphates with milk casein and their performance as biodegradable materials. *Molecules*. 2004; 9 (7): 550-567.
- [20] Vaclavik, V. and Christian E. *Essentials of food science*. 3rd Edition. New York. Springer Science+Business Media, LLC; 2008.
- [21] Kim, C.S. and Walker C.E. Effects of sugar and emulsifiers on starch gelatinization evaluated by differential scanning calorimetry. *Cereal Chemistry*. 1992; 69 (2): 212-217.
- [22] Philippine National Standards. Philippine National Standards/Food and Drug Administration 34:2011 Ethnic milk-based confectioneries (Pastillas and Yema) – specification. 2011. Retrieved from <http://www.fda.gov.ph/attachments/article/153547/pns-fda%2034-2011-milk-based%20confectioneries%20-%20specs.pdf>.