Standardization of quality of fresh cheese through experimental design for dairy sector of Imbabura.

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Summary.

This investigation arises from the need to micro enterprises to establish a systematic study, to understand the behavior the critical variables of quality for cheese, due to adulteration of milk by adding water and its impact on the performance of this product. It developed a design of experiments factorial at two levels for three factors study, recreating the process of making cheese from laboratory level, following the principles of the scientific method and PDCA sequence. Was analyzed the performance and behavior of the critical variables of quality, appearance, smell, taste and overall assessment by altering the levels of the factors of calcium, sodium chloride and rennet chloride study in the process of coagulated analyzed, with an increase of an additional 10% of water in milk. With the conditions of the experimental model, it was concluded that it is possible to compensate weight performance, when there adulteration in the raw material. It was achieved to optimize this variable to 91.75%, which represents an improvement of 6.61% referencing the worst working conditions, and 5.12% on average conditions for cheese making. Finally, was established the proposed of regulation in factors of coagulated, when there is a maximum of 10% adulteration by adding water in milk, which is to raise levels of calcium and rennet chloride 0.3 grams in each unit of referential production 480 g.

Keywords

DOE, Design of Experiments, Quality, Fresh cheese, factorial design.

Resumen.

La presente investigación surgió de la necesidad de las Microempresas de establecer un estudio sistemático que permita conocer el comportamiento de las variables críticas de calidad del queso fresco debido a la adulteración de la leche por la adición de agua y sus consecuencias en el rendimiento de este producto. Se desarrolló un diseño de experimentos de tipo factorial a dos niveles para 3 factores de estudio, recreando el proceso de elaboración de queso fresco que se realiza en las microempresas a nivel de laboratorio, siguiendo los principios del método científico y la metodología PHVA para simplificar su secuencia. Se analizó el rendimiento del gramaje y el comportamiento de las variables críticas de calidad, aspecto físico, olor, sabor y valoración general, al alterar los niveles de los factores de estudio cloruro de calcio, cloruro de sodio y cuajo dentro del proceso de coagulado, con un incremento del 10% adicional de agua en la leche. Con las condiciones del modelo experimental, se pudo concluir que se puede compensar el rendimiento del gramaje cuando existe adulteración en la materia prima. Se consiguió optimizar esta variable hasta el 91.75%, lo cual representa una mejora de 6.61% referenciaando a la peor condición de trabajo y 5.12% respecto a las condiciones promedio para elaboración de queso. Finalmente se estableció la propuesta de regulación en los factores de coagulado, elevando los niveles de cloruro de calcio y cuajo a 0,3 gramos en cada unidad de producción referencial de 480 g.

Palabras Claves

DOE, Diseño de experimentos, Calidad, Queso fresco, Diseño factorial.
1. Introduction

In Latin America the consumption of fresh cheese is habitual because the region has the right climate and conditions conducive to the expansion of cattle ranching, which has contributed to the development and proliferation of companies engaged in dairy production.

Monica Orozco, (2015) in an article entitled "A third of dairy production is dedicated to cheese" for the magazine: Leaders, says that between 2006 and 2014 the consumption of cheese per person in Ecuador increased by more than twice (0.75 to 1.57 kg) and therefore, sales of this industrial sector reported an increase of 171.7 million dollars.

According to the National Agency for Regulation and Control and Health Surveillance (ARCSA), there are 22 companies legally registered for dairy sector in the province of Imbabura, which are 13 considered Micro-companies and nine are classified as medium-sized enterprises whose main activity is production of fresh cheese type low compliance with legal regulations NTE 1528: 2012.

Although it has been advised of the quality of milk, as factor important to get a good yield in making cheese (Tornado, Maria, García Fontán, Prieto & Carballo, 1998), hoarding of large companies and their controls for quality milk, has allowed that essential raw material in the production of cheese has been monopolized, unbalancing competition (Grain International Organisation, 2012). Thus, the cost offered to producers of milk varies depending on the quality, getting itself to pay up to $ 0.30 per liter when the features are not fulfilled and $ 0.47 if they are. (Ochoa, 2015)

With these problems of varying quality and price of milk has led to it being contaminated with some element (especially water) to increase the quantity delivered to companies that have deficiencies in quality control for the reception, making the micro enterprises are a vulnerable sector of this practice (Heredia Montenegro, 2006)

2. Materials and Methods

2.1 Model

Input variable (independent):
- Density milk.

Modification variables (treatment):
- Amount of Calcium Chloride
- Amount of Sodium Chloride
- Amount of Rennet

Response Variable (dependent):
- Amount of fresh cheese produced in grams.
- Physical appearance
- Flavor
- Smell
- Overall evaluation (sensory)

2.2 Location

PROVINCE: IMBABURA
CANTON: IBARRA
PARISH: SAN FRANCISCO

Climatic characteristics of the sector are:
- TEMPERATURE: 18°C
- ALTITUDE: 2,217 m.s.n.m
- RELATIVE HUMIDITY: 52%
- RAINFALL: -40%
- LATITUDE: 0.354124
- LENGTH: -78.133984

2.3 Materials

- Laser Digital Thermometer
  - Temperature Range: -50°C - 330°C
  - Resolution: ±1% or ±1°C
  - Radio distance: 12:1
  - Resolution: 0.1°C / 0.1°F
- Lactodensimeter Quevenne
  - Scale: 1.015 – 1.040:
  - Resolution: 0.001 g/cm³
- Graduated Cylinder 250 ml
- Cup graduated 5 – 15 ml
- Scale with resolution 0.1 LanteScale.
  - Scale: 0.1 – 500.0 gr.
  - Resolution: 0.1 gr.
  - Maximum: 500.0 gr.
- Digital Scale 0.01 LanteScale.
  - Scale: 0.01 – 200.00 gr.
  - Resolution: 0.01 gr.
  - Maximum: 200.00 gr.
- Digital pH meter
  - Range: 0.0 – 14.0 pH
  - Accuracy: ±0.1 pH
  - Resolution: 0.1 pH
  - Automatic temperature compensation.
  - Operating temperature: 0-50 °C
- 4-liter containers.
- fine mesh strainer
- Ingredients: milk (whole), calcium chloride (powder), rennet (powder), sodium chloride.
- Kitchen
- Fridge
- Instruments several kitchen
2.4 Methods

The method of making the cheese was according to the NTE INEN 1528 standard: 2012 General for fresh cheese. Also, the density conditions revealed in milk being adulterated by adding water, simulating the problem faced by micro enterprises.

The method of tasting was based on the model proposed by Haydee, Aranibar, & Cañamer, (2005) which establishes descriptors and a scale that allows evaluation representatively variables sensory character passing the "unpleasant = 1" approach to "extremely nice = 5". In addition to following the considerations made in Murray, Delahunty, & Blaxter, (2001) which established the need for a suitable group of panelists according to the number of factors that you want to relate the estimate.

The experimental design was structured as a 2x3 factorial design, with factors calcium chloride study, rennet and sodium chloride. Respectively levels were established for the minimum and maximum value 0.1g & 0.3g, except the sodium chloride that had the minimum value and maximum 2 g & 7 g.

2.5 Population and sample

44 units as part of the pre-experimentation to define the acceptable experimental region and find appropriate measures to develop research conditions were created.

A sample of 32 units was established to work with a factorial model, free alias, without blocks, for mitigating problems of experimental resolution. For the experimental units it was added 10% water, simulating the adulteration of raw materials and the experiments were performed on a course of 8 days.

Were also generated 10 units of fresh cheese to have the reference model under normal conditions (without adding water) and evaluate model performance.

3. Results

After completing the experimental runs and tasting testing and test, the scores they were found exposed in Table 1.

<table>
<thead>
<tr>
<th>Orden</th>
<th>Corrida</th>
<th>A</th>
<th>B</th>
<th>C</th>
<th>Gramaje</th>
<th>Aspócto (Físico)</th>
<th>Sabor</th>
<th>Olor</th>
<th>YGc</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>1</td>
<td>1</td>
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<td>1</td>
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<td>2</td>
<td>1</td>
<td>1</td>
<td>1</td>
<td>1</td>
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<td>1</td>
<td>1</td>
<td>1</td>
</tr>
<tr>
<td>32</td>
<td>1</td>
<td>1</td>
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<td>1</td>
<td>1</td>
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<td>1</td>
<td>1</td>
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</tr>
</tbody>
</table>

Table 1. - Results of experiments with addition of 10% water

3.1 Weight Variable

The results are shown in Table 2.

<table>
<thead>
<tr>
<th>AJUSTES</th>
<th>RESID</th>
<th>RESS1</th>
<th>RESSE</th>
<th>AA</th>
<th>COOKIE</th>
<th>EAAJ</th>
<th>ECOE</th>
<th>EFFEL</th>
</tr>
</thead>
<tbody>
<tr>
<td>41.36</td>
<td>0.00</td>
<td>0.3528</td>
<td>0.3456</td>
<td>0.25</td>
<td>0.0015</td>
<td>0.0957</td>
<td>0.025</td>
<td></td>
</tr>
<tr>
<td>41.32</td>
<td>0.25</td>
<td>-0.0845</td>
<td>-0.0768</td>
<td>0.25</td>
<td>0.0023</td>
<td>0.0586</td>
<td>0.025</td>
<td></td>
</tr>
<tr>
<td>41.75</td>
<td>0.35</td>
<td>-0.0328</td>
<td>-0.0218</td>
<td>0.25</td>
<td>0.0018</td>
<td>0.0303</td>
<td>0.018</td>
<td></td>
</tr>
<tr>
<td>41.75</td>
<td>0.50</td>
<td>0.0845</td>
<td>0.0718</td>
<td>0.25</td>
<td>0.0017</td>
<td>0.0256</td>
<td>0.018</td>
<td></td>
</tr>
</tbody>
</table>

Table 2. - Results of design 2x3 for "weight" variable

For Table 2. Adjustments to the observed values for residuals (Column 1 and Column 2) reveal a proper model without large dispersions. Residues show the difference between the observed values and the predicted values, this reveal the level of variation covered for model.

The RESS1 column represents standardized residuals vs Studentized residuals, which if they exceed the values +2 & -2.
or -2 show that there are outliers. Within column 3, you do not have any value with this feature of study. Thus, it is considered that there is no difference between the variances of the data.

The column rész-1, shows residues t eliminated from the previous column in the same way used to identify outliers considering the relationship between the residue and removed the estimated standard deviation.

Column 5 shows the leverage (AA1). That is, the distance between the value for each observation to average the data, between 0 and 1. The value for all observations is 0.25 so is concluded that there are no unusual observations or disproportional model.

Considering the standardized with leverage residue has Cook's distance, which reveals the criteria of significant or not, the values of influential observations for a regression model. As noted in column 6, the experimental runs have a similarity with each other little variability.

The DFITS column called EAJTE1 considered the standard deviation by eliminating an observation and how this would change to determine if Cook is misleading when observation than twice the square root of a relationship between the total number of factors and experimental runs. With a value of 1.0, not have errors degenerating the model runs.

The COEF1 and EFFE1 columns show the coefficients and the effects for the model.

With the analysis of weight can understand the effects of the factors studied, as shown in Illustration 1.

The Pareto diagram reveals that the effects of rennet (B) and calcium (A) chloride are relevant to the model analysis.

Illustration 1. - Pareto of Standardized effects

Illustration 2. - Principals Effects for weight in grams

The illustration for graphic effects reveals that calcium chloride and rennet have relevance on the resulting weight. Increasing quantity of rennet, results increased considerably in response. In addition, it can be seen that sodium chloride has no effect on the result.

Illustration 3. - Interactions for weight

Illustration 3 in its first box calcium * rennet, Don't indicate interaction between factors, however, the parallelism reflects calcium chloride is beneficial and contributed to increased resulting grammage.

Summarizing, the model adjusted explains 98.35% of the variability of weight. The estimated standard error shows that there is a deviation of the residuals 1.63. The average absolute error is 114.68, however, this error is irrelevant in the study because the variation in the resulting weight is not by failures in the process, but by changes in factors in form aforethought, and they altered the response variable.

Additionally, Durbin Watson indicator is 1.56 with P = 0.1288 indicating that there is no serial correlation, with a confidence level of 95%. Indicating that the test and the selected model are correct.

Of the coded coefficients can be set confidence intervals for common samples based on a distribution T. The V.I.F (value variance inflation) value is 1.00, which indicates that is the orthogonal model.
With the model for predicting performance for weight, was obtained the equation describing the behavior of the variables:

\[ 97.059 \pm 0.82 \]

was analyzed a factor over another by the method of maximum climb path, as shown in Table 3.

<table>
<thead>
<tr>
<th>Factor</th>
<th>Predicción para</th>
</tr>
</thead>
<tbody>
<tr>
<td>Factor A</td>
<td>Factor B</td>
</tr>
<tr>
<td>(CACLID)</td>
<td>(CUZIO)</td>
</tr>
<tr>
<td>0.0</td>
<td>0.0</td>
</tr>
<tr>
<td>1.0</td>
<td>2.86455</td>
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<tr>
<td>2.0</td>
<td>-10.31</td>
</tr>
<tr>
<td>5.0</td>
<td>-9.3339</td>
</tr>
<tr>
<td>5.0</td>
<td>-7.34877</td>
</tr>
</tbody>
</table>

Table 3. - Maximum climb path

This calculation showed that the behavior of one of the variables might be nonlinear.

Optimization of the response through a desirability function, constituted the model:

<table>
<thead>
<tr>
<th>Respuesta</th>
<th>Meta</th>
<th>Inferior</th>
<th>Objetivo</th>
<th>Superior</th>
<th>Fondezación</th>
<th>Importancia</th>
</tr>
</thead>
<tbody>
<tr>
<td>Oframaje</td>
<td>441</td>
<td>441</td>
<td>1</td>
<td>1</td>
<td>1</td>
<td></td>
</tr>
</tbody>
</table>

Table 5. - Maximum climb path

Using as input the minimum value (411) and maximum (441) of the adjusted model, there are three possible solutions with different combinations of the three factors of study. With the first solution shows a result of 440.750 grams and desirability of 99.17%.

The second solution is hypothetical value in the Curd factor as its practical measurement is unconventional. No possible measurement, the level of resolution and contain the same result maximization of 440.750 grams.

The third solution maintains maximum values and factors Rennet and Calcium; however, Sodium factor is reduced to 2.01 g, lowering the desirability composite design to 439.73. The value optimization was 440.75 grams with setting error in 0.82.

The intervals represent the minimum and maximum optimization considered for standard deviation and confidence for the prediction uncertainty. Thus, the design can increase to 437 grams per unit of fresh cheese produced being unfavorable and considering 445 grams an optimistic scenario.

To find the best treatment, HSU MCB test was performed, as shown in illustration 4.

Illustration 4. - MCB of Hsu test

The test is concluded that treatments T4-T8, T4-T8 have significant differences maximization.

3.2 Appearance Variable

The results of this variable were obtained from the grades given in the tasting test.

Illustration 5. – Weigh vs Appearance

In illustration 5, can be noted that the T5, T1, T3, T7 and T8 treatments are not adequate if these two variables are related, therefore, the object of analysis focuses on the remaining treatments.

For the T6 treatment, it is observed that the best appearance scored is 178 points (x10), however, the amount obtained cheese was not the most suitable if other treatments are observed.

T2 treatment reflects a value of 172 points (x10) for the "appearance" and 1722 grams of cheese produced (only 6 grams of treatment T6).
Whereas the T4 treatment expresses a high value for grams resulting from cheese and a score of 170 (x10) is also a treatment to be considered as acceptable.

With the values of T2, T6 and T4 treatments can set parameters optimization.

<table>
<thead>
<tr>
<th>Parameters</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Resolución</strong></td>
</tr>
<tr>
<td><strong>Calcio</strong> 0.3, <strong>Chorizo</strong> 0.3, <strong>Socio</strong> 7</td>
</tr>
<tr>
<td><strong>Ajuste</strong> 440.750, <strong>Deseabilidad</strong> 93.38%</td>
</tr>
<tr>
<td><strong>Solution 1</strong></td>
</tr>
<tr>
<td><strong>Calcio</strong> 0.3, <strong>Chorizo</strong> 0.3, <strong>Socio</strong> 7</td>
</tr>
<tr>
<td><strong>Ajuste</strong> 440.750, <strong>Deseabilidad</strong> 93.38%</td>
</tr>
<tr>
<td><strong>Solution 2</strong></td>
</tr>
<tr>
<td><strong>Calcio</strong> 0.3, <strong>Chorizo</strong> 0.3, <strong>Socio</strong> 7</td>
</tr>
<tr>
<td><strong>Ajuste</strong> 440.750, <strong>Deseabilidad</strong> 93.38%</td>
</tr>
<tr>
<td><strong>Solution 3</strong></td>
</tr>
<tr>
<td><strong>Calcio</strong> 0.3, <strong>Chorizo</strong> 0.3, <strong>Socio</strong> 7</td>
</tr>
<tr>
<td><strong>Ajuste</strong> 440.750, <strong>Deseabilidad</strong> 93.38%</td>
</tr>
</tbody>
</table>

Were generated three initials solutions with the specified parameters, which can be considered optimal one and two with a weight of 440.750 and 439.757 g respectively.

The level of desirability for response 1 is of 93.38% and solution 2 reaches 90.46%. In addition, can be noticed that solution 3 deteriorates as the optimum response range achieved only 432.264 grams of fresh cheese on a combination of factors 68.43%.

The optimized response therefore would find a confidence interval of 439.058 to 442.442 gram, while the probability interval be extended from 436.967 to 444.533 grams.

### 3.3 Flavor Variable

The results for this variable were obtained from tasting test, and to make a comparison the weight response was established, as detailed in Illustration 6.

By linking the variables weight and flavor, can be discarded T5, T1, T3, T7 and T2 treatments as these have a low score and the amount obtained from fresh cheese is not the best. Moreover, the T4 and T8 treatments show acceptable results, T8 being best placed with a score of 170 grams and 1763 fresh cheese produced.

The T4 treatment, has 1759 grams of fresh cheese, however the score achieved for the flavor got only 164 points.

The T6 treatment scoring is 168, being 2 points better treatment regarding the analysis of the flavor, however the resulting amount of cheese reached only 1725 grams.

Whereas the best treatments according to the flavor optimization, 440.75 grams, occurred in conjunction with the exposed for appearance.

### 3.4 Smell Variable

The results for this variable was derived from tasting tests shown in Illustration 7.

In Illustration 7, shows as better treatment T8, with a score level 168 points and achieved an amount of fresh cheese grams 1763.

Considering the proximity of T4 for its weight (1759 g) could reflect an acceptable value, but the loss of 4 points (x10) in their assessment of the smell, was discarded it as an appropriate treatment.

The T6 treatment recovers score in the rating of the smell, leaving only 2 points (x10) for the best rating; however the amount of cheese achieved regarding T8 causes a loss of 38 grams in four experimental runs.

T5 and T2, T1, T3, T7 treatments are clearly unsuitable both the resulting weight and in relation to the variable “smell”.

Optimizing the answer in this case again coincide 440.75 grams for a bilateral test at 95% confidence.

### 3.5 Overall variable

This variable reflects the general impression getting the product; these results are detailed in Illustration 8.
According to the trend in Figure 8, the T8 treatment has the best rated with 170 (x10) and obtained a weight of 1763 g. If you take into consideration that the general assessment represents as conceived from a global context to fresh cheese produced, this treatment reflect the best experimental composition.

Treatment T4 recedes in weigh 4 grams in four experimental runs, however, the weight given generally for this group of experimental units declined 4 points (x10), which is not favorable when referring to T8.

T6 treatment, approaches in its overall assessment score being 1 point (x10) above T8, however, the amount of cheese produced was reduced 38 g in 4 experimental runs, their suitability considering was removed.

T5, T1, T3, T7 and T2 treatments not have the best rated both the weigh obtained as in the rating given for variable "overall evaluation".

The optimal value, was found in a confidence interval of (439.058 to 442.440) grams with the nominal value of 440.750 g. This analysis for a bilateral test of 95%. Finally, we can say that the response of common optimization between the variables analyzed is the probability interval lower limit 436.967 and 444.533 for the upper limit, the magnitude of which is expressed in grams of fresh cheese.

### 3.6 Validation of measuring instruments

To ensure reliability in the results an R&R test was applied to the scales used in research with standardized dimensions and weight, to determine the variability of instruments.

R-01 for the scale used in the measurement of weight, the results are shown in the figure 9.
In this study, a yield of 7.7% considered acceptable, and a resolution of 4.7% was found suitable to accept that the data are accurate.

3.7 Proposal

With the results of the investigation, the best standard set was to raise factors and rennet calcium chloride to achieve better performance, if exist adulteration in milk up to 10% water addition.

It was proposed a raise 0.3 grams for calcium and rennet chloride values and reject the use of high doses of sodium chloride, since as demonstrated in the study this factor is not representative of the expected yield of fresh cheese.

4. Conclusions

The pre-testing of 44 units with different levels for factors change was made, discriminating treatments unsuitable for investigation.

It was revealed that the percentages of adulteration in milk less than 30% aggregation of water are not suitable for cheese making, because, the paste fails compacted and the process of pressing when They withdrew molds, the cheese loses its original form.

With additional water (10%), curd was consistent and could be transformed into cheese, however when tested with more than 0.3 grams for calcium chloride and rennet, the amounts of the final product was yellow colour and completely bitter, and cheese was presented anomalies in their physical structure, with still clearly visible cracks and brittleness.

In experiment was created 32 samples with two levels in the study factors whose values for the minimum level was 0.1 grams for both calcium chloride and amount of rennet, and 2 grams for sodium chloride. Likewise, the values for the maximum value of 0.3 grams for calcium chloride and rennet, and 7 grams for sodium chloride were taken.

In analyzing the data obtained, was revealed that the effect of sodium chloride factor (C) was not significant for the increase or decrease of the weight of fresh cheese.

The amount of rennet proved to be the most representative effect to change the amount resulting weight and the calcium chloride if it contributes to better performance but minor.

The graphic for principal effects revealed that the significant factors had positive slope, being predominantly rennet. In addition, when checking the interactions between “rennet * calcium” was observed parallelism so it was concluded that there was no interaction between factors. However, for this parallelism was a remarkable difference with what it was concluded that the addition of calcium chloride favored positively and significantly, increasing the expected weight.

This lack of interaction showed why can be prepare fresh cheese without the obligation to add calcium chloride and argues that the addition of this element is not use to produce cheese, but help to replenish the lost calcium in milk in the process of pasteurizing, improving performance.

After checking the statistical assumptions, the weight and its relation to qualitative variables appearance, taste, smell and overall assessment, was verified that treatment T5 (0.1; 0.1; 7) was thoroughly with the worst to have an answer of 1,643 grams and an assessment of 160 points for appearance, flavor and smell to 162, and 164 in the overall rating. It was also determined that the best treatment is the T8 (0.3, 0.3, 7) with an answer of 1763 grams and a score of 168 points for appearance, smell 168 and 170 for taste and overall assessment.

Finally, was established the proposed of regulation in factors of coagulated, when there is a maximum of 10% adulteration by adding water in milk, which is to raise levels of calcium and rennet chloride 0.3 grams in each unit of referential production 480 g.

With the conditions of the experimental model, it was concluded that it is possible to compensate weight performance, when there adulteration in the raw material. It
was achieved to optimize this variable to 91.75%, which represents an improvement of 6.61% referencing the worst working conditions, and 5.12% on average conditions for cheese making.

Thanks
For all, for your confidence.

Bibliographic references


**About the Author**

Christian Rosero was born on December 13, 1988. He made the secondary studies at the High School UTN, and higher studies at the Técnicas del Norte University, Ibarra - Ecuador.