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CRYOSCOPIC INDEX OF CHILLED RAW MILK PRODUCED IN THE MICRO-REGION OF JI-PARANÁ - RONDÔNIA - BRAZIL

ÍNDICE CRIOSCÓPICO DE LEITE CRU REFRIGERADO PRODUZIDO NA MICRORREGIÃO DE JI-PARANÁ – RONDÔNIA - BRASIL

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ABSTRACT
The cryoscopic index is a physical-chemical parameter that determines the freezing point of milk, being mainly used to detect fraud by adding water to the milk. The research aimed to analyze the cryoscopic index in samples of a dairy under the seal Federal Inspection Service located in the micro-region of Ji-Paraná – Rondônia - Brazil. 279 samples were analyzed during a 31-day period (05/16/2017 to 06/16/2017). For the analysis of the data, a statistical test of quality control was applied to verify the stability and capacity of the processes to meet the specifications defined by the current legislation. The results indicated that 16.4% of the samples were outside what the current legislation determines. The results of the cryoscopic indexes observed in the microregion of Ji-Paraná demonstrated the importance of the analysis of platforms recommended by Brazilian legislation, making the inspection for this type of fraud efficient, detecting adulterations.
Keywords: Milk fraud; Freezing point; Decree 9,013

RESUMO
O índice crioscópico é um parâmetro físico-químico que determina o ponto de congelamento do leite, sendo principalmente utilizado para detectar fraude por adição de água no leite. O trabalho objetivou analisar o índice crioscópico em amostras de um laticínio sob chancela Serviço de Inspeção Federal localizado da microrregião de Ji-Paraná – Rondônia. Foram analisadas 279 amostras durante o período de 31 dias (16/05/2017 a 16/06/2017). Para a análise dos dados foi aplicado um teste estatístico de controle da qualidade na verificação da estabilidade e capacidade dos processos em atender a especificações definidas pela legislação vigente . Os resultados indicaram que 16,4% das amostras estavam fora do que determina a legislação vigente. Os resultados dos índices crioscópicos observados na microrregião de Ji-Paraná demonstraram a importância das análises de plataformas preconizadas pela legislação brasileira, tornando a fiscalização para esse tipo de fraude eficiente, detectando adulteração.
Palavras-chave: Fraude no leite, Ponto de congelamento, Decreto 9.013

INTRODUCTION

Milk is one of the most important products of the national agribusiness, generating jobs and income. Brazil is the 5th largest world producer of milk, behind only India, the United States of America, China and Pakistan (FAO, 2017; DIAS, 2017). In human nutrition, milk is one of the main sources of calcium, also providing essential proteins and minerals (EMBRAPA, 2016; FAO, 2017).

Due to its perishable nature, it is essential that the systematic control of milk quality is carried out through physical-chemical and microbiological assessments in order to meet the minimum quality requirements set by current legislation (PANCOTTO, 2011). Therefore, milk is monitored by public health agencies through physical-chemical tests to investigate possible deviations in composition or fraud, which can occur for several reasons, among them, the economic (PEREIRA, 2008; ULE et al., 2016).

According to Decree 9.013 of March 2017 (BRASIL, 2017): “raw materials or products that show adulteration or falsification are considered to be fraudulent ...”. According to this legislation, the following can be considered as cases of adulteration: “raw materials and products with the addition of ingredients, additives, technology aids or substances of any nature with the aim of concealing or hiding changes, deficiencies in quality of raw material, defects in the elaboration or to increase the volume or weight of the product ”.

Among the physical-chemical analyzes to determine economic fraud in milk, the cryocopic index stands out, which corresponds to the freezing point of milk and indicates whether water has been added, its reference value being established by Decree 9,013 and also the addition of reconstituents density (BRASIL, 2017).

In view of the justifications, the aimed of the research was to determine the cryoscopic index of milk samples from a dairy in the microregion of Ji-Paraná - Rondônia - Brazil, in order to verify possible fraud.

MATERIAL AND METHODS

279 samples of refrigerated raw milk from isothermal tank trucks, with a capacity of nine thousand liters, were analyzed in a dairy with the Federal Inspection Service (SIF), located in the municipality of Teixerópolis, Rondônia - Brazil. The collection took place between 04/16/2017 and 05/16/2017, with nine tank trucks being sampled daily at the time of receiving the milk, as established by Decree 9,013 of March 2017. The results were recorded, complying with the program rules of self-control implanted in the industry.

Sample Collection

The refrigerated raw milk was collected in each compartment of the tank trucks from producers in the micro region of Ji-Paraná – Brazil, namely Teixeirópolis, Mirante da Serra, Nova União and Ouro Preto do Oeste. Some of these producers had an individual cooling tank and others used a collective tank, with 50 producers with an individual tank and 150 producers with a collective tank. The analyzes were carried out in the physical and chemical analysis laboratory of a Dairy with a Federal Inspection Service, shortly after receiving the milk.

Analytical Methodology and Determination of the Cryoscopic Index

The methodologies used in this work were performed according to the Manual of Official Methods for Analysis of Food of Animal Origin from the Ministry of Agriculture, Livestock and Supply (MAPA, 2018).

A portable digital electronic cryoscope, model MK 540 L, brand ITR, was used. At each sampling, the refrigerant solution was checked and the equipment was calibrated using standards of 0.000º H and -0.621ºH. Once the equipment was calibrated, 2.5 ml of milk was read from each sample in a cryoscopic tube, in triplicate.

For data analysis, the statistical test of quality control was applied to verify the stability and capacity of the processes to meet specifications pre-defined by legislation. Afterwards, control charts for averages and amplitude were built for the nine samples collected each day and analyzed at the time of receiving the milk.

For data analysis, a statistical test was applied using the free software R Studio (R CORE TEAM, 2017) using the function package for statistical quality control, qcc (SCRUCCA, 2004).

RESULTS AND DISCUSSION

The collected data provided an average temperature of -0.537º H and a standard deviation of 0.016º H in relation to the average. One sample showed -0.616º H and another showed -0.457º H, as minimum and maximum values allowed, respectively as explained in the graphs of Figure 1, related to the control chart for mean and amplitude. Figure 1 shows the specifications No. RIISPOA (2020) and Normative Instruction 76 (2018), in the control charts for average. In this work it is possible to observe the existence of special causes for values outside the control limits (marked in red), influencing the freezing temperature.

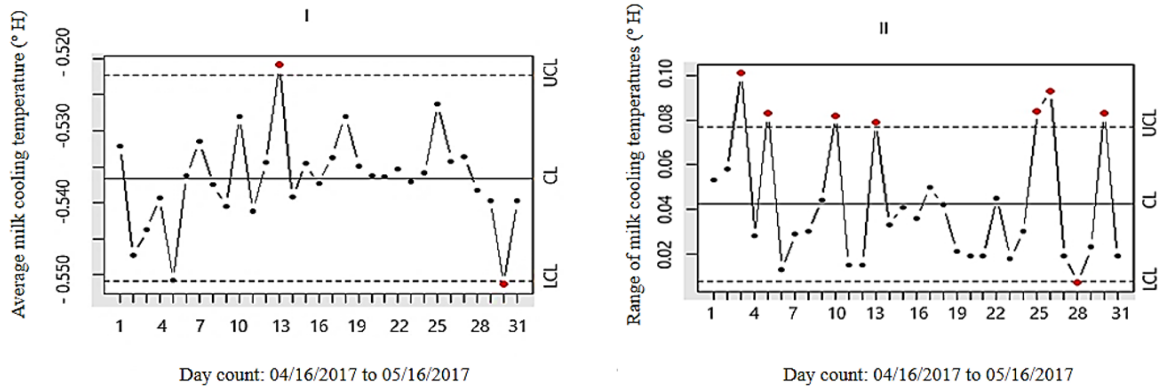

Figure 1. Control charts for freezing temperature of milk: I) Control chart for average II) Control chart for amplitude.

According to Arcari and Santos (2012), the cryoscopic index may show variations in ranges outside the standards acceptable by the legislation due to factors other than fraud by adding water. These variations can be caused by factors such as race, diet quality, drinking management, lactation stage, milk composition, season and geographic region, which was also mentioned by Buchberger (2000) and Senevirathn et al. (2016). This would explain the variation on the freezing point in different countries (ZAGORSKA and CIPROVICA, 2013; ULE et al., 2016). However, Fagnani et al. (2014) state that these slight variations do not cause changes in the freezing point of milk.

In addition, changes can occur due to external factors, such as the presence of water in the lines of milking equipment or any other type of manipulation that leads to the dilution of the product with water (BUTTEL, 2008). Still in Figure 1, it can be seen that the process detected that in two days there were special causes for values outside the control limits. Analyzing the control chart for amplitude, it is observed that the process presented eight days of special causes leading to values outside the control limits, that influenced the variability of the process.

For comparison, Figure 2 shows the control charts for the average between the limits of the legislation. It is observed that there was a frequency of about 40% of the experimental period, that is, there were four days of violation of the limits established by RIISPOA (2020) and IN 76 (2018). Therefore, the graphs represent an out-of-control process, since it was necessary that all points were within the established limits.

The capacity study for milk cryoscopy indexes (Figure 3) was carried out, despite the non-stability of the parameters. To assess the potential of the process, values outside the

control limits were withdrawn. Table 1 contains the control statistics after the withdrawal of the values outside the control limits.

Table 1. Comparison between the capacity of the process by RIISPOA (2020) and IN 76 (2018) for the cryoscopy rates in milk from the microregion of Ji-Paraná – Rondônia – Brazil.

Control statistics	Decree 9.013
Number of observations	189
Average (ºH)	-0.5363
Standard deviation (º H)	0.0094
Lower limit specification (º H)	-0.555
Upper Limit Specification (º H)	-0.530
Value outside the law (%)	16.4
Expected value outside the legislation (%)	27.64
Cp[1]	0.4412
Cpk[2]	0.2221

[1]Cp - potential process capacity; [2]Cpk - effective process capability.

Based on Figures 2 and 3, it is possible to observe cryoscopy variation, as it is observed that the estimated Cp value (potential process capacity) was approximately 0.4412 for Decree 9.013. Thus the cryoscopy oscillation was high, it may be an indication that there are points to be corrected in production, collection and/or transportation. Montgomery (1997) points out in his research that (Cp<1) it is an incapable process. This is also seen in the Cpk value, which measures the effective capacity of the process, which is less than 1 (0.2221) for both legislations.

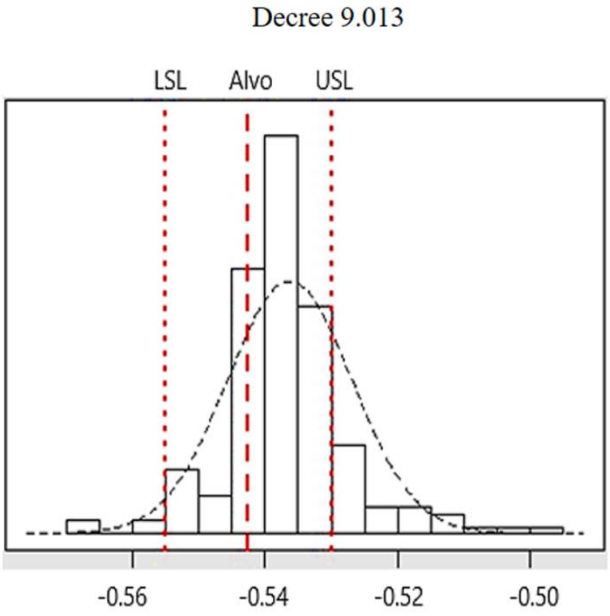

Figure 2. Comparison of specification limits with average control charts.

In addition, analyzing the number of indexes outside of what determines the RIISPOA, (2020) and IN 76 (2018), it is estimated that for every one million collections, approximately 276,442 thousand out-of-specification milk samples are expected between -0.555 and -0.530º H for freezing of milk (cryoscopy), which represents approximately 27.64% of expected violations of the Decree, equivalent to 2.38% below the lower limit and 25.27% above the upper specification limit. The control charts have the ability to verify non-conformities and the maximum and minimum limits for cryoscopy in bovine milk, with the importance of inspecting to monitor a process, improving quality and, in the case of work, checking the process in compliance with the current legislation.

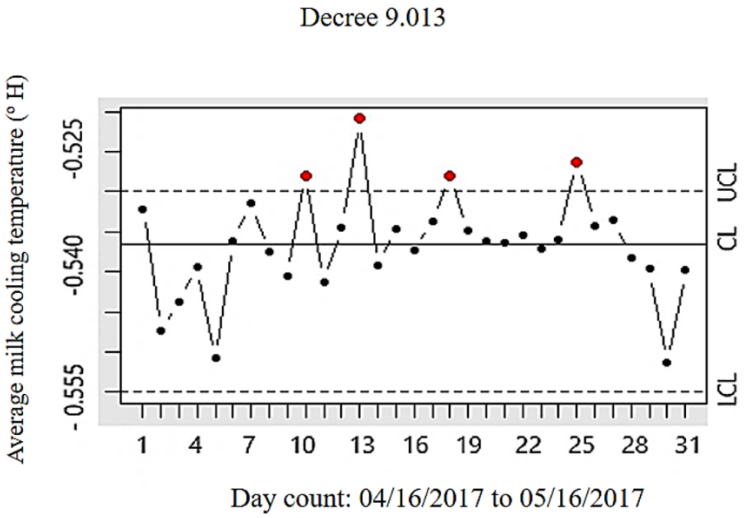

Figure 3. Milk cryoscopy index, cooling temperature on different experimental days.

Amorim (2017), in a study carried out in the Federal District and surroundings, from March 2015 to August 2016, evaluating samples of formal raw milk found that 5.5% of a total of 18 samples were at odds. However, Ribeiro Junior et al. (2013), in a study in Ivaiporã - PR (Brazil) with chilled raw milk, found that 25.66% of a total of 74 samples evaluated, had a cryoscopic index outside the legislation, a value similar to that found in this study.

According to Bisognin et al. (2016) observed compliancewhen evaluating the quality of raw milk in a medium-sized dairy located in the Northwest Region of Rio Grande do Sul (Brazil) during the first semester of 2016, even with the heterogeneity of the milk sampling performed, verifying an average of -0.540 ° H. A result similar to that was found by Castro (2015), who found an average of -0.542 ° H in a survey conducted with raw milk in the Taquari Valley, Rio Grande do Sul (Brazil), remaining within the current legislation.

Almeida (2016), when collecting samples directly from the individual tanks of fifteen rural properties that delivered milk in two dairy products in the municipality of Cacoal – RO (Brazil), verified only two properties outside the current legislation, representing 13.3%. Melo et al. (2018), in a study of raw milk sold in commercial establishments, in the state of Paraíba (Brazil) in the months of June and July 2013, found that 92% (23/25) were outside the cryoscopy standard, being higher than -0.530 ° H. Collaborating with the result obtained by Melo et al. (2018), Medeiros (2017), evaluating the quality of the milk used in artisanal cheese shops in Rio Grande do Norte and Paraíba (Brazil) obtained 88% of the samples outside the cryoscopy standard, below -0.550º H.

However, Gasparini (2018), in studies in the state of Paraná (Brazil), obtained only 0.32% of samples outside the recommended by the legislation, representing a single sample (-0.529º H). However, in studies developed by Beltrão (2018), the authors obtained 20.21% of the results outside the recommended quality limit. This can be explained by the addition of alcohol together with water to mask the fraud, however, if used in large concentrations it prevents the freezing point of the sample from being read by the cryoscope, recording only "error".

CONCLUSIONS

The results indicated that 16.4% of the samples were outside what the current legislation determines. The results of the cryoscopic indexes observed in the microregion of Ji-Paraná demonstrated the importance of the analysis of platforms recommended by Brazilian legislation, making the inspection for this type of fraud efficient, detecting adulteration.

To this end, as determined by the supervisory and regulatory body mentioned by RIISPOA (2020) and IN 76 (2018), properties of the composition should also be measured, such as: total solids (ST), separating them into lipids and non-greasy solids; the total dry extract (EST), which comprises all elements of milk, except water and defatted dry extract (ESD), to generate more accurate information about possible milk fraud.

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