International Journal of New Technology and Research (IJNTR)
ISSN:2454-4116, Volume-5, Issue-5, May 2019 Pages 19-23

Quality of Raw Milk Delivered by Rural Producers for a Dairty Factory in the Southwest Region of Paraná, Brazil

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***Abstract*— Milk is a natural food, rich in several nutrients which help in the development of organisms and functions as nutrition for most species. In order for this food to guarantee its nutritional value, it must follow some physical, chemical and microbiological standards. In this work, the physical-chemical quality of the milk produced in the city of Saudade do Iguaçu, Paraná, was evaluated, delivered to a large dairy factory for food production. Fifteen samples of raw milk were collected, which were submitted to analysis of acidity, fat, cryoscopy, density, presence of antibiotic, alkaline substances, chlorides and starch. Comparing the results obtained to the standards of the Normative Instruction 51/2002, it was confirmed that the densities of the samples were normal. For acidity, there was a non-standard result (sample 11); in the cryoscopy there were two results that did not fit the parameters (sample 1, sample 11), and for fat two samples had a high value (sample 8, sample 11). Samples 1 and 11 were not suitable for production and consumption of dairy products, and proper disposal of samples was necessary. Most of the samples presented satisfactory results according to the legal standard of the federal health legislation of milk quality.**

***Index Terms*— Physical-chemical properties, Quality of the Milk, Quality standards.**

I. INTRODUCTION

Milk is considered to be one of the most complete foods that exist because it has a high nutritional value, fundamental for the human diet; it is source of proteins, lipids, carbohydrates, minerals and vitamins [1]. According to the Normative Instruction - IN 51/2002 of the Ministry of Agriculture, Livestock and Supply (MAPA), milk is understood to be the product of complete and uninterrupted milking, under hygienic conditions of well fed and rested [2] healthy cows.

Being an animal food and being in direct contact with the environment, it is necessary for the producers to perform some care before delivering it to the dairy factory. Attention should be paid to the hygiene and health of the handler (who will milk) as well as of the animal (udders and teats), hygiene, cleaning and sanitizing of equipment, proper handling of milking and refrigerated milk storage until delivery to dairy factory. The milked animal care is of great importance, since it jeopardizes future quality of the product. Therefore, it is essential to ensure its integrity including the treatment of infections with specific antibiotic, which may leave residues when used after the grace period [3].

The quality of milk can be affected when there is withdrawal or addition of some of its compounds, characterizing the action as an attempt to fraud, characterizing the food as not safe, which does not pose a risk to consumer health [4] .In this way, the physical-chemical and hygienic-sanitary analyzes must be carried out to evaluate the characteristics of the milk, guaranteeing to the consumer the absence of adulterations and with quality as required by Law IN 51/2002, referring to the acceptability and classification of normal conditions of the milk [5]. It should be noted that this normative instruction is being replaced this year in Brazil with IN 76, 77 and 78.

In this context, the presence of antibiotics in milk has been one of the biggest problems for the food industry, as they interfere in the manufacture of some dairy products, impacting the consumers health. Another major problem faced, specifically in dairy factories, is the addition of water to the milk, decreasing the yield in the manufacture of the byproducts [3].

Among other possibilities of fraud, it is possible to emphasize alteration in the acidity. If the milk expresses acidity lesser than 14° D (Dornic), it suggests fraud with some alkaline substance. On the other hand, if the acidity is higher than 18°D, the indication is acidification of lactose, possibly caused by the presence and multiplication of microorganisms. Verification of milk density is part of the practical inspection process and adds useful and fast information, although it is not an entirely decisive test. It may raise suspicions about the condition of skimmed or watery milk, which in most cases are confirmed as fraud [6].

Milk and its byproducts are products of daily consumption and, therefore, it is very important to check the quality of the raw milk that arrives at dairy factories. In this sense, the physical-chemical analysis contributes significantly to the

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identification of possible fraud, avoiding that low quality product does not reach the industry and the consumers. The objective of this work was to verify the physical and chemical characteristics of raw milk produced by dairy cows received by a dairy factory located in the city of Saudade do Iguaçu - Paraná, in order to detect possible food fraud, which contravenes the assured standards by Brazilian legislation.

II. Material and Methods

A. Sample, transportation and packaging

Sampling of raw milk was obtained directly from the trucks, with three isothermal tanks in bulk each and with a total capacity of 8,400 liters per truck, which take the milk from approximately 30 producers to the industry. These producers and suppliers integrate five communities in the interior of Saudade do Iguaçu, classified in this work as: Community A (samples 1, 2 and 3); Community B (samples 4, 5 and 6); Community C (samples 7, 8 and 9); Community D (samples 10, 11 and 12) and Community E (samples 13, 14 and 15).

During transportation to the endpoint the milk is mixed in the truck tank. Soon, when arriving to the dairy factory it is informed from which communities and producers the milk was obtained. It should be noted that the volume of milk provided by each producer is small, which justifies the mixing. And if any change in the raw material is observed, all supplier producers will be notified, without exception.

In total, raw milk from five trucks was collected, each with three tanks and each vehicle representing each of the five communities. A sample of milk was obtained from each tank, in a total of 15 samples of raw milk, identified in a scale of 1 to 15. The collection of each milk sample was performed after stirring the milk inside the tanks of the trucks with the aid of cleaned (sanitized and sanitized) and dried flasks so that there was no change in the milk during the analyzesSubmit your manuscript electronically for review.

B. Physical-chemical analyzes

The physical-chemical analyzes were: acidity and alkaline content, water and fat content, antibiotic test, density analysis, chloride test, starch and alkaline substances.

i. Analysis of the acid content

This analysis was performed by titrimetry using a Cap Lab brand acidometer [4] using a volumetric pipette. 10 mL of the milk sample was transferred to a 50 mL beaker, and then five drops of 1% phenolphthalein (m/V) were added. The titration was performed with 0.11 mol/L sodium hydroxide solution (Dornic Solution), until the appearance of a persistent pink coloring for approximately 30 seconds. Each 0.1 ml of the Dornic solution corresponds to 1°D which corresponds to 0.01% acidity expressed as lactic acid. According to IN 51/2002 [2] the normal milk acidity pattern may vary from 14°D to 18°D.

ii. Analysis of the water content - Cryoscopy

The cryoscopy test [8] was performed using a PZL 7000 cryoscope. 2.5 mL of milk was put in a cryoscope tube, and then the tube was placed at the appropriate site of the cryoscope and analyzed. Within two minutes the result appeared on the display. In the equipment the milk is cooled to -3ºC, followed by immediate crystallization by mechanical vibration producing heat release and elevation of milk temperature, reaching *plateau* corresponding to the freezing point. Small amounts of water added cause the freezing point to rise, that is, to approach the freezing point which is equal to 0°H. Therefore, if the cryoscope reading is between -0.530°H and 0.529, the result may indicate milk fraud by watering. If the value obtained is lower than -0.550°H it may indicate high acidity or fraud by addition of salt [5]. The normal value for cryoscopy varies from 0.530 to 0.548°H, and the freezing point of the milk is read in degrees Hortvet [9].

iii. Fat Content – Gerber butirometer method

The determination of the fat content was carried out using the Gerber buberometer method [5]. The analysis consisted of adding 10 mL of specific sulfuric acid at 1.825 g/mL concentration in the butyrometer, slowly incorporating 11 mL of milk (so that the mixture with the acid did not occur) and 1 mL of isoamyl alcohol. Then the butyrometer was closed with a suitable stopper and was stirred to promote complete mixing of the liquids. The butyrometer was then centrifuged at 1200 rpm for 5 minutes at room temperature. After centrifugation, the butyrometer was placed in water bath for 5 minutes and the fat content was read in % (m/v) directly on the butyrometer scale. The average percentage of fat for bovine milk is 3.5%. When the fat content is not changed, the milk is considered as whole, if it has fat content between 0,6 and 2,9% the milk is considered semi-skimmed, and if the content is lesser than 0,5%, the milk is classified as skimmed [10].

iv. Antibiotic residue testing – Snap method

The Snap analysis was done using IDEXX® brand Snap kit [5]. This kit shows positive or negative result. When negative, the milk sample is the same color or darker than the control, otherwise it will be positive [8].

v. Density analysis

The milk density was determined by the use of a Cap Lab brand thermolactodensimetres [6] which are calibrated at a temperature of 15°C. 250 mL of milk was slowly transferred to the beaker so no foaming was formed, and then the thermolactodensimetre was immersed to evaluate the sample. At 15°C the milk density varies from 1,028 g/L to 1.035 g/L, with a mean of 1.032 g/L and represents the sum of the intrinsic density of all its constituents [2]. According to IN 51/2002, the density standard is 1028.0-1035.0 g/L [2].

vi. Chloride test

According to IN 51/2002, the presence of chlorides in milk samples is not allowed [2]. The methodology of the chlorides test [5] seeks to verify if there is residue of this origin in the milk. 10 ml of milk was placed in a test tube, then 5 ml of Silver Nitrate was added slowly through the wall of the tube, and then 5 drops of 5% Potassium Chromate were added. This mixture was stirred and observed if there was some change in the solution color. If the result is positive for residues, the color will be orange or yellow; if negative the color will be dark brown (brick).

vii. Starch test

The purpose of the starch test [11] is to check for starch in milk. 10 mL of milk was placed in a test tube followed by water bath at 38°C for 5 minutes. Soon after, two drops of the

dye lugol (2% iodine) were added. If the coloring does not change or if it becomes slightly yellow, the result is negative for the presence of starch, in contrast, if the coloring turns blue, the result is positive [11]. The legislation provides that starch tests must be negative, and if fraud occurs for positive results, the contaminated milk should be discarded [2].

viii. Test for alkaline substances

The test for alkaline substances is to check if there is any alkaline substance (such as sodium hydroxide, sodium bicarbonate) in the milk. The analysis followed the protocol established by Pancotto (2011) [5], where in a tube with 5 mL of milk was added 5mL of alcohol ether ketone. After mixing, if lump formation occurs, the result is negative for the presence of alkaline substances. On the other hand, if there is no formation of lumps, the sample remains liquid characterizing the result as positive. According to IN 51/2002 [2], samples of fraud milk with some alkaline substance, the milk should be considered unsuitable for consumption

III. Results and Discussion

The results found in the study indicate 3 samples with irregularities from 15 collected. Sample 1 had a freezing point value (0.510°H) below the permitted; sample 8 presented fat value (4.2%) above what is allowed, and sample 11 presented acidity (25°D), cryoscopy (0.555°H), and fat (4.2%) outside the legal standards established by IN 51/2002, as can be observed in Table 1.

Table I - Acidity values (°D), cryoscopy (°H), fat content (%) and density (g/L) from the 15 raw milk samples.

	Acidity (°D)	**Cryoscopy (°H)**	**Fat (%)**	**Density (g/L)**
Samples	14-18	0.530-0.548	3.0-4.0	1028.0-1035.0
1	16	0.510*	3.6	1030.4
2	15	0.535	3.9	1028.6
3	16	0.536	3.8	1030.0
4	16	0.540	3.6	1031.4
5	15	0.542	3.9	1030.2
6	15	0.538	4.0	1031.2
7	15	0.530	3.9	1029.8
8	16	0.546	4.2*	1031.4
9	16	0.536	4.0	1029.6
10	15	0.539	3.6	1032.2
11	25*	0.555 *	4.2*	1030.6
12	18	0.546	3.9	1031.8
13	14	0.541	3.8	1030.6
14	15	0.542	3.7	1030.4
15	18	0.548	4.0	1031.2
Legal standard	14-18	0,530-0,548	3,0-4,0	1028.0-1035.0

*Samples with results not fulfilling the values allowed by Normative Instruction 51/2002 [2].

The physical-chemical methods employed to assess the quality of raw milk are regulated standards in all countries. In this work, it can be observed that some parameters meet the legal conditions; however, the results of three samples were outside the standard values allowed for milk quality. In Brazil, these standards are determined by the Normative Instruction 51/2002 [2].

The natural acidity of milk varies between 14 and 18°D (expressed as lactic acid) and comes from its own constitution, i.e., due to the presence of casein, phosphates, citrates, albumin and carbon dioxide [12]. Regarding this question, there were variations in acidity, being a sample with 14°D; six with 15°D; five with 16°D; two with 18°D, and one with 25°D (Table 1). The latter one, the sample 11 showed high milk acidity. This result, possibly due to the acidification of lactose, caused by microorganisms in multiplication in the milk [12].

The results of the density showed no change in the study samples, so all had densities considered as satisfactory. The milk density at 15°C varies from 1.028 to 1.035, with a mean of 1.032, and represents the sum of the intrinsic density of all its constituents [2]. The density values found (Table 1) are all within the standards established by the IN 51/2002. The density evaluates the weight of the milk, and if possible, to verify if the milk can be fraudulent by watering [15].

The value considered normal for cryoscopy varies from 0.530 to 0.548°H [9]. The cryoscopic index is nothing more than the way of detecting fraud in milk with added water [3]. The cryoscopic values described in Table 1 have a range of 0.510 to 0.555°H, indicating that two milk samples are out of the acceptable standard, samples 1, community A (0.510°H) and 11, community D (0.555°H). From these results, it is suggested that there is addition of water by producers of some communities, probably with the purpose of increasing the volume of milk produced, increasing sales in search of better financial returns. However, this breach interferes with the quality of the industry products, leading to milk disposal and making it unfeasible and jeopardizing new purchases of raw milk from that producer.

In fact, water addition scams can be detected by routine cryoscopic analysis, but depending on the ratio of water to solid reconstituents, the analyzed patterns may remain within the limits established by current legislation, so that there is no detectable watery fraud [17].

For bovine milk, the average percentage of fat is 3.5%, classifying it as integral. The milk categorized as semi-skimmed has fat content ranging from 0.6 to 2.9%. And the one considered as skimmed has fat content lower than 0.5% [10]. In the samples from this study, the values obtained in percentage ranged from 3.6 to 4.2%, with 2 samples being above the permitted level with 4.2% fat (sample 8, community C and sample 11, community D) (Table 1). Fat is, among the components of milk, the one with the greatest variability [14]. In order to avoid interference in the analysis, it is necessary that the milk is stirred in the tank prior to the collection for the laboratory. According to Pancotto [5], because of its density lower than other components, milk fat tends to occupy the upper fraction of the sample. Therefore, it is fundamental to homogenize it before sampling for the analysis, otherwise, because it is lesser density than other milk components, fat will be characterized as supernatant and, therefore, the sampling will show heterogeneity.

The results for fraudulent substances such as chlorides, starch, alkali and antibiotic residues showed negative in all samples, according to Table 2. Supporting these results, we highlight the work Fachinelli [8], who also noted the absence of fraudulent substances in all milk samples .

Table II - Results of analyzes of fraudulent substances (chlorides, starch, alkaline substances and antibiotics)

Samples	**1 to 15**
Fraudulent substances: chlorides, starch, alkaline substances, antibiotics	Negative
Legal standard: chlorides, starch, alkaline substances, antibiotics	Negative

The IN 51/2002 provides that fraudulent milk with some substance should be discarded [2]. Milk out of legal standards should be discarded in a biodigestor located near the dairy.

It is possible to observe a pattern of alterations in sample number 11, where there was acidity, cryoscopy and fat with high results and out of legal standards. Possibly for hygienic conditions from the producer community D. In contrast, the Community B (samples 4, 5, 6) and the Community E (samples 13, 14 and 15) did not show results out the rules from any of the analyzes on this study.

Fraudulent, adulterated or falsified milk is one in which water or other components have been added as preservatives or any elements foreign to their composition [18].

The results show that although some tests have indicated values that are far from the legal standards, the great majority of raw milk received by the dairy is in good quality and free from tampering by fraudulent substances and within acceptable levels for the standards analyzed in this work. In addition, samples out of permitted standards are discarded by the industry as they cannot be used for the manufacture of foods and byproducts. The IN/51 [2] teaches that non-standard milk established by it should be discarded as it is not in good use conditions.

In a study conducted by [16], the authors emphasized the need to perform a greater control of milk analysis for the manufacture of byproducts, since the results obtained by them were considered unsatisfactory from a hygienic-sanitary, technological and nutritional point of view, thereby compromising the consumer health and rights.

In this sense, the results of this study show how the monitoring of possible adulterations in raw milk is important, so that we can ensure the quality of the production of its derivatives.

IV. Conclusion

The physical-chemical analysis showed changes in samples of raw milk received by a dairy factory in the city of Saudade do Iguaçu, Paraná. Most of the results indicated good quality within the legal standards established by the Normative Instruction 51/2002.

For acidity there was a sample out the allowed standards, for the cryoscopy two results were obtained out the allowed standards, being one referring to high acidity, and another due to watering. In the fat analyzes there were two non-standard samples, and for density all the results were within the allowed standards. None of the samples showed fraudulent substances (chlorides, starch, alkaline substances and antibiotics).

The results show that most of the samples are within the standards for receiving dairy raw milk, however, the few samples that have exceeded the permitted limits are discarded by the industry.

Physical-chemical analyzes are of great importance, since they are the ones that verify the integrity of the milk received to go in the factory and produce food. In addition, the care in which these analyzes must be developed stands out, since they are what guarantee the production of good quality food, otherwise it could cause damages to the financial situation of the industry, or even to the consumer, due to food consumption that is not within the allowed legal standards.

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