DOI: 10.12957/demetra.2015.14843

Presence of antibiotics, conservatives and restoratives in pasteurized and UHT milk

Frederico Teixeira Corrêa[1]
Sérgio Augusto de Sousa Campos[1]
Sandra Maria Pinto[1]

[1] Universidade Federal de Lavras, Departamento de Ciência dos Alimentos, Curso de Pós-graduação em Ciência dos Alimentos. Lavras-MG, Brasil.

Correspondência / *Correspondence*
Frederico Teixeira Corrêa
E-mail: fredericotc01@yahoo.com.br

Abstract

Milk is highly consumed by the general population, within several age groups. To ensure consumer health, it is crucial that this food product is free of certain chemical substances. In order to assess the presence of antibiotics, conservatives and restoratives, as well as the physico-chemical quality of whole milk commercialized in the south of the state of Minas Gerais, Brazil, 11 UHT milk brands and four different pasteurized milk brands were bought in local supermarkets. The physico-chemical evaluation was conducted according to official methods. Brands were subjected to the following analysis: total solids content, fat content, crude protein content, ashes, carbohydrates, titratable acidity, density at 15°C (g/ml), freezing point, analysis of the presence of antibiotics, chlorides, formol, hydrogen peroxide, starch, sucrose and hypochlorite. It was concluded that the majority of the samples complied with the quality and safety standards required by the legislation, mainly the UHT milk brands, which were appropriate for consumption.

Key words: Milk. Physico-Chemical Analysis. Quality. Consumer. Identity and Quality Standards for Products and Services.

Introduction

Milk is a high nutritional food product, and it is part of the diet of millions of consumers all over Brazil. Milk quality is largely influenced by microbiological, physical, chemical and processing factors. Because milk is highly susceptible to changes and contamination, components are added on a regular basis in order to conceal actual milk quality. Given the need to set quality parameters, laws were passed on the quality standards that these products should comply with when sold to consumers.

In Brazil, quality parameters are defined by Normative Rule nº 62[1] for pasteurized milk and Ordinance nº 146[2] for UHT milk. Residues of preservatives and elements used for concealing the real characteristics of milk go far beyond the problem of fraud. One example is water that is added to milk; it is generally low-quality and may contain microorganisms and toxic elements.

Inadequate heat treatment, alteration of physical and chemical characteristics and the presence of preservatives and restoratives in milk are elements that can pose health risks. According to Santos & Carvalho,[3] it was found that, in 2013, companies in the Brazilian state of Rio Grande do Sul had been adding many banned substances to milk, including formaldehyde, which can be carcinogenic when ingested in the long run.

Minas Gerais, according to the Brazilian Institute of Geography and Statistics (IBGE), is the largest producer of fluid milk in Brazil, with an approximate volume of 8.39 billion liters in 2010. As for milk production (in billion liters), the states of Rio Grande do Sul (3.63), Paraná (3.60), Goiás (3.20), Santa Catarina (2.40) and São Paulo (1.60) are the 2nd, 3rd, 4th, 5th and 6th largest producers in Brazil, respectively.[4] In the state of Minas Gerais, the southern region has the highest production. Also according to IBGE,[4] Brazil produced about 30.72 billion liters of fluid milk in the same year.

Given the history of deviations in the quality of food products sold in Brazil, the volume of milk production in the south of Minas Gerais (compared with the national production), and the existence of laws for milk production, this study aims to assess the presence of preservatives and restoratives in milk, and the physico-chemical characteristics of 15 milk brands sold in the southern region of Minas Gerais.

Method

All 11 whole UHT milk brands (identified as A, B, C, D, E, F, G, H, I, J and K) chosen for evaluation are sold in the state of Minas Gerais or in Brazil at large. The four brands of pasteurized whole milk (identified as L, M, N and O) are manufactured in the industrial towns of Lavras, Perdões, Boa Esperança and Nepomuceno. All 15 brands have been awarded the Seal of Approval of the Federal Inspection Service (SIF).

Three units of each brand were bought. Each unit belonged to a different lot. The product units were bought between the months of March and December 2013, in supermarkets in the towns of Lavras and Nepomuceno (Minas Gerais, Brazil). The units were transported to the Dairy Sector, Department of Food Science, Federal University of Lavras. Expiry dates were approximately 120 days for UHT milk brands and three to four days for pasteurized milk brands. For the present research, the packages were bought intact and within their expiry date.

The analyses began immediately after the packages were opened. They were performed at the laboratory of physico-chemical analyses of the Dairy Sector, Department of Food Science, Federal University of Lavras. These analyses were performed according to Normative Rule no. 68[5] and Pereira et al.[6] using three lots for each brand. For each package, the analyses were performed in triplicate. Within the same lot, the means of quantitative values and standard deviations were calculated for interpretation of the results. Proximate analyses were made for total solids content (TSC), fat in a butyrometer by the Gerber method, crude protein by the Kjeldahl method, ash content in a muffle furnace at 500-550 °C and carbohydrates.

The analyses of enzymatic and physico-chemical patterns were titratable acidity in grams of lactic acid per 100 mL of milk (° D), density at 15 °C in a thermolactodensimeter, presence of chlorides, freezing point analysis as Hortvet degrees (° H), test for presence of formalin, hydrogen peroxide, starch, sucrose and hypochlorite using the methodology described by Pereira et al.,[6] and a test for presence of β-lactam antibiotics and tetracycline using the Cap-Lab® Twinsensor BT kit.

Results and discussion

Normative Rule no. 62[1] and Ordinance no. 146[2] have established the physical and chemical standards for pasteurized milk and whole UHT milk, respectively. Milk must have the characteristics set out in the legislation. When values do not conform with the predetermined range or substances are added to milk, it is considered to be inappropriate.

Restoratives are added to milk in order to disguise any tampering previously performed, such as addition of water or increase of the pH of acidified milk, or bring to normality some nutrient content that is below the value required by legislation, such as protein or non-fat solids content. Preservatives are intended to increase the shelf life of a product that may already be in a state of deterioration or with a high concentration of microorganisms.

In the analyses of the presence of hydrogen peroxide, formaldehyde, chlorides, hypochlorite, starch, sucrose and antibiotics for the three lots of 11 brands of UHT milk (A, B, C, D, E, F, G, H, I, J and K) and for the three lots of four brands of pasteurized milk (L, M, N and O), antibiotics were only detected in the first and second lot of brand N. For all other cases, including the third lot of brand N, no prohibited substance was detected.

Antibiotics, if consumed improperly, can lead to selection of resistant bacteria in the body and lack of control by gut microbiota, which are extremely harmful to health. Formaldehyde and hydrogen peroxide are substances added to milk to act as preservatives. Formaldehyde is carcinogenic and excess hydrogen peroxide can be toxic and cause mucosal allergy. Hypochlorite may function as a preservative. The function of chlorides is to restore milk density after water is added, thus increasing density, since water density is lower than that of milk. Starch and sucrose are used to make the volume of carbohydrates bigger than it is on the proximate composition; therefore, they are elements that disguise the actual levels of carbohydrates in milk. Consumption of milk and milk products has numerous health benefits, as a result of balanced nutrient concentration.

Proximate composition analysis aims to measure the approximate content of the main milk nutrients such as carbohydrates, dry matter content, protein, minerals, fat and water. Tables 1 and 2 show the results, in%, of proximate composition of the 15 milk brands for the three study lots.

Table 1. Means in percentages (g/100g) and respective standard deviations of total solids content (TSC), fat, non-fat solids content (NFSC), crude protein (CP), carbohydrates and ashes for 11 UHT milk brands (A, B, C, D, E, F, G, H, I, J and K) bought in the towns of Lavras and Nepomuceno (Minas Gerais), 2013.

Brands	TSC (%)	Fat (%)	NFSC (%)	CP (%)	Carb. (%)	Ash content (%)
A	11.78±0.13	3.10±0.10	8.68±0.20	3.44±0.06	4.50±0.22	0.74±0.01
B	11.67±0.21	3.03±0.06	8.57±0.21	3.58±0.03	4.27±0.18	0.72±0.02
C	11.71±0.14	3.27±0.06	8.44±0.15	3.59±0.08	4.13±0.10	0.72±0.03
D	13.35±0.15	4.23±0.15	9.15±0.08	3.92±0.08	4.41±0.12	0.82±0.03
E	11.83±0.09	3.27±0.31	8.57±0.38	3.47±0.10	4.31±0.36	0.75±0.01
F	11.69±0.12	2.97±0.15	8.73±0.20	3.37±0.07	4.46±0.26	0.72±0.03
G	12.10±0.12	3.47±0.21	8.63±0.30	3.74±0.05	4.22±0.20	0.74±0.03
H	12.03±0.06	3.47±0.12	8.56±0.06	3.57±0.21	4.11±0.09	0.72±0.02
I	11.45±0.05	3.03±0.06	8.42±0.03	3.32±0.09	4.31±0.18	0.70±0.02
J	11.41±0.10	3.10±0.10	8.31±0.20	3.46±0.19	4.13±0.05	0.72±0.02
K	11.52±0.10	3.07±0.06	8.45±0.15	3.30±0.10	4.43±0.26	0.72±0.03

Table 2. Means in percentages (g/100g) and respective standard deviations of total solids content (TSC), fat, non-fat solids content (NFSC), crude protein (CP), carbohydrates and ashes, for four pasteurized milk brands (L, M, N and O) bought in the towns of Lavras and Nepomuceno (Minas Gerais), 2013.

Brands	TSC (%)	Fat (%)	NFSC (%)	CP (%)	Carb. (%)	Ash content (%)
L	11.94±0.18	3.47±0.15	8.47±0.03	3.53±0.18	4.19±0.19	0.75±0.01
M	12.06±0.17	3.30±0.20	8.76±0.03	3.43±0.18	4.55±0.19	0.76±0.03
N	11.34±0.22	3.20±0.10	8.14±0.32	3.31±0.16	4.06±0.19	0.71±0.04
O	11.83±0.18	3.37±0.23	8.46±0.06	3.50±0.13	4.21±0.20	0.74±0.01

Brazilian law requires, for whole UHT milk, at least 3% (g/100g) fat in its composition. Out of the 11 brands analyzed, only brand F had a mean of (2.97 ± 0.15) with standard deviation range below 3% (g/100g) fat. All brands of pasteurized milk comply with the rules.

The minimum non-fat solids content required for UHT milk is 8.2% (g /100g), and for pasteurized milk, 8.4% (g/100g). The UHT milk brands E and J (levels of 8.57 ± 0.38 and 8.31 ± 0.20, respectively) and the pasteurized milk brand N (8.14 ± 0.32), showed lower values than those recommended by the Brazilian legislation in at least one of the lots.

Giombelli et al.,[7] while researching pasteurized milk and Type B milk produced in the state of Paraná, observed that pasteurized milk showed worse microbiological and physical-chemical quality than type B milk. It was also observed that the quality of Brazilian milk has improved over time. In 2006, total solids content fell short of standard in 38.75% of a total of 192 pasteurized milk samples. In 2007, this number dropped to 8.11%. In 2008, it fell further, reaching 1.33%.[7]

Bersot,[8] while analyzing UHT milk from the state of Paraná, with regard to the physicochemical characteristics of the samples, found that 7.3% of the results of acidity, 29% for fat and 50.7% for non-fat solids content, were contrary to the Technical Regulation for Identity and Quality of UHT Milk.[2]

Titratable acidity is a quantitative method to measure the acidity of milk, while the alcohol and alizarol solution methods are qualitative. The pH value of milk is slightly below seven, as there is small amount of ions and natural milk proteins that make its pH slightly acidic. Freezing point analysis, given in degrees Hortvet, measures the freezing point of milk, which occurs at a temperature below 0 °C; it is therefore lower than the temperature of water. This analysis is performed to identify fraudulent addition of water to milk.

Milk density is given in g/ml, and it should be between 1.028 and 1.034 for whole milk. Lower values indicate addition of water, since water density is lower than that of milk. Higher density is indicative of addition of restoratives, such as salts and organic compounds, and also of milk fat removal, since fat density is lower than water density. Tables 3 and 4 show the results of mean density at 15 C (g/ml), Dornic acidity (lactic acid g/100 ml) and freezing point in degrees Hortvet for the 15 study milk brands.

Table 3. Means and respective standard deviations of density at 15 °C (g/ml), acidity expressed in Dornic degrees (lactic acid g/100 ml) and freezing point in degrees Hortvet for 11 UHT milk brands (A, B, C, D, E, F, G, H, I, J and K) bought in the towns of Lavras and Nepomuceno (Minas Gerais), 2013.

Brands	Density	Tit. Acidity	F.P. Analysis (°H)
A	1.031±0.00	14.33±0.58	0.54±0.00
B	1.032±0.00	15.00±0.00	0.54±0.01
C	1.033±0.00	16.00±1.00	0.55±0.01
D	1.031±0.00	17.33±0.58	0.54±0.00
E	1.033±0.00	16.67±0.58	0.54±0.00
F	1.032±0.00	15.67±0.57	0.54±0.00
G	1.031±0.00	16.33±1.15	0.54±0.00
H	1.032±0.00	15.33±0.58	0.54±0.01
I	1.031±0.00	15.33±1.53	0.47±0.11
J	1.034±0.00	15.67±0.57	0.55±0.00
K	1.032±0.00	15.67±1.15	0.54±0.00

Table 4. Means and respective standard deviations of density at 15 ° C (g / ml), titratable acidity expressed in Dornic degrees (lactic acid g/100 ml) and freezing point in Hortvet degrees for four brands of pasteurized milk (L, M, N and O) bought in the cities of Lavras and Nepomuceno (Minas Gerais), 2013.

Brands	Density	Tit. Acidity	F.P. Analysis (°H)
L	1.032±0.00	16.67±0.58	0.54±0.00
M	1.032±0.00	16.33±1.15	0.54±0.00
N	1.030±0.00	14.33±1.15	0.53±0.01
O	1.033±0.00	14.67±0.58	0.54±0.00

All the study milk brands showed density values between 1.028 and 1.034 g/ml. Freezing point values for all UHT and pasteurized milk brands ranged between 0.530 and 0550 °H, as required by law. A more negative mean freezing point value for UHT milk, compared with pasteurized milk, can be explained by the addition of sodium citrate, which is allowed in UHT milk to improve its storage stability. Freezing point analysis is the process of measuring the beginning of the milk freezing point. It is the most effective method in determining fraud by addition of water.

Both for UHT milk and for pasteurized milk, permissible acidity is 0.14 to 0.18 grams of lactic acid/100 ml of milk or 14 to 18 ° D (Dornic degrees). In the analysis carried out, it was found that all of UHT and pasteurized milk brands showed acidity between 14 and 18 ° D, except for brand N (14.33 ± 1.15), whose standard deviation range overstepped the threshold of 14 ° D.

Titratable or acquired acidity reflects lactic acid production; thus, it is possibly indicative of hygiene and preservation of milk. Very high acid values may indicate possible contamination of milk, since the microorganisms metabolize lactose to form lactic acid. By contrast, low acid values may indicate the presence of an alkali. Domareski et al.[9] found, in a physical-chemical analysis of 12 samples of UHT milk sold in the Mercosur block, that four samples had a relative density value below 1.028 g/ml, four samples had density of 1.028 g/ml and the other four, density of 1.029 g/ml.

Rheinheimer et al.[10] analyzed 41 milk samples:11 pasteurized milk samples and 30 of UHT milk samples. They found that 77.8% of the pasteurized milk samples and 18.7% of the UHT milk samples showed acidity below the recommended value. The improvement of milk quality in Brazil in recent years is a result of the implementation of good manufacturing practices. Vallin et al.,[11] analyzed 46 samples of raw milk produced in 19 towns in the central region of Paraná; 32 farms use manual milking and 14, mechanical milking. They observed that after the implementation of hygiene practices, there was a mean reduction of 87.90% in total bacteria count in farms with manual milking and 86.99% on farms with mechanical milking. The same authors also observed a mean reduction of somatic cell count in milk by 33.94% on farms with manual milking and 51.85% on farms with mechanical milking.

Conclusion

The UHT and pasteurized milk brands under analysis complied with the existing rules and identity and quality standards for most physico-chemical and enzymatic parameters evaluated. There are still some irregularities, however, especially as far as pasteurized milk brands are concerned. It was found, in this study, that milk quality has increased in Brazil, but further research is required to verify whether or not these improvements are being maintained.

Acknowledgments

The authors are thankful to the Coordenation for the Improvement of Higher Education Personnel (CAPES) for the Master's and doctoral scholarships granted.

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Received: January 30, 2015
Revised: June 02, 2015
Accepted: May 13, 2015