Characterization of Effluents Generated During the Cleaning of Expansion Tanks Used to Store Raw Milk in Brazil

Caracterização dos Efluentes Gerados Durante a Limpeza dos Tanques de Expansão Usados para Armazenar Leite Cru no Brasil

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ABSTRACT
The objective of this study was to characterize the effluents generated during the cleaning of the expansion tanks used to store raw milk in Brazil. Fifteen expansion tanks were chosen and three samples collected from each tank, as well as measuring the temperature and volume. The biological oxygen demand (BOD), chemical oxygen demand (COD), acidity, pH, alkalinity, total phosphorus, nitrogen, fats and oils, hardness, turbidity and total dissolved solids were determined. The statistical design adopted was completely at random and the Scott-Knott test was adopted to compare the means that differed significantly according to the F test. The multivariate data analysis was also carried out and studied by the principal component analysis (PCA) and the grouping analysis for similarity, as represented by a dendogram. The effluents were not treated and some of them (26.7%) were discharged into water courses. The concentrations of the organic matter indicators and the results of the physicochemical analyses of the expansion tank washing effluents showed that the majority of the variables were above the legal standards established by Brazilian legislation. Thus an adequate destiny for these effluents is required. The PCA showed that the variables with the greatest influence on the characteristics of the expansion tank effluents were related to the COD and nitrogen concentrations. A total of 88% of similarity was found between the effluents of the 15 tanks. According to the concentrations obtained for the phosphorus, nitrogen, COD and BOD of the effluents, they could be treated by the wetlands systems.

Keywords: Bulk milk, expansion tank, sanitization, effluent.

RESUMO
O objetivo deste estudo foi caracterizar os efluentes gerados durante a higienização dos tanques de expansão utilizados para armazenar leite cru no Brasil. Foram escolhidos 15 tanques de expansão e coletadas três amostras de cada tanque, além de ser determinada a temperatura e o volume dos efluentes gerados durante a higienização dos mesmos. Foram determinados também a demanda biológica de oxigênio (DBO), demanda química de oxigênio (DQO), acidez, pH, alcalinidade, fósforo total, nitrogênio, gorduras e óleos, dureza, turbidez e sólidos dissolvidos totais. O delineamento estatístico adotado foi o inteiramente casualizado e o teste de Scott-Knott foi adotado para comparar as médias que diferiram significativamente de acordo com o teste F. A análise multivariada dos dados também foi realizada e estudada por meio da análise de componentes principais (PCA) e da análise de agrupamento por similaridade, representada por um dendograma. Os efluentes não eram tratados e parte deles (26,7%) era descartada em cursos d'água. As concentrações dos indicadores de matéria orgânica e os resultados das análises físico-químicas dos efluentes mostraram que a maioria das

variáveis estava acima dos padrões legais estabelecidos pela legislação brasileira. Assim, é necessário um destino adequado para esses efluentes. A PCA mostrou que as variáveis com maior influência nas características dos efluentes dos tanques de expansão foram relacionadas à DQO e às concentrações de nitrogênio. Foi constatada similaridade de 88% entre os efluentes dos 15 tanques. De acordo com as concentrações obtidas para fósforo, nitrogênio, DQO e DBO dos efluentes, eles poderiam ser tratados pelos sistemas de *wetlands*.

Palavras-chave: Leite a granel, tanque de expansão, sanitização, efluentes.

1 INTRODUCTION

The population increase and consequent increase in agricultural and industrial activities have reduced the availability and quality of water resources. The need for practices and policies aimed at the sustainable use of these resources has thus become imperative (CARVALHO et al., 2011). Compliance with environmental norms has become necessary throughout the milk production chain, since dairy farms produce effluents with high organic matter, nitrogen and phosphorus concentrations, which, when badly managed, can cause serious environmental problems (RICO; GARCÍA; RICO, 2011), resulting in the eutrophication of surface water and the death of aquatic organisms.

Agricultural activity is known as a great water consumer and residue generator. However, in Brazil, the agricultural environment is not contemplated for the supply of potable water or for sewage and waste collection and treatment. Instead rural establishments use water from mines, generally unprotected, removed and delivered to the installations for consumption by way of booster pumps. It can also come from drilled or artesian wells.

Milk production has an elevated water pollution potential since large amounts of water are required for dairy farming, when one considers various factors such as the animal feedstuffs, sanitization of their installations and of the milking equipment, including the raw milk storage tanks and utensils (WILLERS et al., 2014). In addition, lactating cows require more water per body weight than other animal categories. Thus dairy farming in Brazil produces large volumes of effluent with high organic loads (RICO; GARCÍA; RICO, 2011).

As the dairy sector modernized and the consumer demand for quality foods grew, so Brazil adopted the bulk collection of milk, and hence refrigeration of the milk in an expansion tank on the farm was implemented. In the bulk system, the raw milk is stored in expansion tanks on the farms for up to 48 hours at 4 ºC, and then transported to the factory in a truck with an isothermal tank (BRASIL, 2018). The sanitization of the expansion tanks thus became the responsibility of the farmer, causing an increase in the disposal of liquid effluent into the environment.

According to the USDA (2014), Brazil is one of the greatest milk producers in the world. The expansion of the dairy culture demands the development of technology that makes it possible to mitigate the impact of this activity on the environment (PELISSARI et al., 2013).

All these facts generate great concern about the exploration of water resources (constantly more depleted) and the great generation of residues, principally effluents that do not receive adequate treatment or destiny. From this one can see the enormous importance of treating the effluents generated on rural dairy farms, but in order to manage these effluents, one must first characterize and quantify them so as to dimension the ideal treatment system. Thus one must first determine the physical, chemical and microbiological characteristics, so as to choose appropriate technologies to treat the effluent such that it can be discharged into the environment, aiming at efficiency and a minimum of negative impact (BATISTA et al., 2014).

However, defining and quantifying the composition of an effluent is a difficult task, since there are a variety of factors that can influence the volume and composition of liquid residues. Thus the objective of the present study was to estimate the difference in the volumes of effluents produced during the cleaning of expansion tanks in Brazil according to their milk storage capacity, and also evaluate the characteristics of these effluents for the future implantation of treatment systems on dairy farms.

2 MATERIAL AND METHODS

2.1 EXPERIMENTAL DESIGN

The experiments were carried out during a 12 month period (June 2015 to May 2016), and the samples collected from farms with expansion tanks in the municipality of Rio Pomba, State of Minas Gerais, Brazil. The 15 expansion tanks selected were initially divided into three categories according to their capacity: i) up to 1000 liters (category 1); ii) from 1000 to 2000 liters (category 2); and above 2000 liters (category 3). Each unit was treated individually, since each presented its own location, maintenance system and conditions. The analysis was carried out by way of a comparison between the means of the variables of interest for each tank individually.

Only the liquid residue was considered, that is, only the water used to wash the expansion tanks. In addition to determining the volume of the effluent produced, the expansion tanks were monitored during the effluent characterization step for 10 months, in order to evaluate the quality of the washing effluent produced. Three collections were made from each tank, one per trimester.

2.2 QUANTITATIVE CHARACTERIZATION OF THE EXPANSION TANK WASHING EFFLUENTS

After the truck had collected the milk from each farm, the expansion tanks were sanitized, and a collection hose fixed to each tank to collect the effluent. The entire washing effluent was collected in a water tank from the other end of the hose.

The volume of effluent generated by each tank was determined for the future dimensioning of the effluent treatment system. The volume of water consumed in the sanitation process was determined by a method known as cubing, in which the time the water takes to fill a recipient of known volume is measured. Since the rate is the volume as a function of time, the volume of the recipient filled was divided by the time it took to do this.

To collect the effluent from the expansion tank (water used to wash it), a hosepipe was used that had the same connection as the hosepipe used by the milk collection trucks. The waters used to wash the floor in the room where the expansion tank was installed and to wash the utensils, were not included in this study.

2.3 QUALITATIVE CHARACTERIZATION OF THE EFFLUENTS, DETERMINATION OF ORGANIC MATTER INDICATORS AND THE PHYSICOCHEMICAL ANALYSIS

In each visit to the farms, notes were taken concerning the tank sanitation steps, the ways the effluent was disposed of and the origin of the washing water, as well as questioning the person responsible for the tank if he/she was contracted or the owner of the tank and if they intended to implant an effluent treatment system.

For each collection, after sanitization, the temperature of the effluent was noted, and 2.7 L of sample removed from the water tank and stored at 4.0 ºC. The analyses for turbidity, pH, alkalinity, and dissolved oxygen (to subsequently obtain the BOD values) were carried out on the same day as the samples were collected. For the remaining analyses, the rest of the sample was acidified and preserved according to the National Guide for the Collection and Conservation of samples (BRANDÃO et al., 2011). The analyses for acidity and hardness were carried out in up to 24 hours after collection. The analyses for dissolved solids – TDS (refrigerated sample), COD and nitrogen (refrigerated and acidified samples) were carried out in up to seven days after collection. The analyses for total phosphorus and for fatty acids (refrigerated and acidified samples) were carried out within 28 days after collection.

Quantification of nitrogen by the Kjeldahl method, measured in $mg.L^{-1}$, represents the sum of the organic nitrogen plus nitrogen in the form of ammonia (APHA, 2012).

The physicochemical analyses and the organic matter indicators of the variables were determined in triplicate (Table 1). The total phosphorus and COD were measured by spectrophotometry (KASUAKI model IL 227), and the determination coefficient (R^2) of the linear regression and the reading range of these variables are described in Table 2. The determination coefficient represents the proportion of the data variation explained by the mathematical model. Values close to one indicate that the proposed model is adequate to describe the phenomenon.

Table 1. Variables analyzed and methodology employed

Variables	Methodology employed
pH	Direct, pH meter NT PHM - TECNOPON
Acidity (mg $CaCO_3.L^{-1}$)	Titulometric method, 2310B as proposed by APHA (2012)
Alkalinity (mg $CaCO_3.L^{-1}$)	Titulometric method, 2320B as proposed by APHA (2012)
Hardness (mg $CaCO_3.L^{-1}$)	Titulometric method, 2340C as proposed by APHA (2012)
Nitrogen ($mg.L^{-1}$)	Semi-Micro-*Kjeldahl* method, 4500-N_{org}C as proposed by APHA (2012)
Total phosphorous ($mg.L^{-1}$)	Colorimetric method, 4500-P E as proposed by APHA (2012)
BOD_5 ($mg.L^{-1}$)	BOD_5 method, 5210B as proposed by APHA (2012)
COD ($mg.L^{-1}$)	Closed reflux method by colorimetry, 5220 D, by APHA (2012)
Turbidity (NTU)	Apparatus TB-100 MS, TECNOPON
TDS ($g.L^{-1}$)	Gravimetric method, 2540C as proposed by APHA (2012)
Fats & oils ($mg.L^{-1}$)	*Soxhlet* extraction, 5520 D as proposed by APHA (2012)
Temperature (ºC)	Infrared thermometer, SKILL-TEC

Table 2. Reading range and R^2 of the variables COD and total phosphorous

Analyses	Reading range ($mg.L^{-1}$)	R^2
COD	0-1200	0.9934
Total phosphorous	0-18	0.9029

2.4 STATISTICAL ANALYSIS

A totally random statistical design was adopted. The Scott-Knott test was used at a level of significance of 5% to compare the means that differed significantly according to the F test. The ExpDes package (FERREIRA; CAVALCANTI; NOGUEIRA, 2011) of the R program was used for all the calculations. The multivariate analyses were carried out using version 2016.04 of the XLSTAT program.

The variables of temperature, pH, acidity, alkalinity, BOD_5, COD, total phosphorus, nitrogen, fats and oils, hardness, turbidity and TDS were studied using the principal components analysis (PCA), a process which examines the whole group of interdependent relationships between the variables, taking the total variance of the data into account. The use of the principal component analysis is indicated when it is important to determine the minimum number of factors that account for the maximum variance in the data. These factors are called the principal components.

The grouping analysis was developed by way of the UPGMA (unweighted pair group method using arithmetic averages) hierarchal grouping method, considering the dissimilarity index as from the Euclidean distance. In the grouping analysis, the groups are formed according to the similarity of the variables, that is, the groups identify the similarities between the fifteen expansion tanks by way of the variables analyzed. It is represented by a graphical figure known as a dendrogram, in which the fifteen tanks are agglomerated according to a criterion of similarity with respect to the variables measured.

2.5 DATA FOR THE DIMENSIONING AND IMPLANTATION OF THE WETLANDS SYSTEM

The variables of temperature, volume, pH, acidity, alkalinity, BOD_5, COD, total phosphorus, nitrogen, fats and oils, hardness, turbidity and TDS were discussed with respect to the feasibility of implanting a wetlands system in the treatment of the effluents characterized above.

3 RESULTS AND DISCUSSION

The 15 expansion tanks selected were not collective, were installed in covered locations of adequate size, were well ventilated, and were sanitized after emptying (normally every other day). The locations were easily accessible to the milk collection lorry. The tanks were cleaned manually, 40% being cleaned by the owner and 60% by professionals contracted for this purpose.

None of those responsible for cleaning the tanks had undergone any training in sanitization or in the rational use of water. On determining the water consumption of a medium sized dairy farm, Willers et al. (2014) reported that the professionals involved in the cleaning procedure also had little or no instruction concerning the adequate use of water.

In general, sanitization of the tanks started with rinsing with running water using a hosepipe, followed by removal of the organic matter using appropriate detergents, brushes and brooms, and finally by further rinsing. Only 46.6% of the tanks were submitted to chemical sanitization using chlorine. The water used to wash the tanks came from wells on 46.7% of the farms and from springs on 53.3% of the farms.

3.1 CHARACTERIZATION OF THE EFFLUENTS FROM THE EXPANSION TANKS

Table 3 shows the capacity of each expansion tank, the locations where the effluents were discharged and the mean values for the temperature, volume and flow rate of the effluents. The tank washing waters (effluents) evaluated were not used for other purposes, and on 73.3% of the farms, were discharged directly onto the soil with no treatment. On 26.7% of the farms, the untreated effluents were channeled into streams. These effluents were neither destined nor treated adequately, mainly due to the investment costs of the treatment systems.

According to the Scott-Knott test there was no difference between the exit temperatures of the effluents ($p>0.05$), and in the cases of tanks T5, T7, T12 and T13, which discharged their effluents into steams, their temperatures were in agreement with the temperature permitted in Brazil for such discharges, which is below 40 ºC [11,12].

The water temperature is a variable that should be controlled and monitored such that the conditions of the location where they are discharged are not changed. Temperature changes can imply in retardation or acceleration of physical, chemical and biological reactions, as also oxygen absorption and the precipitation of compounds, influencing the dissolved gas levels. When slightly raised, gas solubility is reduced, generating odors and ecological imbalance (von SPERLING, 2005). The temperature has a direct influence on the levels of dissolved oxygen, and depending on the size of the reduction in the amount of dissolved oxygen, can result in the death of diverse aquatic beings, including fish.

The water volumes used to sanitize tanks T5 and T8 were larger than those used for the other tanks ($p<0.05$), although these two tanks were not classified as the largest capacity tanks in the present experiment. Tank 5 was classified as a small capacity tank (up to 1000 L) and tank 8 as a medium capacity tank (more than 1000 L and less than 2000 L). Hence it was shown that larger volumes of water were not used for the largest tanks (category above 2000 L), the amount of water used depending more on the operator. It must be mentioned that the flow rate did not influence the results of this study and was not analyzed by the Scott-Knott test, since its value was obtained directly from the volume measurement.

With respect to the water volume used to sanitize the tanks, the rational use of the water is the important factor, adopting techniques and procedures that result in its conservation by way of changes in behavior and consciousness, without compromising the sanitation. In addition methods should be adopted that avoid water pollution, or at least reduce the concentration of pollutants. The importance of water management in the milking sector should also be emphasized, and the need for orientation to implement more sustainable and cleaner production initiatives in Brazil that can minimize water consumption and the production of effluents. Water wastage and a lack of standardization of the raw milk storage tank sanitization procedures are critical, and are a direct reflection of the lack of training and consciousness of the collaborators with respect to good environmental practices.

Table 3 shows the mean values determined in the washing effluents from the 15 expansion tanks for pH, acidity, alkalinity and hardness. It can be seen that the pH values of the effluents of tanks T3, T7, T13 and T15 were significantly higher than those of the effluents of the other tanks ($p<0.05$). According to Resolution CONAMA 430/2011, in Brazil effluents with pH values between 5.0 and 9.0 can be discharged into water courses (BRASIL, 2011). Thus, the effluents of tanks T7 and T13, which were discharged into streams, showed pH values well above that permitted by the legislation in force.

According to Lagger et al. (2000), the pH and hardness values are relevant in the cleaning procedure, and are related to the type of detergent used. Thus the significant difference in pH values between the effluents from the different expansion tanks could be related to the amount and type of detergent used to sanitize each tank. For the variables of acidity, alkalinity and hardness, no limits have been established by the Brazilian legislation for effluents of this nature.

Although there was no difference in the mean acidity values between the effluents from the different tanks, those of tanks T6, T10, T14 and T15, showed high standard deviations (SD), demonstrating great variation in acidity in relation to that of the environment. The effluent from tank T13 was the most alkaline ($p<0.05$) and also presented the highest pH value ($p<0.05$). The alkalinity determined by Pelissari et al. (2013) in their evaluation of the performance of the wetlands system when applied to the treatment of dairy farm effluents was 668 mg of $CaCO_3$/L. This value was above the typical value for the alkalinity of domestic sewage, which is 200 mg/L (von SPERLING, 2005).

Table 3. Capacity of each expansion tank, effluent discharge location and the mean values (n=3) of the effluent temperatures, volumes and discharge rates and also their physicochemical characteristics

Tank	Capacity (L)	Effluent discharge location	Temperature (ºC) ± SD	Volume (L) ± SD	Discharge rate (L/week)	pH ± SD	Acidity (mg $CaCO_3$/L) ± SD	Alkalinity (mg $CaCO_3$/L) ± SD	Hardness (mg $CaCO_3$/L) ± SD	Nitrogen (mg/L) ± SD	Total phosphorous (mg/L) ± SD	Turbidity (NTU) ± SD	TDS (g/L) ± SD	Fats & oils(mg/L) ± SD	COD (mg/L) ± SD	BOD_5 (mg/L) ± SD	Ratio COD/ BOD
T1	1000	soil	23.97 ±2.81[a]	26.33 ±3.06[c]	92.16	6.56 ±0.63[c]	140.01 ±33.22[a]	164.31 ±50.13[c]	126.09 ±90.66[a]	177.70 ±25.47[b]	20.09 ±18.93[a]	887.00± 48.50[a]	1.30 ±0.53[b]	523.27 ±215.44[b]	6875.26± 1418.53[b]	1935.57± 871.36[a]	3.55
T2	680	soil	27.27 ±3.11[a]	35.00 ±2.00[b]	122.50	6.00 ±1.02[c]	146.57 ±48.41[a]	97.15 ±74.36[c]	90.98 ±58.92[a]	131.30 ±62.28[b]	15.27 ±19.66[a]	844.67± 182.28[a]	0.92 ±0.26[b]	323.77 ±184.53[b]	6139.13± 2758.24[b]	2979.78± 1789.15[a]	2.06
T3	1000	soil	25.10 ±6.03[a]	20.17 ±7.22[c]	70.60	10.90 ±0.88[b]	0.00 ±0.00[a]	491.64 ±108.30[c]	69.68 ±27.82[a]	71.83 ±33.32[b]	14.72 ±5.13[a]	641.33± 196.04[a]	1.61 ±0.55[b]	234.46 ±165.73[b]	4860.33± 1469.20[b]	1790.18± 1556.45[a]	2.71
T4	680	soil	21.40 ±3.86[a]	18.33 ±2.08[c]	64.16	6.36 ±0.13[c]	90.97 ±8.99[a]	124.30 ±67.08[c]	93.44 ±33.95[a]	180.33 ±47.52[b]	6.41 ±3.19[a]	750.00± 148.54[a]	0.64 ±0.28[b]	209.94 ±60.56[b]	5896.76± 2451.04[b]	1498.32± 1146.74[a]	3.94
T5	1000	stream	23.20 ±4.85[a]	67.83 ±4.86[a]	237.41	7.66 ±0.23[c]	34.51 ±18.43[a]	178.00 ±69.75[c]	168.28 ±43.89[a]	60.07 ±44.56[b]	6.26 ±4.99[a]	935.33± 70.61[a]	1.26 ±0.48[b]	356.96 ±103.29[b]	5703.58± 868.83[b]	1471.20± 1283.80[a]	3.88
T6	1550	soil	29.20 ±5.03[a]	29.67 ±16.62[b]	103.85	6.78 ±0.76[c]	233.54 ±149.18[a]	149.58 ±129.00[c]	272.47 ±248.74[a]	262.60 ±179.99[a]	42.29 ±42.73[a]	1433.00± 765.00[a]	1.79 ±1.27[b]	1255.53 ±813.96[a]	9670.98± 2806.05[a]	3200.85± 1863.62[a]	3.02
T7	1500	stream	29.60 ±1.31[a]	39.00 ±12.17[b]	136.50	9.53 ±0.70[b]	8.55 ±14.81[a]	187.68 ±54.30[c]	170.33 ±179.85[a]	61.43 ±45.74[b]	17.79 ±6.71[a]	534.67± 306.93[a]	0.89 ±0.59[b]	297.35 ±298.20[b]	4154.23± 866.92[b]	1710.96± 1659.12[a]	2.43
T8	1500	soil	24.80 ±1.57[a]	57.67 ±14.01[a]	201.85	6.71 ±0.51[c]	92.88 ±18.40[a]	86.16 ±25.86[c]	125.86 ±10.99[a]	168.57 ±74.71[b]	7.11 ±5.43[a]	956.67± 37.54[a]	1.27 ±0.43[b]	462.88 ±55.31[b]	7945.90± 1352.95[a]	2151.11± 1131.99[a]	3.69
T9	1500	soil	23.67 ±2.52[a]	32.33 ±5.51[b]	113.16	7.04 ±0.32[c]	46.19 ±19.38[a]	32.62 ±6.46[c]	45.47 ±27.95[a]	41.83 ±14.82[b]	0.40 ±0.69[a]	31.37± 13.24[a]	0.42 ±0.32[b]	107.42 ±105.63[b]	4350.88± 1537.92[b]	2712.21± 2253.18[a]	1.60
T10	1070	soil	27.70 ±3.62[a]	17.17 ±1.44[c]	60.10	7.20 ±0.34[c]	146.29 ±109.52[a]	183.65 ±89.94[c]	160.34 ±118.38[a]	278.27 ±169.89[a]	40.57 ±56.86[a]	1426.60± 1578.24[a]	2.48 ±2.08[b]	897.24 ±692.65[a]	10469.26± 5910.97[a]	2938.71± 2812.00[a]	3.56
T11	2000	soil	21.67 ±3.79[a]	24.67 ±4.80[c]	86.35	6.59 ±0.39[c]	90.09 ±46.95[a]	132.17 ±19.11[c]	147.11 ±63.33[a]	99.27 ±11.32[b]	7.46 ±6.46[a]	927.67± 66.91[a]	0.97 ±0.46[b]	412.62 ±72.75[b]	5871.58± 1109.24[b]	2913.28± 1335.06[a]	2.02
T12	2000	stream	21.03 ±0.75[a]	35.67 ±7.57[b]	124.85	6.92 ±0.61[c]	84.40 ±89.91[a]	139.74 ±81.09[c]	107.94 ±42.01[a]	56.20 ±39.63[b]	6.93 ±4.36[a]	497.00± 376.48[a]	0.64 ±0.57[b]	236.33 ±162.15[b]	5975.80± 4623.87[b]	1848.34± 1472.71[a]	3.23
T13	3000	stream	24.50 ±2.50[a]	34.50 ±0.50[b]	120.75	12.54 ±0.05[a]	0.0 0 ±0.00[a]	2141.00 ±89.00[a]	54.00 ±5.00[a]	41.10 ±5.85[b]	7.29 ±0.27[a]	324.00± 2.00[a]	2.71 ±0.04[b]	117.34 ±10.82[b]	4224.33± 32.80[b]	1683.23± 1466.65[a]	2.51
T14	2500	soil	22.37 ±4.90[a]	41.00 ±3.46[b]	143.50	6.77 ±0.52[c]	225.46 ± 141.55[a]	352.86 ±88.87[c]	153.17 ±66.58[a]	269.17 ±83.72[a]	67.81 ±61.35[a]	1032.33± 71.81[a]	4.09 ±3.38[a]	674.64 ±577.18[a]	8811.93± 4929.57[a]	2304.67± 2437.33[a]	3.82
T15	3000	soil	23.23 ±3.51[a]	34.33 ±8.33[b]	120.16	10.67 ±2.94[b]	97.52 ± 168.92[a]	1682.21 ±983.19[b]	246.33 ±8.14[a]	422.00 ±339.50[a]	65.37 ±78.90[a]	4478.00± 4470.43[a]	5.96 ±1.86[a]	974.43 ±435.08[a]	13624.54± 5068.53[a]	7021.38± 558.23[a]	1.94

Means followed by the same letter in the same column do not differ according to the Scott-Knott test ($p>0.05$).

The values for hardness of the effluents from the different tanks did not vary ($p>0.05$). Studies have shown that the criterion of hardness is not important in sanitation, but is important in pipe maintenance, due to the accumulation of calcium and magnesium in water transport systems.

The nitrogen concentrations (Table 3) were found to be higher in the effluents from tanks T6, T10, T14 and T15 as compared to the values found in the effluents from the other tanks ($p<0.05$). These tanks probably showed larger amounts of milk residue on initiating the washing procedure, contributing to higher values for nitrogen and phosphorus (Table 3).

The values for nitrogen found in the effluents from tanks T5, T7, T12 and T13, which were discharged into streams, were above the limits established by the Brazilian legislation, which recommends a limit of 20.0 mg/L [11,12]. These high concentrations determine the high eutrophic power of these effluents. Results above those recommended by the legislation were expected in this experiment, since effluents from dairy activities are rich in phosphorous and nitrogen.

The total phosphorous contents of the effluents from the different tanks did not differ significantly between the samples ($p>0.05$), although they were fairly high. However there is no limit in Brazilian legislation for the phosphorous content of effluents discharged into water courses. According to von Sperling (2005), one of the common anthropogenic origins of phosphorous is that of detergents, which could explain the high concentration of this element in the samples obtained in the present study.

On examining the feasibility of the wetlands system in the treatment of effluents in cold climates, Mumñoz et al. (2006) reported that this type of treatment was feasible for a total phosphorous content of 44 mg/L in dairy establishment effluents. This showed the feasibility of using the wetlands system to treat the effluents studied in the present work, which showed similar values for phosphorous. Also, these systems adapt well to tropical climates.

Bortoluzzi et al. (2020) found values of total nitrogen in effluent from the whey industry close to this work. Wood et al. (2007) measured the total phosphorous contents in effluents produced by cattle installations and found very high values of 89.3 mg/L and values of 540 mg/L for nitrogen, probably due to the high milk concentrations in the effluents. In the environmental legislation of some European countries, the discharge of waste is based on the amounts of some reference nutrients, normally nitrogen or phosphorous, indicating the importance of characterizing these nutrients in effluents. Currently Brazil has no legislation concerning the application of dairy cattle waste to the soil. In European legislation, the permitted amounts of nitrogen and phosphorous are balanced against the amounts available in the soil, and the type of culture to be planted there. Hence, according to agronomical recommendations, only that amount capable of being absorbed by the culture can be applied (BATISTA et al., 2014).

It was shown that the values for turbidity of the effluent samples from the expansion tanks (Table 3) did not differ significantly ($p>0.05$). Turbidity is related to the transparency of the water and hence an elevated value for turbidity indicates high suspended organic and inorganic matter contents. The Brazilian legislation has established no limits for the turbidity of effluents in order to discharge them.

The values for TDS were significantly higher for the effluents of tanks T14 and T15 ($p<0.05$) than those obtained for the effluents of the other tanks (Table 3). Once again no limits have been established for this variable for the discharge of effluents.

The values for fats and oils of the effluents were higher for those from tanks T6, T10, T14 and T15 ($p<0.05$) than for the effluents from the other tanks (Table 3). Higher fats and oils concentrations in the effluents from the tanks are related to the fact that these effluents contain larger milk residues, since the same effluents had higher nitrogen and phosphorous contents (Table 3) and also higher turbidity values (table 3). Knowledge of the amount of fats and oils in the effluent is useful in the elaboration of adequate projects and the operation of treatment systems for residual waters, and can also call attention to determined difficulties in treatment (APHA, 2012).

In Brazil, the maximum amount of fats and oils permitted by law is 50 mg/L [11,12], and hence the effluents from all the expansion tanks were above the amount permitted for discharge. However, only tanks T5, T7, T12 and T13 discharged their effluents into streams.

It should be noted that the effluent from tank T6 had a fat content equivalent to 0.12% (1255.53 mg/L). On this farm, as on others, it was noted that the stirring mechanism present in the tank had been turned off, probably to economize electrical energy. Possibly the stirring time in tank T6 was well reduced, such that a residue of cream formed in the tank since fat is less dense and tends to rise in the milk. When the expansion tank is emptied under these conditions, the cream may remain in the tank until the end of the emptying procedure and deposit itself on the bottom of the tank, justifying the elevated values of fat in the effluent.

The variables of BOD_5 and COD (Table 3) were above the limits established by the Deliberative Norm Group nº 01, which recommends limits of 60 $mg.L^{-1}$ and 180 $mg.L^{-1}$, respectively (MINAS GERAIS, 2008). The effluents evaluated in this study should not have been discharged into water courses, since the values found for BOD_5 and COD were way above the permitted values. Thus the effluents from tanks T5, T7, T12 and T13 should be submitted to treatment before discharging them into the streams, in order to conform to the Brazilian legislation.

Results above those recommended by the legislation were expected, since effluents produced by dairy activities are rich in organic matter. According to Schaafsma; Baldwin; Streb (1999), effluents from dairy farms show high concentrations of nutrients and organic matter, resulting in a greater oxygen demand to stabilize the organic matter.

There were no significant differences between the values for BOD_5 in the effluents from the different expansion tanks ($p>0.05$), and these were similar to the values found by Wood et al. (2007) and Newman; Clausen; Neafsey (1999), who measured the effluents produced from cattle installations and found values of 2683 $mg.L^{-1}$ and 2811 $mg.L^{-1}$ respectively, for the BOD.

The BOD provides information about the biodegradable fraction of the organic matter in the water, since it indirectly determines the concentration of biodegradable organic matter according to the oxygen demand exerted by microorganisms during respiration (JOUANNEAU et al., 2014). According to von Sperling (2005), the BOD and COD are the most important variables in characterizing the degree of pollution of a water body, since they portray the organic matter content in an indirect way, and are therefore indications of the potential consumption of dissolved oxygen.

With respect to the COD, the effluents from tanks T6, T8, T10, T14 and T15 presented significantly higher values ($p<0.05$) than those from the other tanks (Table 3), probably because the effluents from these tanks had higher milk concentrations. The effluents from the other tanks showed values for COD similar to those found by Wood et al. (2007), who, on evaluating the effluents produced by dairy farms, found COD values of 6690 mg/L. However, Moraes; Paula Junior (2004), on evaluating the anaerobic biodegradability of cattle farming residues, and Pelissari et al. (2013), on evaluating the efficiency of wetlands systems in the treatment of effluents from dairy cattle farms, found COD values of 1520 mg/L and 1008 mg/L, respectively, values way below those found in the effluents from the expansion tanks.

In the characterization of the effluents from the expansion tanks, they were found to contain high concentrations of organic matter, as represented by the values for BOD and COD, justifying the need for methods/ technologies and for specific legislation in Brazil to determine the correct treatment and destiny.

Since the BOD only measures the biodegradable fraction, the closer the value of the BOD is to that of the COD, the easier the biodegradation of the effluent. Thus the BOD/COD ratio predicts how biodegradable the sample is, and as from this ratio, one can indicate the type of treatment most suitable for the effluent to be treated. In the case under analysis, the BOD/COD ratio was not that low, but it was not close to 1 (one). On the other hand the ratio indicated that there were considerable amounts of biodegradable matter in the expansion tanks, since biological treatments are commonly applied to effluents with BOD/COD ratios of 3.00, and the mean of the ratios in the present study was 2.93. Thus the treatment indicated for the effluents from the expansion tanks could be biological.

3.2 MEASUREMENTS OF THE CORRELATION BETWEEN THE VARIABLES STUDIED FOR THE EXPANSION TANKS EFFLUENTS

The study of the correlation between the variables studied was summarized in a matrix. In the correlation matrix, it was concluded there was a relationship between the variables for values close to -1 or +1. Thus Table 4 was established, taking the correlation between variables into account as from Pearson coefficients greater than 0.8.

Table 4. Correlations between the variables as from Pearson’s coefficient

Variable	Correlated variables (Pearson’s coefficient >0.8)
pH	Alkalinity
Alkalinity	pH
Hardness	Fats & oils
BOD	Turbidity
COD	Fats & oils, total phosphorous, nitrogen and turbidity
Fats & oils	Hardness, COD, nitrogen
Total phosphorous	COD, nitrogen, TDS
Nitrogen	COD, Fats & oils, total phosphorous and turbidity
Turbidity	BOD, COD, nitrogen & TDS
TDS	Phosphorous and turbidity

Table 4 shows significant correlation between the pH value and alkalinity, that is, the higher the pH value, the greater the alkalinity of the effluent. The pH value represents the concentration of hydrogen ions, giving an indication of the acidity, neutrality or alkalinity condition of the water. The alkalinity represents the amount of ions in the water that react with and hence neutralize the hydrogen ions, and is therefore a measure of the capacity of the water to neutralize the acids (capacity to resist pH changes). Primavesi et al. (2002) used the multivariate analysis to evaluate the quality of the water close to the milk production system and also observed that the pH value correlated with the alkalinity.

In the present study, an expressive relationship was found between the fats and oils and the variables of hardness, COD and nitrogen, such that the correlation was significantly positive, that is, the effluents with higher fats and oils contents showed higher values for hardness, COD and nitrogen. The COD, for its part, presented a high correlation with phosphorous, nitrogen and turbidity, signifying that effluents with higher COD values showed higher values for phosphorous, nitrogen, turbidity and fats and oils.

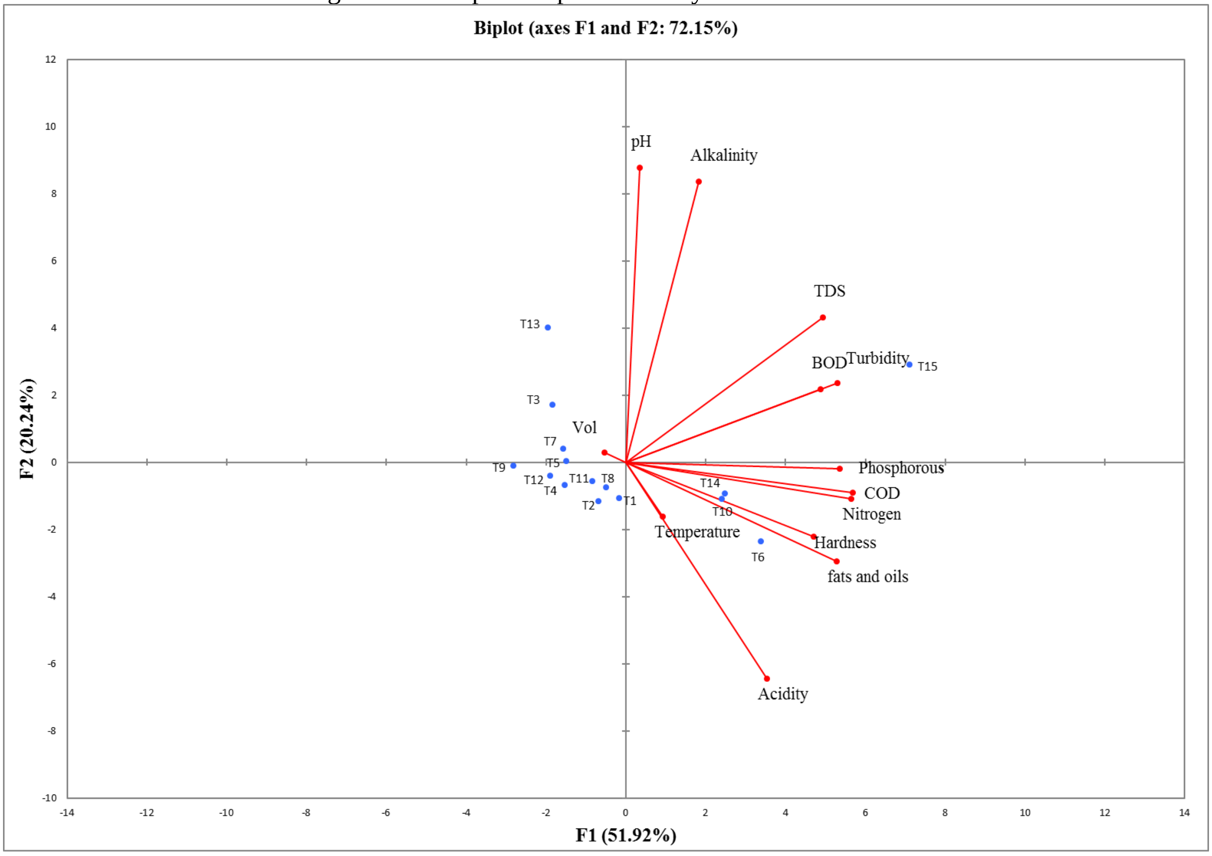
There was also a significantly positive correlation for nitrogen with COD, phosphorous, turbidity and fats and oils, meaning that the effluents with greater nitrogen contents showed higher values for COD, phosphorous, turbidity and fats and oils.

Table 4 also shows a positive relationship between the variables of BOD and turbidity. The more turbid effluents showed higher values for BOD, COD, nitrogen and TDS, this correlation probably being connected to higher milk concentrations in the effluent. There was also a significantly positive relationship between the variable of phosphorous with the COD, nitrogen content and TDS. Thus the higher the phosphorous concentration, the greater the values found for COD, nitrogen and TDS. According to von Sperling (2005), the phosphorous in water is mostly present in the forms of orthophosphate, polyphosphate or organic phosphorous, and these phosphorous constituents are commonly present in the form of the dissolved solid, which could explain the strong positive correlation between phosphorous and TDS.

3.3 PRINCIPAL COMPONENT ANALYSIS

For the success of the principal components' analysis, it is desirable that the first two principal components accumulate a percentage of the explained variance equal or above 70%. In the present study, the first two components explained 72.15% of the variance. The first principal component, or the factor most important in the evaluation of the expansion tank effluents in this analysis, explained 51.92% of the data variance, and the second principal component explained 20.24% of the variance (Figure 1).

Figure 1. Principal components analysis of the variables.



In the first component, the variables of COD and nitrogen presented values above 0.9, indicating that these variables were the most significant ones in defining the characterization of the effluents from the expansion tanks. That is, the variables that most influenced the characteristics of the tank effluents were related to the COD and nitrogen concentrations, since they had the highest values for the squared cosines in the F1 component (Table 5).

Table 5. Squared cosines of the variables

	F1	F2
Temperature	0.025	0.029
pH	0.004	0.860
Acidity	0.356	0.464
Alkalinity	0.096	0.780
Hardness	0.632	0.055
BOD	0.679	0.053
COD	0.924*	0.009
Fats & oils	0.796	0.097
Phosphorous	0.822	0.000
Nitrogen	0.909*	0.013
Turbidity	0.803	0.062
TDS	0.696	0.207
Volume	0.008	0.001

(*): larger squared cosine values for the variables correspond to variables that are important in characterizing the effluent

The PCA allows one to observe the behavior of the characteristics of the 15 tanks. According to Figure 1, tanks T6, T10, T14 and T15 belong to a different group than the other tanks. As already noted, it is probable that the people responsible for the sanitization of these four tanks left more milk residues in the tanks before starting the washing process, since they presented values significantly higher for the nitrogen content and COD.

3.4 GROUPING ANALYSIS OF THE EXPANSION TANK EFFLUENTS

Using the grouping analysis to study the 15 expansion tanks (Figure 2), including the variables of temperature, pH, acidity, alkalinity, BOD_5, COD, total phosphorous, nitrogen, fats and oils, hardness, turbidity and total dissolved solids, the formation of 6 groups was detected. Elements in the same class tend to group together, occupying the same region on the graph (Figure 2).

The groupings permit a perception of the general variation in the characteristics of the tank effluents investigated, making it possible to detect the tanks with alterations in the variables analyzed. With respect to T13, a change in the worker responsible for the sanitization of the tank was observed for each effluent collection. This factor could have influenced the certain difference of this tank in relation to the others.

The effluents from tank T9 showed lower values for a variety of the variables analyzed when compared with the effluents from the other tanks, namely the values for pH, alkalinity, nitrogen, COD, TDS and fats and oils, which showed low concentrations for the effluents from this tank. Possibly this tank contained a smaller amount of milk on starting the sanitization procedure, explaining this particularity.

The effluents from tank T15 presented significantly higher ($p<0.05$) values for diverse variables when compared with the effluents from the other tanks, namely for the concentrations of nitrogen, fats and oils, and for the values for COD and TDS. These facts indicated that this tank discharged an effluent with a larger amount of milk, explaining the separation of this tank from the others on the grouping dendrogram.

As from the fourth group, the tanks presented approximately 98% of similarity. Analyzing the variables separately and comparing with the grouping analysis, taking all the groups into consideration including the three tanks with particularities described above, even so there was more than 88% similarity between the effluents from the 15 tanks (Figure 2). Thus it was proposed to consider the characteristics of all the tanks in a unified way to describe the effluents generated during the cleaning of the expansion tanks used in the bulking process of raw milk in Brazil, presenting Table 6 to show this characterization.

Figure 2. Dendrogram generated by the grouping by similarity of the effluents from the expansion tanks according to the variables of: temperature, pH, acidity, alkalinity, BOD5, COD, total phosphorous, nitrogen, fats & oils, hardness, turbidity and TDS.

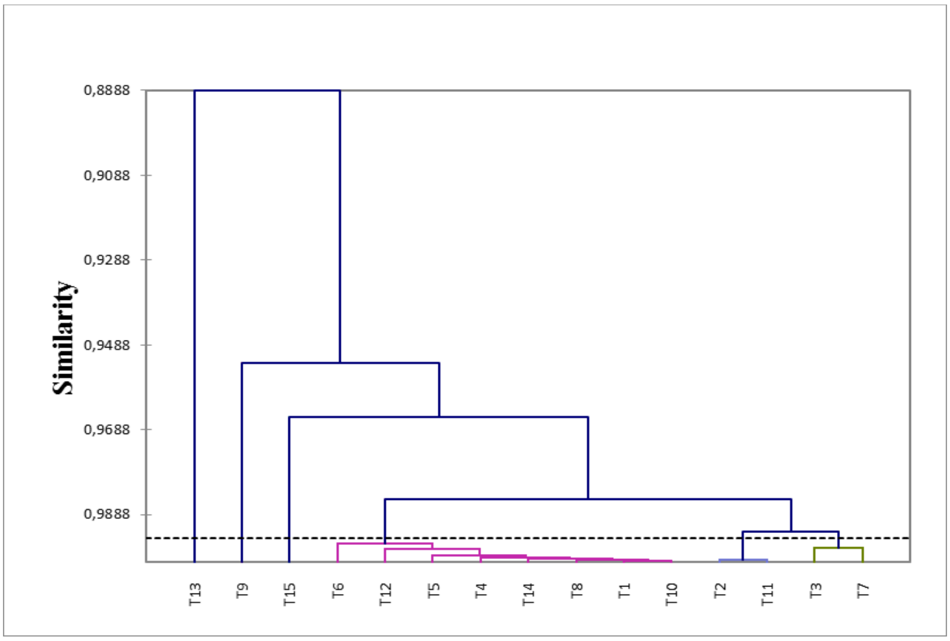


Table 6. Characterization in a unified way to describe the effluents generated during cleaning of the expansion tanks used in the bulk raw milk process in Brazil

Variables	Minimum	Maximum	Mean	Standard deviation (SD)
Temperature (ºC)	21.03	29.60	24.58	2.74
pH	6.00	12.54	7.88	2.01
Acidity (mg $CaCO_3.L^{-1}$)	0.00	233.54	95.80	73.31
Alkalinity (mg $CaCO_3.L^{-1}$)	32.62	2141.00	409.54	625.90
Hardness (mg $CaCO_3.L^{-1}$)	45.47	272.47	135.43	64.46
BOD_5 ($mg.L^{-1}$)	1471.20	7021.38	2543.99	1370.66
COD ($mg.L^{-1}$)	4154.23	13624.54	6971.63	2680.56
Fats & oils ($mg.L^{-1}$)	107.42	1255.53	472.28	338.01
Total phosphorous ($mg.L^{-1}$)	0.40	67.81	21.72	21.86
Nitrogen ($mg.L^{-1}$)	41.10	422.00	154.78	112.23
Turbidity (NTU)	31.36	4478.00	1046.64	1019.05
TDS ($g.L^{-1}$)	0.42	5.96	1.80	1.50
Volume (L)	17.16	67.83	34.24	13.81

The mean value found for the pH of the effluents from the expansion tanks (Table 6) was close to the values of 7.2, 7.6 and 7.2 found, respectively, by Rico; García; Rico (2011), Ruane et al. (2011), and Pelissari et al. (2013), on characterizing the effluents from dairy cattle farms.

On evaluating the anaerobic biodegradability of residues from cattle farms, Moraes; Paula Junior (2004) found residual waters with values for acidity and alkalinity of 265 mg $CaCO_3$/L and 151mg $CaCO_3$/L, respectively, considerably different from the mean values found in the effluents from the expansion tanks (Table 6).

The mean value found for BOD_5 (Table 6) was close to the values reported by Newman; Clausen; Neafsey (1999), Dunne et al. (2005), and Wood et al. (2007), with values of 2680 mg/L, 2300 mg/L and 2811 mg/L, respectively, when characterizing the effluents from cattle farming installations. A high standard deviation was found for the values of BOD_5 of the treatments, this fact being directly related to the daily handling of the installations and forms of sanitization used, directly influencing the composition of the effluent produced.

The mean value found for COD (Table 6) was similar to that verified by Wood et al. (2007) of 6144 mg/L and Ruane et al. (2011) of 5750 mg/L, on characterizing the effluents from cattle farms, with a view to treating them. However the COD found was much smaller than that reported by Rico; García; Rico (2011) of 14,280 mg/L and much higher than that found by Silva; Roston (2010) of 1026 mg/L, when characterizing the effluents generated by cattle farm installations.

The phosphorous concentration found in the effluents from the expansion tanks (Table 6) was close to that reported by Newman; Clausen; Neafsey (1999) of 25.7 mg/L, when studying the influence of cold climates on the treatment of residual waters from dairy agro-industries. This variable also presented a high standard deviation (Table 6). Ruane et al. (2011) found a higher value of 36.01 mg/L and Rico; García; Rico (2011) an even higher value of 86 mg/L, indicating the great variability of this parameter.

The nitrogen concentration presented high standard deviations and the value found for the effluents from the expansion tanks (Table 6) was relatively close to the valor reported by Newman; Clausen; Neafsey (1999) of 102 mg/L for the effluents from cattle farms, but much smaller than that reported by Rico; García; Rico (2011) of 1140 mg/L, and at the same time, much higher than that found by Silva; Roston (2010) of 14.06 mg/L and by Pelissari et al. (2014) of 69 mg/L.

It should be noted that the high standard deviations noted for the variables analyzed demonstrated an elevated degree of variability in the amount of water and detergents used, amongst other components employed. Wood et al. (2007) also reported an elevated value for the standard deviation, although these authors were characterizing effluents produced in a milking room.

Considering the tanks that discharged their effluents into streams, the characterization of these effluents all showed the non-conformity of the variables with the standards stipulated by the Brazilian hydric legislation currently in force. With the exception of the temperature and the pH value, all the other variables showed values above those stipulated by the legislation (BRASIL, 2011) for discharge into surface water bodies. Thus some actions must be implemented to minimize the impact of these effluents.

Greenway; Woolley (2000) quantified the assimilation of 0.6-156 mg/L of nitrogen and 1.6-37.5 mg/L of phosphorous by *Typha* spp in wetlands systems. Thus the effluents generated during the cleaning of the expansion tanks used in the raw milk bulking process can be indicated for treatment by the wetlands system, since they presented mean nitrogen and phosphorous contents of 154.78 mg/L and 21.72 mg/L, respectively (Table 6).

In the wetlands systems, the high organic matter values represented by the BOD and COD (Table 6) are degraded, due to the activity of microorganisms to obtain energy and carbon for their metabolism and reproduction. This occurs by way of oxidation-reduction reactions of the organic and inorganic compounds present in the effluents.

According to Silva; Roston (2010), the wetlands systems show great potential for application in the treatment of residues from dairy farms, and are also easy to operate and maintain. The authors demonstrated that these systems were efficient in reducing the BOD, COD, solids, nitrogen and phosphorous loads. Sharma et al. (2013) also indicated the wetlands systems as feasible in the treatment of residual waters, and examined a milking room in Hokkaido, Japan. Mantovi et al. (2003) demonstrated that the use of the wetlands system provided an adequate treatment to reduce the levels of pollutants to acceptable levels for discharge into surface waters in effluents produced during dairy farm activities.

Zhang et al. (2014) indicated the wetlands systems as promising in the global context, considering the need for low cost and sustainability. These authors detected efficiency in the high level of removal of COD, nitrogen and phosphorous, but these removals showed seasonal variations due to the activity of the macrophyte, that is, it can act differently according to the season. However, seasonality did not influence the characterization of the effluents to be treated. Dunne et al. (2005), on determining the quality and quantity of effluents produced on an Irish farm, and the seasonal efficiency of the treatment of the effluent studied, concluded there was no seasonal variation in either the volume of effluent produced or the concentration of the effluent quality variables (taking into consideration the entrance rates). The same authors suggested that the effluents coming from the farm yards should be managed since they contained considerable amounts of nutrients and contaminants.

The evaluation of the wetlands units in the literature showed that this type of system was adequate and presented great potential for application on dairy cattle farms in Brazil, due, principally, to the favorable climate in various parts of the country, to the low costs (implantation, operation and maintenance), operational simplicity, good efficiency in the removal of organic matter and nutrients and also good possibilities for re-usage of the treated effluent. However, despite this and other available technologies that can be applied to the treatment of residual waters in the production of the raw material for milk industrialization, effluent treatment does not predominate in this segment of the country. There is thus a need to divulge and establish criteria that stimulate and make feasible the implementation of technologies for the treatment of effluents in Brazilian milk producing units.

4 CONCLUSION

None of the effluents from the 15 expansion tanks evaluated had been treated, and some of them discharged the cleaning water directly into water courses. As from the results of the analyses of the effluent samples, it was concluded that the majority of the variables were above the legal Brazilian standards. It is therefore necessary that the individuals responsible for the sanitization of the expansion tanks used for the bulk storage of raw milk in Brazil, receive training for the adequate treatment and destiny of the effluents coming from these tanks.

It was shown that the largest tanks (category larger than 2000 L) did not use more water to sanitize the tanks, the amount being mainly the responsibility of the task operator. The water volume used in washing the tanks should be rationalized by way of the training and consciousness of the operators, resulting in changes in their behavior, without compromising the sanitization procedure. According to the literature, the concentrations found for phosphorous, nitrogen, COD and BOD in the effluents produced during the cleaning of the expansion tanks, could be treated by the wetlands systems. In addition, the COD/BOD ratio found showed that the treatment indicated for these effluents could be biological. Future studies are suggested to analyze the feasibility of this suggestion by implanting such a system for the treatment of the effluent of an expansion tank and determining the reductions in the concentrations of the variables.

These results also serve as a technical reference in the evaluations of the impact on the environment of sanitizing the equipment used by the sector producing the raw material in the milk production chain. In addition the data could aid in promoting public policies for the regions with higher concentrations of milk producing farms in Brazil.

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