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Physicochemical evaluation of acid whey obtained from doble crema cheese produced in Belén, Boyacá, Colombia

Evaluación fisicoquímica del lactosuero ácido obtenido del queso doble crema producido en Belén, Boyacá, Colombia

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Abstract The composition of acid whey makes it less usable, and it is an environmental and technical problem. This research aims to estimate the physicochemical characteristics of the doble crema cheese acid whey, from nine factories in the municipality of Belén in Boyacá. Laboratory methods were validated using the Lactoscan analyzer determinations and comparing them with reference methods. Accuracy was expressed in terms of relative error (% E_{det}) and f- and p-values; then, correlation curves were elaborated. To perform the physicochemical characterization of whey, fat, Solids-Not-Fat (SNF), protein, lactose, pH, total ash, calcium, density, and acidity were determined. The statistic of the comparison between analysis methods showed low percentages of relative error, there were no significant differences, and they had correlation coefficients greater than 0,8. The results of the physicochemical characterization showed differences in the content of protein, SNF, lactose, fat, ash, calcium, acidity, and pH. Each manufacturer has a manufacturing process for cheese and whey. There were no statistical differences for SNF, density, lactose, fat, ash, and pH within each company. However, there were differences between protein and acidity, even though companies work with the same quality of raw material and processing, there are deficiencies in the coagulation, drainage, and conservation of the whey. The analyzed acid whey is a good source of protein, calcium, lactose and has low fat content, which is attractive for manufacturing food products.

Keywords: cheesemaking; cheese; whey; comprehensive food utilization.

Resumen La composición del lactosuero ácido hace que sea menos aprovechable y lo convierte en un problema ambiental y técnico. El objetivo de esta investigación consistió en estimar las características fisicoquímicas del lactosuero ácido producto del proceso de elaboración del queso doble crema de nueve empresas del municipio de Belén en Boyacá. Se llevó a cabo una validación de métodos de laboratorio entre las determinaciones obtenidas del equipo analizador Lactoscan y los métodos de referencia. La exactitud se expresó en términos de error relativo (% E_{det}), la precisión mediante valor f y p, y se elaboraron curvas de correlación. Para la caracterización fisicoquímica de los lactosueros se determinó el contenido de grasa, SNG, proteína, lactosa, pH, cenizas totales, calcio, densidad y acidez. El análisis estadístico de la comparación entre métodos de análisis arrojó bajos porcentajes en el error relativo, no hubo diferencias significativas y se encontraron coeficientes de correlación mayores a 0,8. Los resultados de la caracterización fisicoquímica arrojaron diferencias para el contenido de proteína, SNG, lactosa, grasa, cenizas, calcio, acidez y pH. Cada empresa elabora el queso y el suero de manera diferente, en ellas no se registraron diferencias estadísticas para SNG, densidad, lactosa, grasa, cenizas y pH, pero sí para proteína y acidez. Esto se debe a que trabajan con la misma calidad y procesamiento de la materia prima, pero hay falencias en la coagulación, desuere y conservación del suero. Los lactosueros ácidos analizados son buena fuente de proteína, calcio, lactosa y tienen bajos contenidos de grasa, lo que los hace atractivos para la elaboración de productos alimenticios.

Palabras clave: fabricación de queso; queso; suero de leche; lactosuero; aprovechamiento integral de los alimentos.

Introduction

Whey is defined as a by-product of cheesemaking (Simonis *et al.*, 2019) and represents 80 – 90% of the volume of milk (Skryplonek *et al.*, 2019). It contains about 0,6 – 0,8 % of protein, 0,4 – 0,5 % of fat, 4,5 – 5,0 % of lactose, and 8,0 – 10 % of mineral salts. Depending on the coagulation method, it is possible to obtain acid whey (pH < 5,0) or sweet whey (pH between 6,0 and 7,0) (Kravtsov *et al.*, 2020). Globally, 50 % of the whey is processed using expensive technology, this exposes the current situation due to not having economic and environmentally friendly methodologies that favor its total and global use. The high production of whey (190 billion Kg/year) and the quality of its nutrients make it a promising source for food and bioengineering processes (Skryplonek *et al.*, 2019; Merkel, Fárová *et al.*, 2021; Rocha-Mendoza *et al.*, 2021; Fischer & Kleinschmidt, 2021; Shelke *et al.*, 2022). Technological advances have transformed it into other products such as proteins, bio-proteins, biopolymers, bioethanol, hydrogen, methane, and energy (Karim & Aider, 2022). European countries have the technology to take advantage of over 95 % of the whey they produce. It is used to prepare high-value products with functional properties of technological and commercial interest (Costa *et al.*, 2018; Buhler., 2019; Conde-Báez, 2019; Choi *et al.*, 2019; Da Silva Duarte *et al.*, 2020).

In Colombia, the cheese consumption has increased, so did whey production, thus generating around 1100 million Kg/year (Federación Colombiana de Ganaderos [FEDEGÁN], 2021). In the Department of Boyacá, the Chiquinquirá Valley and the Industrial Corridor are the largest producers of cheese and generate high volumes of whey. The cheese companies from the municipality of Belén produce on average 2375,128 kg/day of doble crema cheese whey (Torres, 2021); 60 % of the companies transform it into ricotta cheese, and the rest is used as animal feed or is dumped at effluents. Moreover, foreign trade policies in Colombia have given free access to powdered whey; by 2021, it was the third most imported dairy product 8811 t (Ministerio de Agricultura y Desarrollo Rural, 2021). This scenario should change, so actions from the government, industry, and academia must be favored so that the country processes its own whey.

Consumerism currently seek products that provide health benefits, and food designers focus on raw materials with functional components; whey fits in that line (Simonis *et al.*, 2019; Skryplonek *et al.*, 2019). There are many studies published that enable a better knowledge of its composition and behavior in the face of industrial processes (Karim & Aider, 2022), and providing and designing cost-effective forms of use, sustainability and management (Zandona *et al.*, 2021).

The composition of whey changes according to the type and way of cheesemaking, but also to the origin of the raw material, the heat treatment, the coagulation and conservation methods (Rocha-Mendoza *et al.*, 2021). Protein content is the most valuable constituent of whey due to its biological value, wide variety, and application of functional properties, as well as a plethora of health benefits associated with bioactive peptides derived from proteolytic processes (Skryplonek *et al.*, 2019; Simonis *et al.*, 2019; Lajnaf *et al.*, 2020; Rocha-Mendoza *et al.*, 2021; Zandona *et al.*, 2021; Da Silva Duarte *et al.*, 2020; Bustamante *et al.*, 2021; Karim & Aider, 2022). Lactose is considered one of the basic and most abundant components of whey (Menchik *et al.*, 2019), it is an ideal substrate for fermentation processes and to obtain valuable products (Ramírez-Navas, 2012). It offers health benefits such as favoring the absorption of several minerals, registering low glycemic index, and generating prebiotic effects by the production of galactooligosaccharides (Fischer & Kleinschmidt, 2021). In the industrial sector, it can be used in confectionery, bakery (Posada *et al.*, 2011; Zandona *et al.*, 2021), and to obtain lactic acid, polylactic acid (PLA), pyruvic acid, acrylic acid, lactitol, mannitol, lactosyl urea, lactosaccharose, sorbitol, among others (Asunis *et al.*, 2020; Zandona *et al.*, 2021; Karim & Aider, 2022). Whey is low-fat, but it could confer turbidity and oxidation compounds due to deficiencies in preservation methods (Smith *et al.*, 2016; Skryplonek *et al.*, 2019). It contains a substantial number of phospholipids, which is attractive for populations who suffer cardiovascular events (Simonis *et al.*, 2019; Rocha-Mendoza *et al.*, 2021). The color of the whey is greenish yellow, and the solids content is around 6,7 % (Skryplonek *et al.*, 2019), but they may increase due to a fine content of casein and fat, which gives a white color to some of them (Ramírez-Navas & Rodríguez De Stouvenel, 2012). Whey acidity is caused by the high content of lactic acid (Merkel, Fárová, *et al.*, 2021), acid whey contains a greater amount than sweet whey, thus generating industrial disadvantages due to the instability of the lactate ions in drying processes (Rocha-Mendoza *et al.*, 2021).

Whey with high lactic acid content, low pH, and a high concentration of minerals cannot be used in the industry and becomes an environmental and technical problem, especially in small and medium-sized companies (Skryplonek *et al.*, 2019; Merkel, Fárová, *et al.*, 2021). It is an obstacle in the industry because the behavior of lactose in its amorphous state after drying and spraying processes generate crystalline forms and very hygroscopic powders, leading to products with a high degree of clumping and lower shelf life (Nielsen *et al.*, 2021; Bustamante *et al.*, 2021; Merkel, Fárová *et al.*, 2021; Rocha-Mendoza *et al.*, 2021). Therefore, other techniques should be used for the fractionation and concentration of proteins (Ramírez-Navas *et al.*, 2018), lactose, and minerals or use acid whey directly in food processing. This research aims to contribute to the study of the chemical and physical characteristics of doble crema cheese acid whey in dairy companies from Belén, Boyacá, Colombia, and then have a basic knowledge to propose potential uses, value generation, and business models that lead to a real application.

Materials and Methods

The study was carried out in nine doble crema cheesemakers located in the municipality of Belén, Boyacá, Colombia. It was carried out in two phases, as described in Pineda (2023): Phase I. Comparison of methods; and Phase II. Physicochemical characterization of the acid whey produced by these companies,

Phase I. Comparison of methods

The objective was to validate the accuracy, precision, and correlation of the physicochemical characterization made using the Lactoscan DP Milkotronic ultrasonic milk analyzer against the results of Association of Official Agricultural Chemists (AOAC) reference methods employed by an external certified laboratory (**Table 1**). The fat percentage was determined using AOAC 2000.18 (AOAC, 2019), SNF was determined by gravimetry, protein content was determined using AOAC 991.20I (AOAC,2019), lactose was determined using AOAC 930.28 (AOAC, 2019) and pH was determined using AOAC 981.12 (AOAC, 2010). A dairy company participating in the research was selected and 18 acid whey samples of 500 mL were taken randomly at the draining stage prior to curd salting at days 1, 15, and 30. Samples were stored and transported to the laboratories in an expanded polystyrene refrigerator at a temperature of 4 ± 0,1 °C. All samples were analyzed on the same day of sampling.

Accuracy was expressed in terms of determined error (Edet) and relative determined error (% Edet), preciseness through the F-value, and probability of significance (*P* - value) through a Latin Square Design. The analysis of variance (ANOVA) was performed to determine the dispersion and verify the reliability of the data given by the Lactoscan. Significant differences between group means were estimated at a 95 % significance level. The characterizations were made in triplicate. The data were tabulated and processed through the R statistical software version 4.0.2. To compare the methods, the following hypotheses were proposed: $H_{Research}$: "there are no significant differences between the results of the physicochemical analyses carried out in the Lactoscan and those of the laboratory for the acid whey samples". H_{null}: "There are significant differences between the results or at least in one of the physicochemical analyses carried out in the Lactoscan and those of the reference laboratory for the acid whey samples." Correlation curves were made, obtaining linearity parameters such as slope of the line, intercept, and the correlation coefficient.

Table 1

Physicochemical characterization and reference methods employed by the external laboratory

Physicochemical Analysis	Method employed in the external laboratory	Principle
Fat (% m/m)	AOAC 2000.18 (AOAC, 2019)	Gerber's fat method by weight
SNF (%)	Ultrasound	Ultrasound
Protein (% m/m)	AOAC 991.20I: (AOAC, 2019)	Kjeldahl
Lactose (% m/m)	AOAC 930.28: (AOAC, 2019)	Gravimetric method
pH	AOAC 981.12: (AOAC, 2010)	Potentiometer

Note. This table presents the physicochemical analyses and AOAC methods employed for each determination.

Phase II. Physicochemical characterization of acid whey

After verifying the accuracy, precision, and correlation of the test results, the physicochemical characterization of the doble crema cheese acid whey produced by nine dairy companies in the municipality of Belén, Boyacá, was carried out using the ultrasonic milk analyzer. For this, 27 samples of 1000 mL each were taken over three process dates (1, 15, and 30 days) immediately after the draining stage prior to curd salting. Samples were stored and transported to the UNAD Duitama chemistry laboratory in an expanded polystyrene refrigerator at a temperature of 4 ± 0,1 °C. All samples were analyzed on the same day of sampling. The fat (%, m/m) was determined using the Lactoscan following the Gerber method, SNF (%) by gravimetry, protein (%, m/m) by the Kjeldahl method, lactose (%, m/m) and applying the Official Methods of Analysis of AOAC it was determined pH by AOAC 981.12 (AOAC, 2010), total ash percentage by AOAC 945.46 (AOAC, 2019), calcium in mg/100 g by AOAC 985.35 (AOAC, 2019), density by AOAC 962.37 (AOAC, 2000), acidity as lactic acid (%, m/m) by AOAC 947.05 (AOAC, 2000) and titration with 0.1 N NaOH; all previous analyses were conducted through analytical tests in the CEAD Duitama Chemistry laboratory, except for the calcium content which is calculated in a certified external laboratory.

All analyses were performed in the CEAD Duitama Chemistry laboratory, except for the calcium content that was determined in a certified external laboratory. The results obtained were compared with the values allowed in the resolution 02310:1986, "Dairy By-products" (Ministerio de Salud, 1986). A completely randomized block design was formulated, the analysis of variance (ANOVA) was conducted, and significant differences between the means were estimated at a 95 % significance level. The characterizations were made in triplicate. Quality control graphs were made (*graphs X*) to determine whether the physicochemical parameters of acid whey meet the regulatory standards, and to analyze the variability of the process and its impact on the quality of whey and doble crema cheese. The data were tabulated and processed through R statistical software version 4.0.2.

RESULTS AND DISCUSSION

Phase I. Comparison of methods and analysis

Table 2, shows results obtained for each physicochemical parameter determined by the Lactoscan SP Milkotronic ultrasonic milk analyzer and by the conventional reference methods.

The means obtained by the comparison method for protein (%), fat (%), and density (Kg/m³) were consistent since they recorded a low dispersion between data. For the parameters SNF (%), lactose (%), and pH, the value of the standard deviation is higher, thus they generate a higher degree of dispersion. However, it is inferred that the obtained values give reliable information of the average of each determination when using the Lactoscan.

The mean value of the protein content (%) obtained by the Lactoscan has an accuracy of 0,03 % with respect to the value accepted as a reference, the difference between means had an *f*-value of (0,02) and a *p*-value > 0,05, indicating homogeneity in the measurements by both methods. The correlation coefficient was 0,81, which indicates a linear association between methods. Since the automated method uses the Kjeldahl method as a principle, the same one that was employed to determine the AOAC 991.20I (AOAC, 2019) reference value, it leads to these results.

Table 2

Statistics to verify the precision, accuracy, and correlation between the results obtained by the Lactoscan and the reference methods.

Statistical determination	Protein (%)	Fat (%)	SNF (%)	Lactose (%)	Density (Kg/m³)	pH
Mean (EL)*	2,75	2,38	7,99	6,35	1,027	4,53
Mean (MR)	2,75	2,02	7,98	6,25	1,027	4,60
Dev. Standard (EL)*	0,08	1,36	0,56	0,40	0,0014	0,16
E_{det}	0,0007	0,36	0,009	0,10	0,0007	-0,07
% E_{det}	0,03	18,06	0,11	1,60	0,07	-1,43
Accuracy: F-value ($F_{t\ 1.12}$: 4,747	0,02	3,87	0,01	3,09	0,04	3,72
Accuracy: *P*-value (α=0,05)	0,89	0,052	0,92	0,056	0,84	0,053
Slope Intercept	1,73	1,08	0,9838	0,4613	0,0552	0,0552
Slope of the line	0,35	1,447	1,0016	1,0899	0,9469	0,9469
Correlation Coefficient	0,81	0,96	0,90	0,97	0,96	0,82

Note. * EL: Lactoscan SP Milkotronic ultrasonic milk analyzer. **MR: Reference methods employed in the external laboratory.

The fat content (%) shown by the Lactoscan equipment presents a deviation of 18,06 % from the reference value. The f-value (3,87) and the *p*-value (0,052) indicate that there are no statistical differences between the measurements. The correlation coefficient (0,96) shows a strong positive association between the methods, maybe because the automated method uses the Gerber method as a principle, the same that was used to determine the AOAC 2000.18 (AOAC, 2019) reference value. The Lactoscan SNF (%) determinations present an accuracy of 0,11 %, an f-value of 0,1 with a *p*-value > 0,05, and a positive correlation of 0,90; thus, they show uniformity between the methods. Regarding lactose (%), the comparison of results showed an accuracy of 1,6 %, an f-value of 3,09 with a *p*-value > 0,05, and a positive correlation of 0,97; thus, coherence between the two methods was observed. The density values (Kg/m^3) obtained by the procedures recorded an accuracy of 0,07 %, statistical homogeneity (f = 0,04, p = 0,84), and a positive linear correlation of 0,96; thus, it indicates equality between results. Regarding pH, the deviation from the mean reference value was lower by 1,43 %, indicating that the ultrasonic automated equipment determinations were lower than those obtained by the reference values, but the comparisons among determinations are consistent according to the test: f = 3,72, *p*-value > 0,05, and the determination of a positive linear association of 0,82.

Phase II. Physicochemical characterization of the acid whey

Table 3 presents the results of each physicochemical parameter given by the Lactoscan SP Milkotronic ultrasonic milk analyzer for the acid whey samples from each company. Quality control graphs (*X-graphs*) of each parameter are presented in **Figure 1**. Each physicochemical determination is discussed below, as presented by Pineda (2023).

The SNF of each acid whey sample showed significant differences among them (p < 0,05). Each company tries to manage the same origin of the raw material and processing; the three sampling dates did not register significant differences (p > 0,05). The whey samples have an average of 7,05 % ± 0, 22 SNF and between 6,67 - 7,43 %, values higher than those shown in other studies. Menchik *et al.* (2019) report 3,3 - 3,7 % ; Lievore *et al.* (2015), 6 % ; Skryplonek *et al.* (2019), 6,4 %; Rocha-Mendoza *et al.* (2021), 6,5 %; Shelke *et al.* (2022), 6,7 %.

Table 3

Physicochemical characteristics of doble crema cheese acid whey.

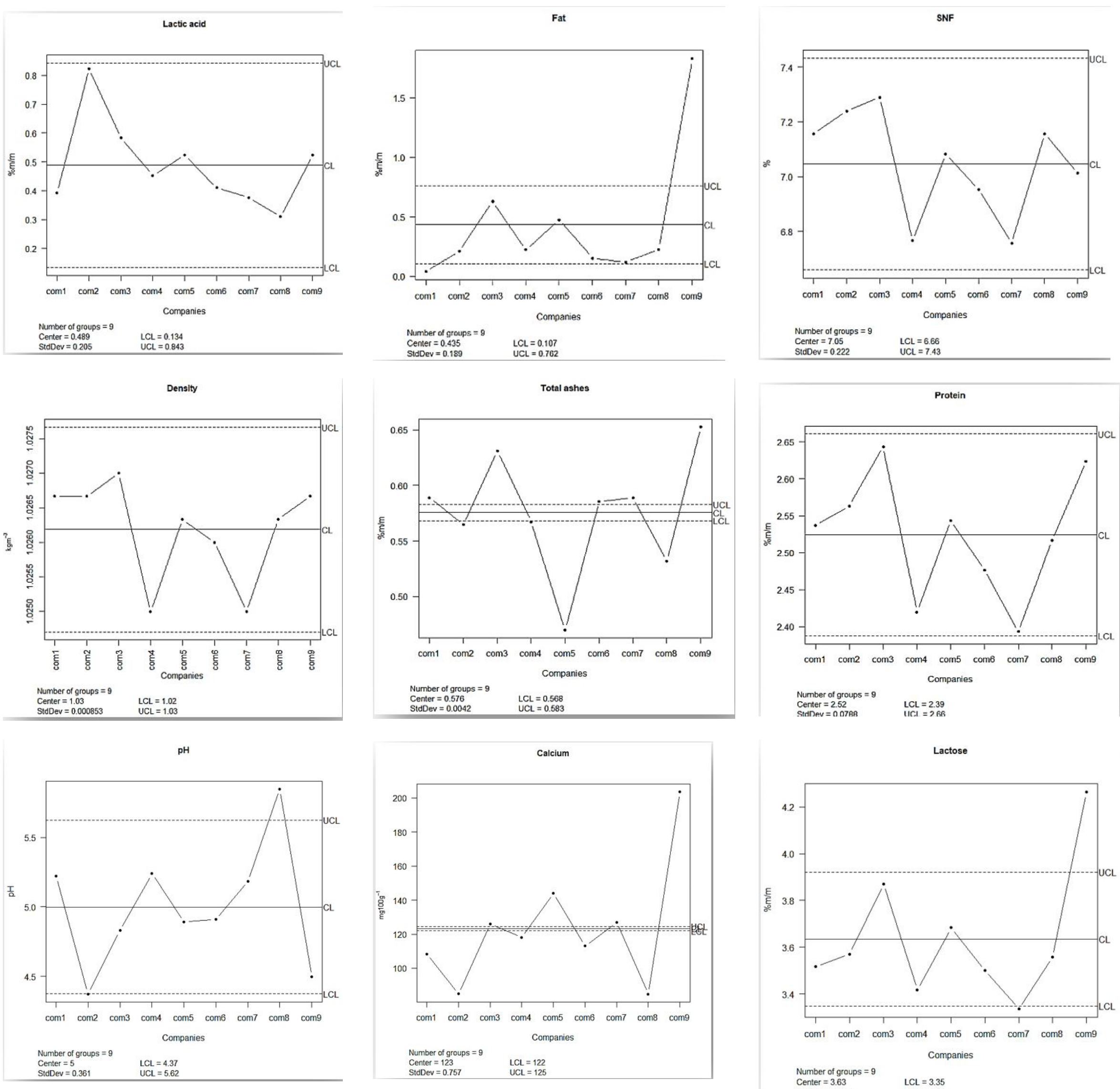
Company	SNF (%)	Protein (%)	Lactose (%)	Fat (%)	Total ash (%)	Calcium (mg/100 g)	Acidity (%)	Density	pH
Comp1	7,16 ± 0,2[a]	2,54 ± 0,1[a]	3, 52 ± 0,1[a]	0,04 ± 0,03[a]	0,59 ± 0,0[a]	108 ± 0,2[c]	0,40 ± 0,1[a]	1,026 ± 0,0[a]	5,22 ± 0,1[a]
Comp2	7,24 ± 0,4[b]	2,57 ± 0,1[b]	3, 57 ± 0,1[b]	0,21 ± 0,4[b]	0,56 ± 0,0[c]	85 ± 0,4[b]	0,84 ± 0,5[b]	1,027 ± 0,0[a]	4,37 ± 0,2[b]
Comp3	7,29 ± 0,04[c]	2,64 ± 0,02[c]	3,87 ± 0,1[c]	0,63 ± 0,1[c]	0,63 ± 0,01[e]	126 ± 0,0[a]	0,58 ± 0,1[c]	1,027 ± 0,0[a]	4,83 ± 0,3[c]
Comp4	6,77 ± 0,06[d]	2,42 ± 0,03[d]	3,42 ± 0,1[d]	0,22 ± 0,1[d]	0,57 ± 0,0[c]	118 ± 0,1[b]	0,44 ± 0,1[d]	1,025 ± 0,0[a]	5,20 ± 0,4[d]
Comp5	7,08 ± 0,1[e]	2,54 ± 0,1[e]	3,68 ± 0,2[e]	0,48 ± 0,3[e]	0,47 ± 0,01[g]	144 ± 0,1[d]	0,52 ± 0,2[e]	1,026 ± 0,0[a]	4,86 ± 0,5[e]
Comp6	6,95 ± 0,2[f]	2,48 ± 0,1[f]	3,49 ± 0,2[f]	0,15 ± 0,2[f]	0,59 ± 0,0a[b]	113 ± 0,2[e]	0,41 ± 0,1[f]	1,026 ± 0,0[a]	4,91 ± 0,6[f]
Comp7	6,76 ± 0,2[g]	2,39 ± 0,1[g]	3,34 ± 0,02[g]	0,12 ± 0,1[g]	0,59 ± 0,0[b]	127 ± 0,2[a]	0,38 ± 0,1[g]	1,025 ± 0,0[a]	5,18 ± 0,7[g]
Comp8	7,16 ± 0,3[h]	2,52 ± 0,1[h]	3,56 ± 0,1[h]	0,23 ± 0,2[h]	0,53 ± 0,01[h]	84,8 ± 0,3[f]	0,31 ± 0,2[h]	1,026 ± 0,0[a]	5,85 ± 0,8[h]
Comp9	7,01 ± 0,6[i]	2,62 ± 0,2[i]	4,27 ± 0,5[i]	3,41 ± 0,1[i]	0,65 ± 0,0[i]	204 ± 0,6[g]	0,52 ± 0,2[i]	1,027 ± 0,0[a]	4,5 ± 0,9[i]

Note. The table presents the statistical means followed by the standard deviations (±). Different letters in the same column indicate significant differences (p ≤ 0,05) according to the LSD test.

The quality graphs (**Figure 1**) present a process with a stability pattern since the SNF contents are within the control limits. However, a high SNF content in whey suggest reviewing and monitoring the draining stage because of the migration of casein solids, which alters the characteristic color (**Figure 2**).

Figure 1

Quality control graphs (-X graphs) of each physicochemical characterization of acid whey samples.



Nota. The authors.

Regarding density, the ANOVA did not register significant differences ($p > 0{,}05$) among companies and sampling dates. Values ranging from 1,024 – 1,027 kg/m^3 were obtained and on average 1,026 ± 0,0 kg/m^3, close to those reported by Skryplonek et al. (2019) of 1,025 g/mL.

The quality graphs (**Figure 1**) presented a stability pattern since all the points are within the control limits. They indicate that the migration of casein solids to whey is constant in all companies and validated the arguments that the draining stage is done differently in each company in terms of the intensity of the cutting, agitation, and temperature management. There is no standard process for making cheese of a specific type; therefore, the cheese yield in these companies must be evaluated.

Figure 2

Acid whey samples obtained after the coagulation process of doble crema cheese.

The statistical analysis indicates that the protein content of the doble crema cheese acid whey recorded significant differences among companies (p = 0,0071) and process dates (p = 0,0476). The whey recorded a protein content of 2,39 – 2,67 % and on average 2,52 % ± 0,08, a content greater than that set in Resolution 2310 (1986) (0,7 %) but close to that reported by Simonis *et al.* (2019) of 2,26 %.

The analysis of protein values from similar studies are variable and depend on several factors ranging from the origin of the raw material to the type of cheese to be made (Smith *et al.*, 2016), but the most influencing factor are milk clotting and obtaining the whey. In enzymatic denaturation processes, the protein content is higher and can range from 0,97 - 1,22 % (Lievore *et al.*, 2015) or 3,2 to 4,35 mg/g (Menchik *et al.*, 2019), while the acid pathway generates values lower than 0,48 % (Smith *et al.*, 2016), 0,55 % (Rocha-Mendoza *et al.*, 2021), and 0,94 % (Shelke *et al.*, 2022). When fractionation and purification processes were applied to acid whey, obtaining values of 10,3 – 10,5 % by nanofiltration, and 17, 65 % by ultrafiltration (Wronkowska *et al.*, 2018; Merkel, Voropaeva *et al.*, 2021). The obtained data (**Table 3**) are superior to those recorded in the literature. Two key factors are considered to report high protein content: deficiencies in the coagulation process and the non-application of heat treatments to post-draining whey. The quality control graphs (*X-graphs*) of protein (**Figure 1**) present a process under the control limits, but the behavior of the data indicates a variability with respect to the mean. Therefore, it is possible to infer a migration of casein solids to whey in different proportions. Companies do not apply thermal methods to preserve whey, this can also explain the high protein content because high temperatures cause protein denaturation that can generate a detriment in its content (Smith *et al.*, 2016). The results show acid whey with a good protein content and can be considered a high nutritional source in food products.

Lactose is the major component of acid whey in each company (**Table 3**). The statistical analysis determined differences between the data (p = 0,001), each company handles milk with different percentages of lactose; the origin of the milk is not the same for all. They have different suppliers but try to maintain the same origin of the raw material because there were no differences between sampling dates within each company ($p > 0,05$). The average lactose content was 3,63 % ± 0,17 and can range from 3, 35 to 3, 92 %, these values are below the standard (4,5 %) and those reported in Rocha-Mendoza *et al.* (2021) of 4,90 %, Shelke *et al.* (2022) of 4,37 %, Merkel, Fárová, *et al.* (2021) of 12, 4 %. Nano and ultrafiltration techniques yield among 70 %, 63,51 % and 67 % lactose (Smith *et al.*, 2016; Merkel, Voropaeva *et al.*, 2021). The quality control graph (*X-graphs*) shown in **Figure 1** shows a high variability in lactose content despite 78 % of companies obtain acid whey with contents within the control limits.

The lack of homogeneity and dispersion with respect to the mean of the data is attributed to the lack of management and control to preserve whey, which favors the development of lactic acid. Menchik *et al.* (2019) reported values of 1,9 to 2,1 % of lactose and indicated that whey with these contents can be used in value-added products such as fermented and sports drinks, snacks and infant formulas, and other processes such as obtaining biogas, biodegradable packaging, and metabolites of industrial and pharmaceutical interest.

Regarding the fat content, statistical differences were evidenced between the samples ($p < 0,05$). This may be due to the actions of total or partial skimming of milk and the draining method. Within each company, the same conditions are maintained at least in the skimming stage. There were no differences between sampling dates ($p = 0,778$). The whey recorded a content of 0,11 – 0,76 % and on average 0,43 % ± 0,19, values much higher than those reported by Smith *et al.* (2016) of 0,012 %, Simonis *et al.* (2019) of 0,026 %, Rocha-Mendoza *et al.* (2021) is 0,04 %, contents of 0,51 % have also been reported (Shelke *et al.*, 2022). The quality control graphs (*X-graphs*) of fat content exhibit variable and scattered behavior versus the average of the data, indicating that the skimming and draining operations are not handled in the same way in all companies and can increase the fat content in whey. According to the literature, acid whey presents a characteristic of interest given that, when used as food ingredient, it would provide a low content of triglycerides, but a significant source of functional phospholipids (Rocha-Mendoza *et al.*, 2021).

Each company produces whey with different ash contents ($p < 0,05$), but it is the same ($p= 0,533$) between dates for the same company. Whey can record a mineral content between 0,57 and 0,58 % and on average 0,56 % ± 0,004. These values are higher than those presented by Shelke *et al.* (2022) of 0,43 %, and lower than those set by the standard (0,8 %) and those found in Lievore *et al.* (2015) of 0,83 %; Menchik *et al.* (2019), 0,75 %; Rocha-Mendoza *et al.* (2021), 0,8 %; and Merkel, Voropaeva, *et al.* (2021), 0,67 %. Higher values of 5,89 % and 5,96 % have been reported when treating acid whey by nano and ultrafiltration (Wronkowska *et al.*, 2018).

The quality control graph (*X-graphs*) of the ash content (**Figure 1**) shows an out-of-control process, deducing that the mineral content is very variable, and a range cannot be determined. This may be due to multiple factors from genetics to feeding of cattle, as well as to the doble crema cheese making process. Along with proteins, the mineral content is one of the most important characteristics of whey for industrial use since it can contain up to 80 % of milk minerals. The results obtained (**Table 3**) indicate that acid whey can contribute a good mineral content to the products.

Regarding the calcium content, ANOVA showed differences among companies ($p < 0,05$) but not among sampling dates within each company ($p = 0,252$). This whey can record a calcium content of 122 – 125 mg/100 g and on average 123 ± 0,8 mg/100 g. Menchik *et al.* (2019) presented similar results of 120 mg/100 g, and Rocha-Mendoza *et al.* (2021) of 120 – 128 mg/100 g. The quality control graphs (*X-graphs*) of calcium content (**Figure 1**) exhibit an out-of-control process by inferring that it is highly variable, and a given range cannot be determined. Like the ash content, there are several situations that can condition the presence of calcium in the acid whey, but the form of coagulation of the milk, obtaining the curd and the control of the pH, are those that most favor the formation of calcium dihydrogen phosphate. The lower the pH, the greater the amount of soluble calcium (Smith *et al.*, 2016; Merkel, Voropaeva, *et al.*, 2021) and the lower separation by clarification generating problems in the equipment, a negative aspect that mitigates exploitation actions (Merkel, Voropaeva, *et al.*, 2021; Rocha-Mendoza *et al.*, 2021). In this context, the direct use of acid whey is recommended, for instance, to manufacture dairy drinks to benefit from the nutritional contribution of the calcium content and other components.

Regarding the acid content expressed as percentage of lactic acid (%), the ANOVA showed statistical differences ($p < 0,05$) between companies and between sampling dates. It indicates that each unit performs the making process of doble crema cheese differently, and the management and conservation of whey do not favor the conversion to lactic acid, which can vary from 0,13 to 0,84 %. On average they can record 0,49 % ± 0,2, the values obtained are between those presented by Smith *et al.* (2016) of 0,72 % and Skryplonek *et al.* (2019) of 0,49 %. The quality control graphs (*X-graphs*) of **Figure 1** present a stability pattern given that all the points are within the control limits, but very far from the central axis. They also have a very wide range, generating values outside of those set in Resolution 2310: 1986 (Ministerio de Salud, 1986) and supporting the arguments that the factors may favor the formation of lactic acid after the draining process of cheese in each company. It indicates that the acidity increases during the storage of the whey and is an indicator of bacterial growth (Smith *et al.*, 2016).

The pH values indicate that this is acid whey (**Table 3**). They recorded significant differences ($p = 0,019$) among companies, but not between sample dates for each company ($p > 0,05$). This whey can record values from 4,3 to 5,6 and on average 5 ± 0,36, close and similar to those presented by Smith *et al.* (2016) of 4,58; Skryplonek *et al.* (2019), 4,85; Simonis *et al.* (2019), 4,54; and Merkel, Voropaeva, *et al.* (2021), 4,58. The quality control graphs (*X-graphs*) of pH (**Figure 1**) present points outside the control limits. Additionally, there is a greater dispersion and a very wide range, indicating that the content of organic acids is variable as a result of the non-standardized coagulation of milk, since some companies use lactic or acetic acid and/or acid milk. The pH can decrease by the natural degradation of lactose to lactic acid in the absence of conservation methods.

Conclusion

The statistical analysis of the comparison of methods indicates that the results given by the Lactoscan SP Milkotronic ultrasonic milk analysis is a reliable alternative comparable with the reference methods; therefore, the proposed research hypothesis was accepted.

It is imperative to know the composition of whey to project its use in the industry. Therefore, it should be known as a co-product, instead of a by-product, to be used in different sectors. The significant differences in the studied parameters among acid whey samples is due to the way in each company produces the doble crema cheese. However, there are similarities in the manufacturing process. This is relevant to know the quality of the whey generated by this group of companies and ensure that it remains constant over time, thus allowing the valuation and decision-making on possible applications of acid whey.

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