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Physicochemical Characteristics of Milk By-Products

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The physicochemical characteristics of milk by-product should be assessed by dairy industry to evaluate the potential of different types of wheys to become ingredients. The aim of this study was to evaluate the physicochemical characteristics of the main dairy industry residues: cheese whey, ricotta whey, and butter whey. Whole milk was also evaluated as reference. Analyses of proximate composition, determination of Ca^{2+}+Mg^{2+}, pH, titratable acidity, water activity, and instrumental color, all in triplicate, were performed. The results of proximate composition, and titratable acidity showed significant increases (moisture and titratable acidity) and reductions (ashes, lipids, proteins, carbohydrates, energy value, pH, and Ca^{2+}+Mg^{2+}) between the different types of wheys and milk. The lipid and caloric reduction are considered relevant to dairy industry, mainly to be used in products with high level of fat harmful to health. There was no significant difference between the values for water activity. Furthermore, the color showed difference in which the milk was the whitest sample. Principal component analysis explained 94.40% of the total variance of the data. PC1 separated butter whey and ricotta whey from milk and cheese whey. In view of the results, many physicochemical attributes of the different dairy by-products studied were interesting, therefore the application of these types of wheys in the development of other products, partially or even entirely, can be suggested. Despite the similarity of butter whey to milk, the indication of the most appropriate whey to be used will depend on the product and the aim of its application.

1. Introduction

The conversion of milk into other products (cheese, ricotta, butter, etc.) generates a large amount of by-products that are eliminated by discharging into streams, without or with prior treatment in many degrees. Treatment is rarely enough to prevent severe and serious environmental impact. The cheese whey was classified as 150 times more polluting than ordinary sewage (The lancet, 1922). The damage is mainly due to the pollution caused by both the large amount of types of wheys generated and its chemical properties (high organic matter content, i.e. high chemical (COD) and biochemistry (BOD) oxygen demand). Lactose and lactic acid are the main sources of pollution due to their high oxygen absorption capacity. The aquatic biota, more precisely fishes, are suffocated by the water in which the types of wheys quickly remove the dissolved oxygen (Rivas et al., 2010).
The solutions presented so far, however, are unfortunately not so clear. The use of whey as animal feed, while feasible in theory and to some extent in practice, seems to be limited by commercial reasons. Previous storage of whey in tanks prior to disposal causes the fermentation of lactose, and the production of lactic acid, butyric acid and other organic acids, almost as detrimental as the lactose. Lately, many studies are being developed using lactose by-products in the production of alcohol (bio-ethanol). Reports of whey treatment on land showed that the soil had become "sick" (putrefaction). Evidence of biological methods of treatment by addition of specific bacteria (including pig manure) has yet to show satisfactory effects (The lancet, 1922).
Although there is little information on the composition of the different lactic types of wheys, they are known to have bioactive substances to human health and low fat presence in general. More specifically, cheese wheys have proteins and peptides of high biological value regulating various physiological functions in human body

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such as blood pressure, response glycemia, inflammatory processes, and stimulation of immune system (Athira et al., 2015). In addition, ricotta whey has high lactose content (Sansonetti et al., 2009), and butter whey has a little less lactose and presence of phospholipids that play an important role in many metabolic processes in human organisms, including, according to Kuchta-Noctor (2016), in vitro anticancer properties.

Among the alternatives of reutilization that minimize environmental aggression, the application of different dairy types of wheys in human feeding seems quite feasible. However, the development of novel food products proves to be increasingly challenging as consumers' expectations for these products are both palatable and healthy. At the same time, the industries' economy would be affected by the reduction of raw material costs (Sansonetti et al, 2009). In this context, given the scarce information and a better understanding of its nutritional and technological potential in terms of reuse, the aim of the present study was to evaluate the physicochemical properties of cheese whey, ricotta whey, and butter whey.

2. Materials and methods

2.1 Whole milk and milk by-products acquisition

The by-products were obtained from whole cow's milk by rennet cheese (addition and action of 7% rennet (HA-LA®) and heating at 45±1ºC), ricotta (addition and action of 2% acetic acid (maratá®) with heating at 95±1ºC), and butter (action of centrifugation at 25±2ºC) production of an agro-industry located at Federal Institute of Alagoas (Satuba/AL/Brazil) (Figure 1). The frozen samples were sent in thermal boxes, from up to 12 hours, to the Food Technology Laboratory of the School of Pharmacy at Federal University of Bahia (Salvador/BA/Brazil).

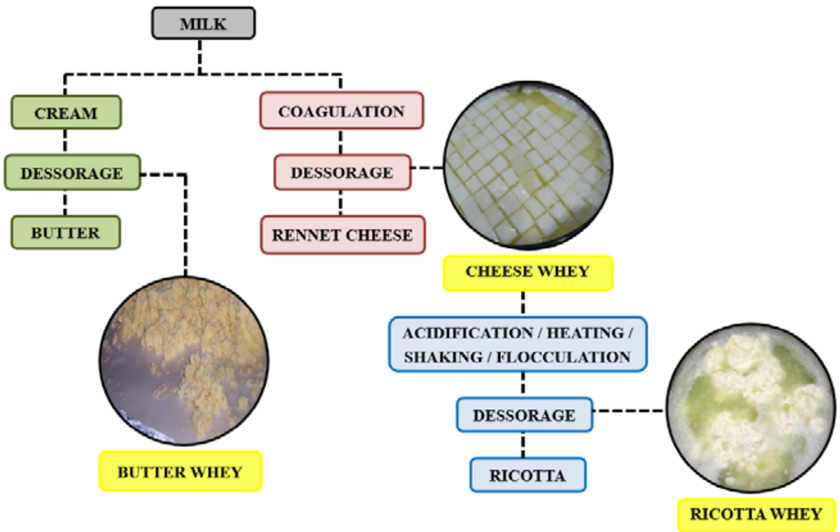

Figure 1: Milk by-products acquisition.

2.2 Proximate composition determination

Moisture, ashes, dietary fibers, and proteins were determined by using the methods of the Association of Official Analytical Chemists (AOAC, 2012). The lipid fraction was determined by the Bligh and Dyer method (Bligh and Dyer, 1959). Additionally, total carbohydrate (Nifext fraction) was calculated by the difference of the measured values for moisture, ashes, dietary fibers, proteins, and lipids. The energetic content was determined based on the Atwater coefficient of proteins, lipids, and carbohydrates as 4 $kcal.g^{-1}$, 9 $kcal.g^{-1}$, and 4 $kcal.g^{-1}$, respectively (FAO, 2003).

2.3 pH and titratable acidity

The pH was measured directly using a digital pH-meter (model DM-23 Digimed; Digicrom Analítica Ltda. - Brazil) previously calibrated with pH 4.0 and 7.0 buffer solutions. The titratable acidity was measured by titration with 0.1 N NaOH and results were expressed as % lactic acid, according to the AOAC methods (AOAC, 2012).

2.4 Water activity

The water activity (Aw) was obtained by direct measurement in an Aqua-Lab digital thermohygrometer (model AquaLab Lite; Decagon Devices Inc. - USA) at a controlled temperature of 25 ± 2ºC.

2.5 Calcium (Ca) and Magnesium (Mg)

The Calcium (Ca) and Magnesium (Mg) determination was done by titration with EDTA (Ethylenediamine Tetraacetic Acid) solution according to methodology described by Bird et al. (1961).

2.6 Instrumental color

The color of the samples was determined by instrumental measurement using a Minolta colorimeter (Chroma Meter, CR-400 - Japan), where measurements were expressed in L* (transition from black 0 to white 100), a* (transition from green -a* to red +a*), and b* (transition from blue -b* to yellow +b*). The colorimeter was calibrated with illuminant D65 and the standard observer was 2°.

2.7 Statistical analysis

All analyses were performed in triplicate and the statistical analyses were performed using the software Statistica 7.0 (StatSoft - USA). One-way analysis of variance (One-way Anova) and Tukey HSD test, at a significance level of 0.05, were used to define the difference between each whey and milk. Principal Component Analysis (PCA) was performed with all obtained data to overview the parameters that were affected by the different types of wheys and milk.

3. Result and discussion

3.1 Proximate composition determination

The nutritional profile is presented in Table 1. Higher moisture values were found for milk processing effluents in comparison to whole milk. This characteristic can explain the lower content found in the other nutrients (ashes, lipids, proteins, carbohydrates) and energetic value, where all of them were diluted. Moreover, butter whey showed protein, lipid, and ash levels more similar to milk than the other types of wheys. There is little information on the composition of dairy effluents, nevertheless the results of the present study were considerably close to those found by other research that also characterized different types of wheys, such as the ricotta whey (Bald et al., 2014, cheese whey (Bald et al., 2014) and butter whey (Morin et al., 2006).
Composition range of the different types of wheys in relation to milk was as follow: increased the moisture (7.74% (butter whey), 10.29% (cheese whey), and 11.40% (ricotta whey)) and reduced ash (8.42% (butter whey), 49.47% (ricotta whey), and 51.58% (cheese whey)), lipids (81.59% (butter whey), 91.11% (cheese whey), and 99.36% (ricotta whey)), proteins (7.32% (butter whey), 77.13% (cheese whey), and 84.45% (ricotta whey)), carbohydrates (36.65% (cheese whey), 42.40% (ricotta whey), and 47.12% (butter whey)), and energy content (53.37% (butter whey), 65.09% (cheese whey), and 72.17% (ricotta whey)) (Table 1). None of the samples had fibers in their composition. Moreover, the most significant change was in the lipid fraction. Another interesting observation was the reduction of the energy value, which is considered advantageous for providing health benefits (WHO, 2003).

Table 1: Proximate composition, energetic content, water activity, pH, titratable acidity, instrumental color, and Ca^{2+}+Mg^{2+} of by-products and milk.

Observed Parameters	Ricotta Whey	Butter Whey	Cheese Whey	Whole Milk
Moisture (%)	94.47±0.31^{a}	91.36±0.51^{c}	93.53±0.46^{b}	84.80±0.55^{d}
Ashes (%)	0.48±0.03^{c}	0.87±0.02^{b}	0.46±0.01^{c}	0.95±0.04^{a}
Lipids (%)	0.02±0.00^{d}	0.58±0.10^{b}	0.28±0.03^{c}	3.15±0.21^{a}
Proteins (%)	0.51±0.04^{d}	3.04±0.00^{b}	0.76±0.01^{c}	3.28±0.00^{a}
Carbohydrates (%)	4.51±0.17^{c}	4.14±0.11^{d}	4.96±0.27^{b}	7.83±0.32^{a}
Energetic content (kcal/100g)	20.26±1.36^{d}	33.94±2.38^{b}	25.41±1.75^{c}	72.79±1.44^{a}
Water activity (Aw)	0.70±0.01^{a}	0.70±0.00^{a}	0.70±0.00^{a}	0.71±0.01^{a}
pH	4.41±0.07^{d}	4.86±0.09^{c}	6.36±0.05^{b}	6.62±0.06^{a}
Titratable acidity (in lactic acid)	0.52±0.01^{a}	0.39±0.08^{b}	0.21±0.01^{c}	0.19±0.01^{c}
L*	31.29±0.90^{d}	81.49±0.71^{b}	41.37±0.83^{c}	86.34±0.42^{a}
a*	-11.55±0.25^{c}	-7.02±0.12^{a}	-9.48±0.25^{b}	-7.25±0.17^{a}
b*	2.15±0.58^{c}	20.43±1.13^{a}	11.67±1.09^{b}	21.67±0.18^{a}
Ca^{2+}+Mg^{2+} (mg/100mL)	28.11 ± 3.42^{d}	99.57 ± 3.01^{b}	43.34 ± 2.03^{c}	124.17 ± 5.37^{a}

3.2 pH and titratable acidity

According to the results presented in Table 1, the cheese whey presented a pH close to the milk, while the ricotta whey and the butter whey presented a much different value. Regarding titratable acidity, the cheese whey presented a value similar to milk whereas ricotta whey showed the highest one. On the other hand, the values of titratable acidity followed contrary behavior observed for pH. The variation of pH can be explained by the existence of several acidic compounds in whey like lactic acid, fatty acids, etc. Some studies have showed that the average pH values of bovine whey can range from 6.03 to 6.71 (Bald et al., 2014; Macwan et al., 2016), which corroborates the results found in the present study. Naturally, due to its production process, ricotta whey's pH was more acidic (mean of 4.41) compared to butter whey, cheese whey, and milk. This pH was also more acidic than the average presented by Bald et al. (2014) with a value of 5.44 when analyzing ricotta whey. This variation might be associated with the different types of cheese whey used in the production of ricotta, implying different pHs in their wheys.

Taking into consideration the average found in this study (4.8), the butter whey analyzed by Antunes et al. (2012) obtained a pH of 4.4. However, both values are in accordance with the U.S. Food and Drug Administration (FDA), which determines a pH ranging from 4.4 to 4.8. In the present study, the result of pH for whole milk was close to neutrality, in agreement to what is found the literature for milk (Walstra et al., 2006).

3.3 Water activity

As seen in Table 1, although the different milk by-products have more moisture than milk, there was no significant difference for the water activity (Aw) between them, indicating that the increase occurred for the contents of bound water and not for free water (available). This is a remarkable achievement because it is clear the importance of water activity in food quality, especially regarding the representation of water availability for the development of microorganisms and the occurrence of spoilage reactions (Rahman and Labuza, 2007).

3.4 Calcium (Ca) and Magnesium (Mg)

Table 1 shows the Ca^{2+}+Mg^{2+} values of milk and its residues. The milk had the highest concentration of Ca^{2+}+Mg^{2+} followed by butter whey, cheese whey, and ricotta whey, respectively. Considering the obtaining process of the evaluated dairy types of wheys, butter whey, cheese whey and ricotta whey have reduced ashes contents, including Ca^{2+}+Mg^{2+}, compared to whole milk (Morin, Pouliot & Jiménez-Flores, 2006; Bald et al., 2014; Qiu et al., 2015; Cortellino & Rizzolo, 2018).

Franzoi et al. (2018) (123.35 mg/100mL), and Martino et al. (2001) (102.00 mg/100mL) found similar results to what is displayed herein for Ca^{2+}+Mg^{2+} in cow's milk. Taking into account the different influences on food composition, Jensen et al. (2012) analyzed the composition of milk from different cow breeds: Jersey (158,59 mg/100mL) and Holstein-Friesian (122.55 mg/100mL). Besides that, Haug et al. (2015) indicate that the milk composition of cows varies in different regions of Norway: north (137.00 mg/100ml), south (133.90 mg/100ml), east (131.90 mg/100ml), and west (132.70 mg/100mL).

The value for cheese whey of the present research was lower, but similar to what was found by Martino et al. (2001) (54.80 mg/100mL). In contrast, Franzoi et al. (2018) found a much lower content (28.87 mg/100mL). It is worth to remember that this variation occurs naturally and, in addition to the factors mentioned above, it is mainly related to the type of cheese and/or cheese processing from which the whey originates.

3.5 Instrumental color

For instrumental color, the lightness refers to the proximity of white or black. As observed in Table 1, the milk was the lightest one (highest value of L*) when compared to the others, followed by butter, cheese, and ricotta wheys, respectively. The white color of milk results from the presence of colloidal particles, such as milk fat globules and casein micelles, capable of scattering light in the visible spectrum (García-Pérez et al., 2005), which explains the high L* value of the milk in contrast with the other types of wheys.

Furthermore, it was observed lower greenness (a*) and yellowness (b*) value of the cheese whey and ricotta whey comparing with butter whey and whole milk (which did not have significant difference between them). This fact is linked to lipid fraction (Table 1) in the same order of types of wheys (Qiu et al., 2015).

Considering that color is one of the most important quality attributes that influences consumer's choices and is directly linked to the appearance of dairy products (Aidoo et al., 2017), ricotta whey and cheese whey may affect the color of the product which they can be added.

The literature does not present instrumental color data for dairy by-products, however, other characterization studies of pasteurized whole milk presented similar performance: Schaffer et al. (1992) with L* (86.10), a* (-2.10), and b* (7.80) and Bonacina et al. (2017) with L* (mean 89.37), a* (mean -7.76), and b* (mean 19.79).

3.6 Principal component analysis (PCA)

PCA explained 94.40% of the total variance of the data (Figure 2), in which the first component (PC1) predominated and contributed with a greater percentage of variance explanation (73.50%) than the second component (PC2) (20.90%). PCA separated all types of wheys and milk based on physicochemical parameters (proximal composition, pH, titratable acidity, Aw, instrumental color, and Ca^{2+}+Mg^{2+}). PC1 separated butter whey and ricotta whey (one group) from milk and cheese whey (another group). The cheese whey characteristics are more similar to the whole milk than butter and ricotta wheys. PC2 separated butter whey, ricotta whey, and milk (one group) from cheese whey (another group). Butter whey and ricotta whey can be identified by greater redness and titratable acidity. The cheese whey by greater Ca^{2+}+Mg^{2+}, yellowness, pH, and lightness, and the whole milk by greater amount of minerals, lipids, carbohydrates, proteins, energy value, and water activity.

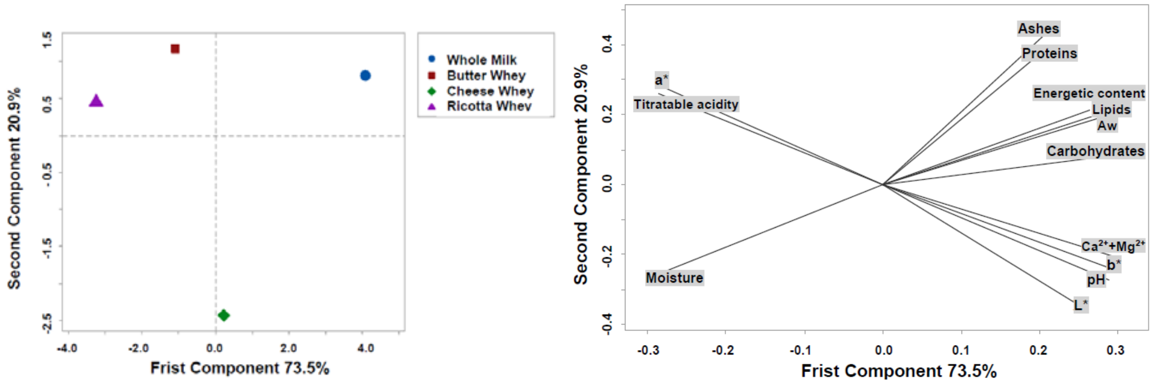

Figure 2: Physicochemical and instrumental data of different types of *wheys and whole milk in the plane defined by the first two principal components*.

4. Conclusions

This study investigated the main physicochemical properties of cheese whey, ricotta whey, and butter whey. In physicochemical aspects, the different types of wheys had a remarkable reduction of the lipid fraction and energetic content. Although there was a difference in moisture, the water activity did not vary among samples. The pH and titratable acidity of cheese whey were closer to milk than the other types of wheys. Moreover, the instrumental color allowed to identify that the milk was the whitest and opaquest sample followed by butter whey, cheese whey, and ricotta whey.

Thus, many physicochemical parameters of the different dairy types of wheys studied are interesting and the use of cheese whey, ricotta whey, and butter whey showed to be potential ingredients in food products. The butter whey presented the most similar characteristics to the milk, however, the choice of the most appropriate whey to be used in food depends on the product that the whey will be added and the aim of its application. Anyway, the reuse of these dairy by-products can be characterized as an alternative in the reduction of environmental impact with socioeconomic importance, aiming at minimizing pollution and costs. However, further experiments should be conducted in order to better understand the technological potential of the application of these by-products in human food, including possible sensory changes.

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