

## **Effect of milking time on key physicochemical parameters and fatty acids profile of milk, cream and butter**

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## Abstract

The transformation of cow milk to butter and cream is one of the many possibilities for diversifying dairy products. In this context, the aim of this study is to contribute in evaluating the effect of milking time on the physicochemical characteristics and fatty acids profile of milk, cream and butter. The study of physicochemical composition showed that milk contained an average of 12.3 % dry matter, 3.4 % of fat and 3.3 % of total nitrogenous matter. The pH and acidity were 6.66 and 16.01°D. For butter, the average contents of water (15.2 %), fat (83.1 %) and non-fat dry matter (1.75 %) comply with the requirements of international standards and Belgian regulations. Analysis of the other parameters of the butter revealed an average color of 28.95 (expressed on the yellow scale), a low oxidation rate of the order of 0.0005 mEq.  $O_2.g^{-1}$  and a texture which correlates positively with the depth of the butter clods. The mean pH value is 4.51. The comparison between evening and morning milking showed that the difference in composition of milks remains not significant ( $p > 0.05$ ). So it is for the composition and parameters of the butter, except for the color which is significantly more yellow for the evening butters (29.7) compared to those of morning (26.2).

**Keywords:** Butter, milk, cow, quality, fatty acids, milking time.

## Effet du temps des traites sur les paramètres physicochimiques clés et le profil en acide gras du lait, de la crème et du beurre

### Résumé

La transformation du lait en beurre est l'une des voies de diversification des produits laitiers. Dans ce contexte, cette étude propose d'évaluer l'influence des traites du soir et du matin sur le processing et la composition physicochimique et le profil en acides gras du lait, de la crème et du beurre. L'étude de la composition physicochimique montre que le lait analysé contient en moyenne 12,3% de la matière sèche, 3,4% de la matière grasse et 3,3% de la matière azotée totale. Le pH et l'acidité représentent respectivement des valeurs de 6,66 et 16,01°D. Pour le beurre, la moyenne de la teneur en eau (15,2%), matière grasse (83,1%) et l'extrait sec dégraissé (1,75%) concorde avec les normes internationales. L'analyse des autres paramètres du beurre révèle une couleur moyenne de 28,9 (exprimée sur l'échelle b\*), une faible oxydation d'une moyenne de 0,0005 mEq O<sub>2</sub>.g<sup>-1</sup> et une texture qui corrèle positivement avec la profondeur du beurre. La valeur moyenne du pH est de 4,51. La comparaison entre le lait des traites du soir et du matin ne montre aucune différence ( $P > 0,005$ ) significative pour les différents paramètres analysés. La même remarque a été observée pour les composés du beurre à l'exception de la couleur qui a été significativement plus jaune (29,7) pour les beurres du soir comparativement pour les beurres du matin (26,2).

**Mots clés :** Beurre, lait, vache, qualité, acides gras, période de traite.

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## دراسة تأثير فترة الحلب على الخواص الفيزيوكيميائية والأحماض الدهنية للحليب، القشدة والزبدة

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### ملخص

يعد تحويل الحليب إلى الزبدة إحدى طرق تنويع منتجات الألبان. في هذا السياق، تقترح هذه الدراسة تقييم تأثير فترة الحلب في المساء والصباح على المعالجة والتركيب الكيميائي والفيزيائي ونسبة الأحماض الدهنية للحليب والقشدة والزبدة. أظهرت دراسة التركيب الفيزيائي والكيميائي أن الحليب الذي تم تحليله يحتوي في المتوسط على 12.3٪ من المادة الجافة و 3.4٪ من الدهون و 3.3٪ من إجمالي المادة النيتروجينية. كما يمثل الرقم الهيدروجيني والحموضة قيم 6.66 و 16.01 درجة، على التوالي. بالنسبة للزبدة، يتوافق متوسط محتوى الماء (15.2٪) والدهون (83.1٪) والمواد الصلبة منزوعة الدسم (1.75٪) مع المعايير الدولية. كما كشف تحليل المكونات الأخرى للزبدة عن متوسط لون 28.9 (معبّرًا عنه على مقياس \* b)، وأكسدة ضعيفة بمتوسط 0.0005 mEq O<sub>2</sub>.g<sup>-1</sup> وصلابة ترتبط بشكل إيجابي مع سمك الزبدة. أما متوسط قيمة الأس الهيدروجيني فقد قدرت ب 4.51. وفي الأخير لم تظهر المقارنة بين حلب المساء والحلب الصباحي أي فرق احصائي ( $P < 0.005$ ) للمعلومات المختلفة التي تم تحليلها في إطار هاته الدراسة. في حين لوحظت نفس الملاحظة بالنسبة لمركبات الزبدة ماعدا اللون الذي كان أكثر اصفراراً (29.7) لزبدة المساء مقارنة بالزبدة الصباحية (26.2).

**كلمات مفتاحية:** زبدة، حليب، بقر، جودة، أحماض دهنية، فترة الحلب.

## Introduction

The Walloon Region (Belgium) is an area rich in traditions and farm products. Nowadays, many dairy cattle farmers continue to produce butter as well as cream, cheeses, yogurts, ice cream, etc. These products, with a typical taste, are sold either directly to the farm, or to merchants and restaurants, as well as markets. The Walloon Region has recognized the importance of this diversification and, since 2002, supported initiatives to help farmers to process milk within their own exploitation.

More accurate and precise estimation of processing abilities would allow better supervision of farmers and will improve processing yields and valorization of milk at farm scale and in short circuits. The quality profile in cow milk is influenced by diet, with variations predominantly caused by differing amounts of fresh forage and concentrates eaten (Croissant et al., 2007; Coppa et al., 2013; Schwendel, 2015). Other factors reported to influence the physicochemical characteristics of milk include differences within and between breed (Soyeurt et al., 2008; Maurice-Van Eijndhoven et al., 2011), season (Heck et al., 2009), climate (Kamleh et al., 2010), stage of lactation (Craninx et al., 2008; Noutfia et al., 2014), and management (Fall et al., 2008). However, no specific study was dedicated to evaluate the effect of milking time on key quality parameters of raw milk and dairy product developed at farm/traditional scale.

The aim of the present investigation is to assess the key quality parameters and fatty acids profile of milk, cream and butter based on milking time.

## Material and methods

### Sampling

The milk used in making butter comes from the complete milking (evening or morning) of a cow. In total, 19 samples of milk, from 19 different cows, have been processed in butter. For each sample, 16 kg of raw milk were skimmed. The cream obtained is divided into two bowls each containing 800 g of cream, in order to be processed into two portions of butter.

### Butter manufacturing process

The method of butter manufacturing was developed based on processing methods reported by different authors: (Angers, 2002; Bakirci et al., 2002; Boutonnier, 2007; Couvreur et al., 2006; Lamothe et al., 2008). The main steps of the processing method are summarized in Figure1.

16 kg of row milk were skimmed using a fat separator (Elecram – Type 125 – France). From each cream obtained, two batches of butter were processed (2\*800 g of cream). The cream was churned using a KitchenAid apparatus (KitchenAid Artisan – USA).

<b>Raw milk</b>
Mixing
Heating (39 °C)
Skimming
Pasteurization of cream (82 °C-20 s)
Cooling (25 °C)
Inoculation with lactic strains (0.375 ‰)
Biological maturation
Physical maturation
Churning
Draining
Washing of butter grains
Kneading
<b>Butter</b>
Conservation at + 5 °C

**Figure 1:** Process of manufacturing butter at laboratory scale.

### Repeatability of manufacturing process

The study of the repeatability and level of control of the process adopted for butter manufacture on the laboratory scale covered the following aspects:

- Quantification of drained buttermilk after churning;
- Determination of churning time;
- Determination and comparison of the quantities of produced butter.

### Physicochemical analyses of milk and butter

Physicochemical composition of milk and butter was determined, as well as butter and cream yields. The milk pH, acidity, total solids (TS), fat (F) and solids not fat (SNF) contents were determined according to (AFNOR, 1993). The butter pH, TS, F and SNF contents were determined according to AFNOR-1995. In addition, peroxide value (as described by Bakirci et al., 2002) and texture (as described by Couvreur et al., 2006) using a texturometer TA-XT2 (Stable micro system, Surrey, England) with a probe of 10 mm diameter (with a penetration force of 25 kg) were determined.

The color of milk, cream and butter was measured in triplicate using a spectrophotometer Hunterlab Miniscan XE (Version 3.5, Reston, VA, USA). The results were expressed using the CIE Lab color parameters:  $L^*$  (lightness value),  $a^*$  (green-red),  $b^*$  (blue-yellow).

These analyses are supplemented by the results of spectral analysis of milk using infrared (MIR) from Battice by the Milk Committee. This allowed to determine some parameters of milk like fatty acids composition, lactose and crude protein (CP) contents...

Then, this study was focused on the comparison of the physicochemical composition of samples of milk, cream and butter from the morning and evening milking.

All measurements were done in two replicates.

### **Statistical analysis of results**

The time of milking was treated as factor whereas all other parameters analyzed were treated as variables. Initially, a determination of mean, standard error of the mean (SEM), minimum and maximum of each analyzed parameter for milk, cream and butter, was performed.

Then, the significance of differences between morning and evening milking were studied through an analysis of variance (ANOVA-1) by determining  $p$ -value. Thus, for  $\alpha$  (the risk of error) equal to 0.05, a difference between two parameters is significant if  $p < 0.05$  and not significant if  $p > 0.05$ . Data from this work have been treated by the software Excel (Version 2007, Microsoft, USA).

## Results and discussion

### Repeatability of the butter manufacturing process at the laboratory scale

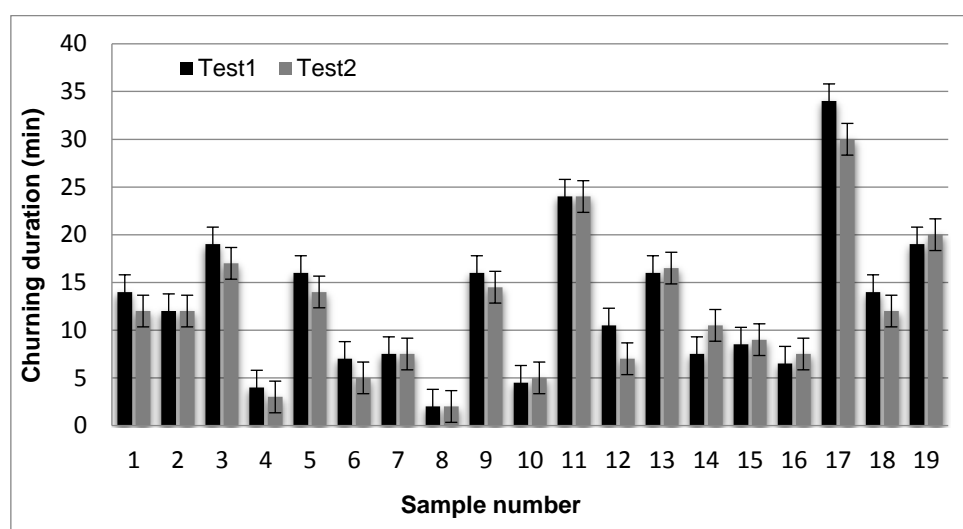
#### *Quantification of drained buttermilk after churning*

The average quantity of buttermilk obtained after churning is 393.5 g. The difference between the quantities of buttermilk discharged between test 1 and 2 does not exceed 31 g.

The statistical analysis of the results shows that this difference is not significant ( $p > 0.05$ ). Consequently, the step of draining buttermilk has been well carried out.

#### *Determination of churning time*

The churning time of the various butter samples is shown in figure 2.



**Figure 2:** Duration of churning of laboratory-made butter samples.

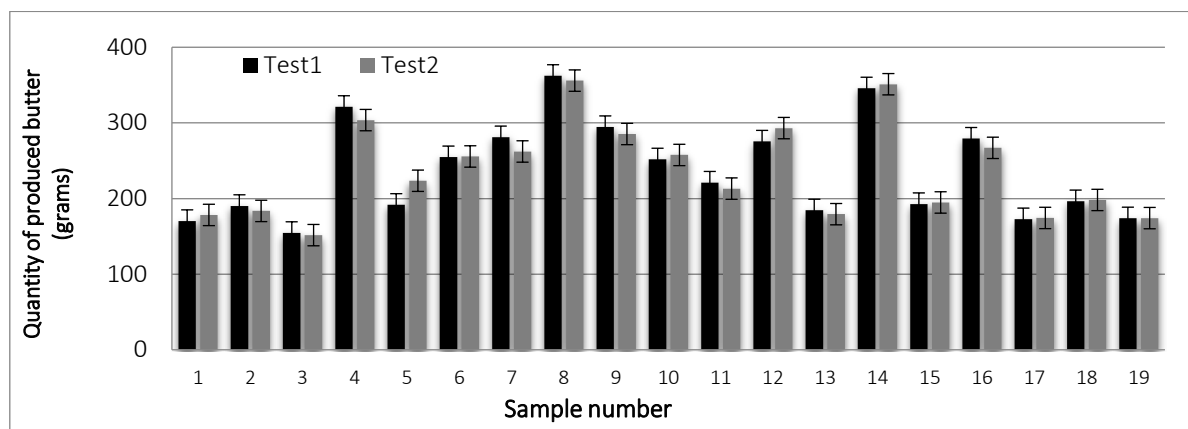
In light of data in this graph, the churning duration varies considerably from one sample to another with a maximum of 34 min and a minimum of 2 min.

However, the average churning durations for test 1 and 2 were 12 min 42 s and 12 min 00 s respectively. The comparison of these two means showed that there was no significant difference ( $p > 0.05$ ) in the churning time between the two tests.



### **Determination and comparison of the quantities of produced butter**

The butter quantities produced from the different milk samples are shown in figure 3.



**Figure 3:** Quantity of produced butter at laboratory scale.

Results presented in figure 3 show significant differences between butter quantities produced from the different milk samples. These differences could be due to the richness of milk samples in fat and fatty acids. The duration of churning, feeding and the individual effect may have an influence on the quantities of produced butter.

The average difference between butter quantities made from milk samples of test 1 and 2 is 0.6 g. This non-significant difference ( $p > 0.05$ ) shows that the production of butter in the laboratory was carried out in a repeatable manner.

### **Physicochemical characterization of milks and butters**

#### **Milk analysis**

The physicochemical characteristics of milk used in the manufacture of butter are given in table 1.

**Table 1.** Physicochemical characteristics of milk (n=19).

Parameter	Mean	SEM	Min	Max
pH	6.66	0.01	6.56	6.77
Acidity (°D)	16.0	0.3	13.0	18.7
DM (%)	12.3	0.2	10.9	14.4
Fat (%)	3.4	0.2	2.3	5.2
SNF (%)	8.9	0.1	8.2	10.5
TNM (%)	3.3	0.1	2.7	4.1
Free fatty acids (%)	1.84	0.26	0.38	4.47
Saturated fatty acids (%)	2.62	0.12	1.91	3.86
Mono-unsaturated fatty acids (%)	1.08	0.08	0.63	2.14
Poly-unsaturated fatty acids (%)	0.16	0.01	0.10	0.22
Short chain fatty acids (%)	0.37	0.02	0.27	0.51
Medium chain fatty acids (%)	1.89	0.10	1.35	2.88
Long chain fatty acids (%)	1.57	0.12	0.96	3.15
Color:				
L*	92.28	0.23	89.67	93.70
a*	-1.51	0.15	-3.00	-0.67
b*	10.70	0.27	8.26	13.07

DM: Dry Matter; SNF: Solids Non Fat; TNM: Total Nitrogenous Matter.

The physicochemical parameters of milk used in butter production (pH and Dornic acidity) are 6.66 and 16.0 °D respectively (Table 1). These results are in accordance with those reported in the literature (Fox et al., 2000; Tsioulpas et al., 2007).

The analyzed milk contains, on average, 12.3 % of DM, 8.9 % fat-free solids, 3.3 % TNM and 3.4 % of fat. The DM content is slightly lower than the mean values of 12.5 % and 13.2 % respectively reported by Amiot et al. (2002) and Courtet Leymarios (2010). Several factors may explain this difference, including diet, breed, season and stage of lactation. (Le Jaouen, 2004).

For fat, the rate of 3.4 % is within the range of 3.3 % and 4.7 % given by Fox et al. (2000) for cow's milk. These authors report a fraction of 3.4 % for TNM, which presents a deviation of 0.15 % compared to the average result of 3.3 % found in this study. Another study shows that the content of TNM can vary between 2.9 % and 5.0 % (Amiot et al., 2002).

In addition, the percentages of saturated and unsaturated fatty acids were 68.2 % and 31.8 %, respectively. These are consistent with the study conducted by Boutonnier (2006). The distribution of these fatty acids according to the chain length gives contents

of 0.37 % for short chain fatty acids, 1.89 % for medium chain fatty acids and 1.57 % for long chain fatty acids.

The color of the milk has an average value of 10.7 (expressed on b\* scale), with maximum and minimum values of 13.1 and 8.26. This variation can be due to the richness of the different milk samples with beta-carotene component. (Casalis et al., 1972).

### Butter analysis

The various components of the laboratory-produced butters are shown in table 2.

**Table 2.** Quality characteristics of butter (n=19).

Parameter	Mean	SEM	Min	Max
pH	4.51	0.03	4.18	4.86
Water content (%)	15.2	0.3	12.1	22.4
Fat (%)	83.1	0.3	75.0	86.3
SNF (%)	1.8	0.0	1.2	2.6
Butter yield (%)	3.8	0.2	2.4	6.1
Yield factor (%)	1.1	0.0	0.9	1.8
Peroxide Value (mEq O <sub>2</sub> .g <sup>-1</sup> )	0.0005	0.0000	0.0000	0.0011
Color:				
L*	88.96	0.37	84.68	93.79
a*	4.71	0.27	1.56	8.22
b*	28.95	0.92	18.71	40.11
Texture (N) :				
At 5 mm	3.9	0.2	2.2	7.3
At 10 mm	9.3	0.5	4.7	18.8
At 18 mm	20.2	1.1	10.4	40.6

The produced butter has an average pH of  $4.51 \pm 0.16$  (Table2). This value is slightly lower than those reported by Sagdiç et al. (2004) and Samet-Bali et al. (2009), with pH values of 4.66 and 4.70 respectively. This slight difference can be explained by the seeding rate of lactic acid bacteria and the duration of biological and physical maturations.

The mean water and fat contents of the produced butter are 15.2 % and 83.1 % respectively (Table 2). On the one hand, these average contents comply with the requirements of Belgian legislation, which imposes a maximum percentage of 16 % for water and a minimum of 80 % for fat content (Arrêté royal, 1988). On the other hand, the mean levels of water and fat contents of butter are comparable with the study conducted by Koczon et al. (2008), who have a content of 15.8 % and 82.9 % for water and fat contents respectively. Similarly, these mean results are included in the intervals

reported by Hermida et al. (2001). These intervals range from 13.0 to 17.0 % for water content and from 81.1 % to 85.5 % for fat. The butter yield is 3.8 % on average with maximum and minimum values of 6.1 % and 2.4 % (Table 2). In general, this yield should be around 4.8 % (Boutonnier and Dunant, 1985).

The relatively low average yield obtained in this study can be attributed to the use of individual milks and non-standardized creams with fat. This chemical component (fat) plays an important role in improving butter yield and churning efficiency.

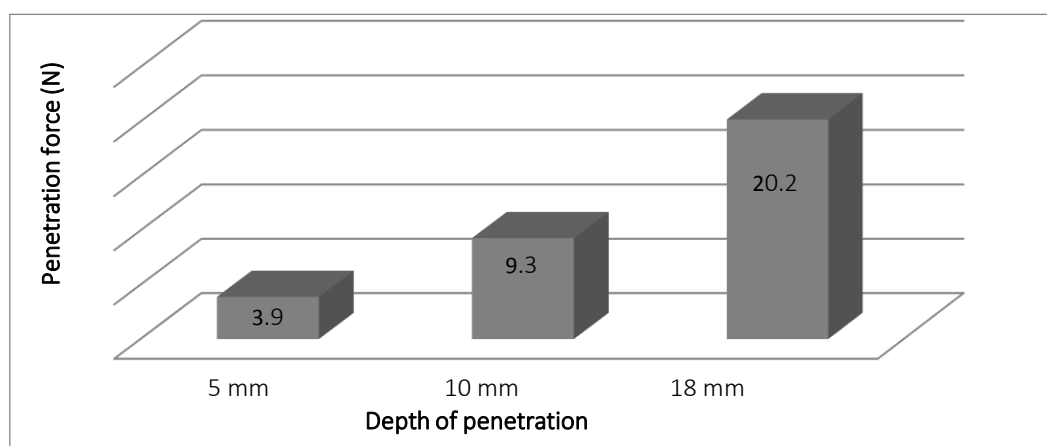
Other elements related to the manufacturing process such as skimming temperature, duration and temperature of physical maturation can contribute to the improvement of this yield.

The peroxide value which is an indicator for oxidation, expressed in mEq O<sub>2</sub>/g, has an overall average of 0.0005 for the various butter samples (Table 2). This value is lower than the data in the literature. The reported values are respectively: 0.42 to 0.46 for Simsek, (2011), 0.0012 to 0.0017 for Bakirci et al. (2002) and 0.00078 for Ozturk et al. (2006).

The rate of oxidation of butter produced in the laboratory is very limited. This can be justified by the freshness of milk and cream used in processing. In addition, the milk is skimmed 2 to 3 hours after milking and the cream obtained does not undergo any temporary storage before its transformation into butter.

The color of butter (expressed on b\* scale) has an average value of 28.95 and maximum and minimum values of 40.11 and 18.71 (Table 2). This reflects the great variability between the different samples analyzed. This could be attributed to the fat richness in beta-carotene. According to the literature, saturation of butter in yellow color fluctuates between 21.1 and 22.1 (Simsek, 2011).

The appreciation of the texture of butter permits to observe a markedly increasing evolution of the penetration force with the depth. Figure 4 shows the penetration force (Newton) at three different locations of the clods of butter. This positive evolution is in agreement with the findings of Couvreur et al. (2006).



**Figure 4:** Evolution of the texture of butter according to depth.

## Comparison between morning and evening fabrications

### Composition of milk

The average contents of the morning and evening milks used in the manufacture of butter are reported in table 3.

**Table 3.** Physicochemical composition of morning and evening milks (n=19).

Parameter	Morning	Evening	P-value
pH	6.65 <sup>a</sup>	6.68 <sup>a</sup>	0.23
Acidity (°D)	15.7 <sup>a</sup>	16.4 <sup>a</sup>	0.35
DM (%)	12.1 <sup>a</sup>	12.5 <sup>a</sup>	0.39
Fat (%)	3.1 <sup>a</sup>	3.8 <sup>a</sup>	0.07
SNF (%)	9.0 <sup>a</sup>	8.7 <sup>a</sup>	0.23
TNM (%)	3.3 <sup>a</sup>	3.3 <sup>a</sup>	0.97
Free fatty acids (%)	1.54 <sup>a</sup>	2.13 <sup>a</sup>	0.36
Saturated fatty acids (%)	2.53 <sup>a</sup>	2.74 <sup>a</sup>	0.42
Mono-unsaturated fatty acids (%)	1.12 <sup>a</sup>	1.36 <sup>a</sup>	0.22
Poly-unsaturated fatty acids (%)	0.37 <sup>a</sup>	0.38 <sup>a</sup>	0.24
Short chain fatty acids (%)	1.83 <sup>a</sup>	1.97 <sup>a</sup>	0.69
Medium chain fatty acids (%)	1.42 <sup>a</sup>	1.73 <sup>a</sup>	0.52
Color :			
L*	92.3 <sup>a</sup>	92.1 <sup>a</sup>	-
a*	-1.86 <sup>a</sup>	-1.08 <sup>a</sup>	-
b*	10.33 <sup>a</sup>	10.82 <sup>a</sup>	0.36

Means values with superscript <sup>a</sup> are statically non different ( $P>0.05$ )

In general, data presented in table 4 indicate that the physicochemical composition of evening milks seems to be richer than in the morning. The major differences are found for fat (3.8 % vs 3.1 %) and free fatty acids (2.13 % vs 1.54 %). On the other hand, very small differences were noticed for TNM, short-chain fatty acids and milk color. These differences in the composition of the evening and morning milks may be justified by effects due to feed intake, race and randomness in the choice of cows (individual effect). However, any difference between the composition of evening and morning milks remains significant. These findings correlated with the study by Rémond et al. (2010), which indicated that butter, protein and lactose levels were very similar between evening and morning treatments.

### ***Physicochemical characteristics of creams obtained from evening and morning milks***

Analyses of cream were focused on determination of pH and color (b\*). Table 4 presents the results of these analyses.

**Table 4.** Physicochemical characteristics of morning and evening creams (n=19).

	Morning	Evening	P-value
Color (b*)	12.6 <sup>a</sup>	13.8 <sup>a</sup>	0.21
pH	6.62 <sup>a</sup>	6.70 <sup>a</sup>	0.02

*Means values with superscript <sup>a</sup> are statically non different (P>0.05)*

According to table 4, a difference of 0.08 is observed between the pH of creams of the evening and the morning. This significant difference ( $p < 0.05$ ) can be attributed to the relatively high pH of evening milk (6.70) compared to morning milk (6.62). In addition, skimming can increase the concentration of the fatty fraction (already high in evening milk), characterized by a low acidity, of the cream resulting a decrease in its acidity.

For color, no difference was observed in color b\* scale between creams obtained from morning and evening milks ( $P > 0.05$ ). On the other hand, the skimming yields for evening and morning milks vary with an insignificant manner between 12.6 and 13.8 respectively.

### ***Physicochemical characteristics of butters obtained from evening and morning milks***

The differences observed between butter samples produced during the morning and evening transformations are shown in table 5.

**Table 5.** Physicochemical characteristics of morning and evening butters (n=19).

Parameter	Morning	Evening	P-value
pH	4.49 <sup>a</sup>	4.51 <sup>a</sup>	0.78
Water content (%)	15.6 <sup>a</sup>	14.8 <sup>a</sup>	0.19
Fat (%)	82.6 <sup>a</sup>	83.5 <sup>a</sup>	0.18
Butter yield (%)	3.6 <sup>a</sup>	4.1 <sup>a</sup>	0.14
Peroxide Value (mEq O <sub>2</sub> .g <sup>-1</sup> )	0.0005 <sup>a</sup>	0.0005 <sup>a</sup>	
Color :			
L*	90.4 <sup>a</sup>	87.9 <sup>b</sup>	0.0004
a*	3.90 <sup>a</sup>	4.90 <sup>b</sup>	0.03
b*	26.2 <sup>a</sup>	29.7 <sup>b</sup>	0.03
Texture (N):			
At 5 mm	4.0 <sup>a</sup>	3.5 <sup>a</sup>	0.26
At 10 mm	9.6 <sup>a</sup>	8.4 <sup>a</sup>	0.32
At 18 mm	20.5 <sup>a</sup>	18.1 <sup>a</sup>	0.28

Means values with superscript <sup>a</sup> and <sup>b</sup> are significantly different ( $P < 0.05$ )

According to the results in Table 5, with the exception of color, no significant difference was recorded for the physicochemical parameters of morning and evening butter. Indeed, evening butter has a darker yellow color than morning butter with mean values (expressed on the b\* scale) of 29.7 and 26.2 respectively.

This difference in color can be mainly attributed to the composition of the feed ration given to cows selected for evening milking compared to those ones selected for morning milking.

The literature shows that butters from milk treated from cows fed with corn silages are less colorful than butters from grass silages which are themselves less colorful than those from grazed grass. The butters are more yellow with a ration based on hay compared to a ration based on corn silage because the grass dried under good conditions does not lose all its carotenes. Also, it was reported that the color of the milk product can be indicator of physico-chemical changes and also an indicator of consumer's preferences (Watson et al., 1934; Hurtaud et al., 2007 Milovanovic et al., 2020)

## CONCLUSION

This work is focusing on several aspects related to the manufacture and analysis of milk and butter composition.

Thus, a manufacturing process for butter has been developed on a laboratory scale. This process has been studied.

The physicochemical composition of milk has been determined and is consistent with the literature.

The processing of milk permits the production of butter with an average yield lower than those reported by the literature. Moreover, yield fluctuates greatly from one sample to another. This finding can be attributed mainly to the use of individual non-standardized in fat milks. Improving manufacturing parameters can positively influence the yield of butter.

Concerning butter physicochemical composition, the average results are in concordance with the literature and with the requirements of Belgian legislation.

The analysis of the qualitative parameters of butter revealed a mean color superior to those reported in literature, a texture that correlates positively with the depth of butter clods and a low peroxide value. There was no significant difference in the physicochemical composition of milks in the evening or in the morning. So it was for the composition of butters made from these milks with the exception of the color which is significantly more yellow for evening butters compared to morning butters.

This difference in color can be attributed to the composition of the feed ration given to cows selected for evening milking compared to those selected for morning milking.

This work lays the foundation for future studies and also opens an important debate on the time of milking on technological criteria to be considered when choosing the collecting milk time. More studies are necessary to fully elucidate the effect of milking time and processing on the nutritional quality of milk and processed products.

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