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ASSESSMENT OF CHEMICAL AND SENSORY QUALITY OF UNSALTED AND SALTED SWEET CREAM BUTTER DURING STORAGE AT DIFFERENT TEMPERATURES AND TIME

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ABSTRACT. Quality of butter depends on many factors such as quality of raw material, production method, ingredients used, type of packaging. Chemical changes taking place during storage of final product are also important. Extent of oxidation and the amount of free fatty acids in Estonian butter have not been investigated recently in experimental studies and have been evaluated at national level only with regard to intervention buying-in. The purpose of this work was to evaluate the quality of salted and unsalted sweet cream butter produced in continuous butter machine and stored at different storage temperatures and time periods. Three batches of salted and unsalted sweet cream butter were prepared and were stored at three different temperatures: at –20 °C for 24 weeks, at +5 °C for 12 weeks, at +20 °C for 8 weeks. Dry matter and salt content, peroxide value, acid value and organoleptic properties were evaluated. No major differences were found when comparing acid values and peroxide values at different storage temperatures. There were no significant differences between salted and unsalted butter samples and no age trends for the values. At all storage temperatures, the level of acid value (maximum value 0.81 mmol 100 g^{-1} fat) was lower than the upper limit established for high-quality butter (1.2 mmol 100 g^{-1} fat). The peroxide value (maximum value 0.050 meq per kg fat) was also lower than the upper limit established for high-quality butter (0.3 meq kg^{-1} fat) at all storage temperatures. After eight weeks of storage the sensory characteristics of butter – appearance, taste and flavour – scored at least 4 points or higher on 5 point scale that corresponds to high-quality butter.

Introduction

Quality of butter depends on a number of factors such as the quality of raw material; type of processing; ingredients; efficiency of working; distribution of buttermilk, salt, starter culture; packaging; storage conditions, *etc*. Chemical changes taking place during storage of final product are also very important and in some cases may be detrimental. Objective assessment of quality of butter includes chemical, physical and microbiological testing and subjective evaluation means the sensory assessment of organoleptic characteristics of butter. There are specific requirements for labelling fat spreads according to type of fat and fat content, but there is no official standard or a set of chemical or sensory requirements for high-quality butter in Estonia or EU. The only quality requirements applied for butter are applicable to intervention buying-in. EU Regulation No. 1272/2009 states the following requirements for butter: fat content not less than 82%; butter contains only milk fat; water content up to 16%; solids non-fat up to 2%, free fatty acid content less than 1.2 mmol per 100 g fat and peroxide value less than 0.3 meq oxygen per kg fat. Butter may not contain coliforms. Appearance, taste, aroma, consistency (the organoleptic characteristics) of the product must be at least four points out of five and water dispersion at least four points.

Salt reduces water activity (a_w) of food and serves as preservative and antimicrobial agent in food. Preservative effect of salt is caused by salt-induced growth of osmotic pressure. As a result of the increase in osmotic pressure the cells of microorganisms become dehydrated to such an extent that vital processes in protoplasm are inhibited or interrupted (Mariutti, Bragagnolo, 2017). Salt contents of butter and plasma vary a number of times because the salt dissolves in water and, therefore, remains in plasma phase of butter (Patel, 2016).

Quality of the butter depends on the content of free fatty acids of the butterfat and it varies widely depending on the extent of hydrolysis of the triglycerides. The number of free fatty acids can increase significantly as the result of lipolysis. In addition to natural lipase, milk can also contain the lipase of microbial origin. Hydrolysis of milk fat produces free fatty acids from triglycerides, free fatty acids, in turn, are oxidized to aldehydes, ketones (Henno, 2005; Fearon, 2011). Short-chain fatty acids, containing from 4 to 12 carbon atoms, often cause rancid and impure taste. Rancid and impure taste can be perceived by a trained expert if the free fatty acid content is 1.2 to 1.5 mmol per 100 g fat and by the consumer at a concentration of 2.0 to 2.2 mmol per 100 g fat (Henno, 2005).

Oxidation of fat is one of the main reactions affecting the quality of fat besides hydrolysis. Milk fat oxidation occurs in several successive stages: unsaturated fatty acids react with oxygen to form peroxides, which determine a series of chain reactions. The final products of the chain of reactions are several volatile substances with specific rancid smell. As a result of oxidation, the off-flavour of fat, cardboard, fish or of metal may occur. Extent of fat oxidation can be estimated by determining peroxide value and acid value of fat: the lower peroxide and acid values, the better the quality of fats (Shahidi, Zhong, 2005; Walstra *et al.*, 2006; Fox, Kelly, 2012).

High-quality sweet cream butter has slightly sweet, clean, pleasant taste and delicate aroma, while the cooked taste is considered a good characteristic. Various off-flavours and tastes as bitter, acid, cheesy, feed, metal, fish off-flavour and taste can be transmitted from raw material, can be formed via chemical processes during storage and as a result of activities by microorganisms present in the product (Lozano *et al*., 2007; Krause *et al*., 2008). Spontaneous oxidation can cause fatty and fish off-flavour and smell, especially during long-term storage, even at low temperatures (–20 °C) (Walstra *et al.*, 2006). Salt that increases the amount of ions in butter plasma and promotes chemical changes in fats, can also cause bitter off-taste. Sweet cream butter should have almost white to light yellow colour. The added salts can cause mottled, marble, striped and uneven colour of butter (Chandan, 2015).

Extent of oxidation and the amount of free fatty acids in Estonian butter has not been investigated recently in experimental studies and have been determined at national level only with regard to intervention buying-in. The purpose of this work was to evaluate the quality of salted and unsalted sweet cream butter at different storage temperatures and time periods characterized by acid value, peroxide value and sensory quality (appearance, taste and aroma).

Materials and Methods

The investigated butter samples were produced by Estonian butter producing company in continuous butter making machine. Six kilograms of fresh butter were taken after passing the working section of butter machine and 2% of commercial salt "Extra" was added to half of the sample. Salt was ground in coffee mill for achieving finer particle sizes and better solubility prior to adding into butter. The salt was sieved into butter and pressed with a spoon until the salt was completely dissolved and evenly distributed in butter. The quality of salt mixing was evaluated visually.

The samples of salted (SB) and unsalted butter (USB) were divided into portions and packed into plastic film and in aluminium foil to prevention of access of air and avoid the impact of light. Three batches of salted and unsalted sweet cream butter were prepared. Butter samples were stored at three different temperatures: at –20 °C for 24 weeks, at +5 °C for 12 weeks, at +20 °C for 8 weeks. The following parameters were determined in butter samples (in total 90 samples): content of dry matter and salt, peroxide value, acid value and organoleptic properties. A plan of analysis (Table 1) describes all analyses carried out at different storage temperatures.

Table 1. Plan of analyses of unsalted butter (USB) and salted butter (SB) samples stored at different temperatures (Teder, 2016)

Week	Storage temperature		
	–20 °C	+5 °C	+20 °C
0		DM, AV, PV, S*	
2			AV, PV
3		AV, PV	
4	AV, PV, SEN	SEN	AV, PV, SEN
6		AV, PV	AV, PV
8	AV, PV, SEN, DM, S	SEN, DM, S	AV, PV, SEN, DM, S
9		AV, PV	
12	AV, PV	AV, PV	
16	AV, PV		
20	AV, PV		
24	AV, PV		

*DM – dry matter, AV – acid value, PV – peroxide value, S – salt content, SEN – sensory analysis

The dry matter was determined using a moisture analyser Kern DBS 60-3 (KERN & Sohn GmbH): 1.5 to 2.0 g of butter was weighed to sample plate and measurement was done at 140 °C for 10 minutes (Kern DBS, 2013). Evaluation of salt content of butter was based on the chloride content. In order to determine the chloride content, the standard methodology for the determination of the chloride content in cheese products (EVS-EN ISO 5943 V2: 2006) was applied. The chloride content was determined from the pre-separated plasma phase. For analysis, approximately 0.5 grams of plasma of SB or 6 to 7 grams of plasma of

USB was weighed to the nearest 1 mg. The determination was continued according to the standard methodology. The content of free fatty acids of butterfat was evaluated by the acid value using the standard method EVS-EN ISO 660:2009. Oxidation was assessed according to ISO 3976:2006 via peroxides formed during storage of butter.

Sensory evaluation was done by panel of 27 general consumers aged 20 to 61 years on week 4 and week 8 for each batch of USB and SB stored under different temperature conditions. After samples were kept at +5 °C for 12 hours and at room temperature for at least 10 minutes before analysis, evaluation carried out as a blank test. Sample size was approximately 7 g and brown bread and/or water were used to neutralize taste in mouth. The appearance, taste and aroma were evaluated. Characteristics were evaluated in 5-point system: 1 – strong defects, 2 – weak defects, 3 – satisfactory, 4 – good, 5 – very good, no defects.

The statistical analysis of the data was performed with Microsoft Excel 2013. Student t-tests were used to compare the mean values at different time points, significant effects were declared at $P < 0.05$.

Results and Discussion

The similarity of the three different batches of butter was evaluated by dry matter content and salt content. The average dry matter content of the three batches of USB was 84.06% and 85.91% in the case of SB. The dry matter content of SB was about 2% higher than of USB due to 2% of added salt. The salt content of butter samples was evaluated according to their sodium chloride (NaCl) content. The amount of NaCl found in the USB was 0.152% (due to natural NaCl in the milk). The average salt content of 10.72% in the plasma phase of butter corresponds to 2% salt content of butter. Both the dry matter content and salt content of all three batches were similar ($P > 0.05$) and thus, the batches did not significantly influence the parameters studied in the work.

The acid value indicates the amount of free fatty acids in the milk fat. In current study, the acid value of USB and UB were determined immediately after the butter was made and later after certain intervals (Table 1). The acid value of butter at different storage temperatures remained between 0.71 and 0.81 mmol 100 g^{-1} fat during storage period (Fig. 1). A statistically significant temporal trend was observed only for butter stored at +5 °C, the acid value of which increased at the end of the shelf life. No significant differences were observed in the levels of free fatty acids of USB and SB (except of week 12 and week 16 of butter stored at –20 °C). At all storage temperatures, the acid value of both USB and SB samples remained lower (maximum value 0.81 mmol 100 g^{-1} fat at +5 °C on week 12 and at +20 °C on week 4) than the limit 1.2 mmol 100 g^{-1} fat set for high-quality butter (EU Regulation No 1272/2009).

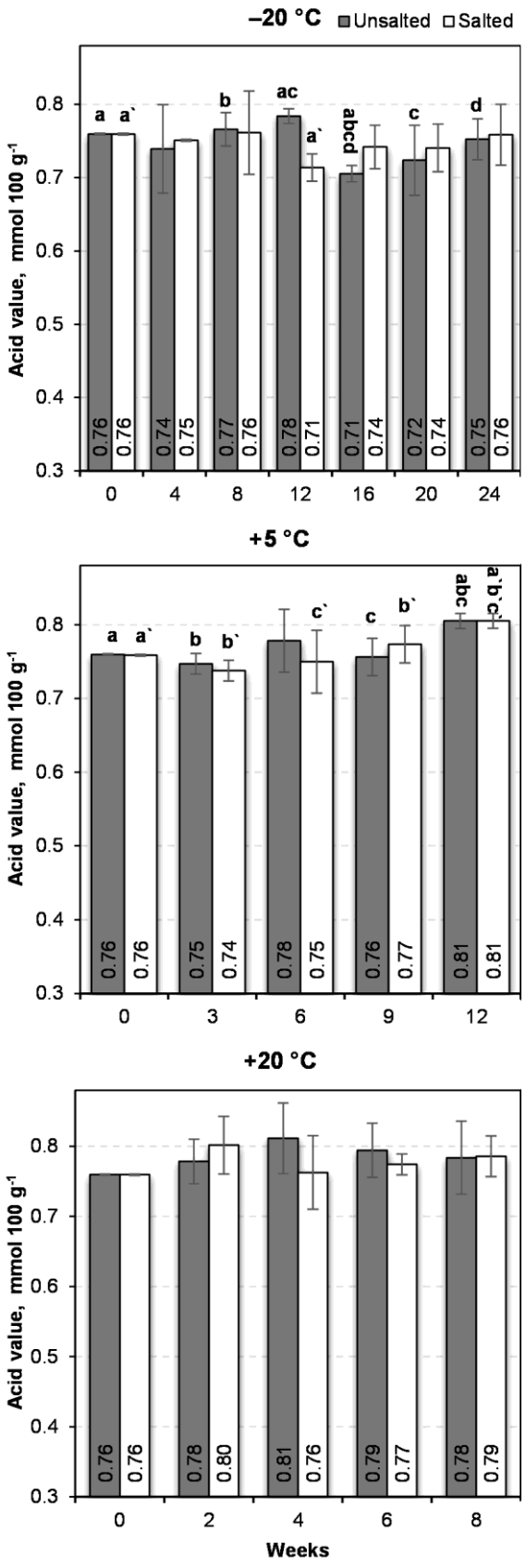

Figure 1. Acid values of unsalted and salted butter stored at –20; +5; +20 °C. The mean values with different letters (a, b, c, d) within unsalted butter and (a', b', c') within salted butter are significantly different ($P < 0.05$) (Teder, 2016)

In addition to the original plan of analyses (Table 1), the acid value was also determined after six months of storage. The average levels of acid value of three batches of USB and SB stored in a refrigerator at +5 °C for six months were 0.80 and 0.79 mmol 100 g^{-1} fat, respectively. The average acid value of butter stored at + 20 °C for six months was 0.98 mmol 100 g^{-1} fat for USB and 0.99 mmol 100 g^{-1} fat for SB for a high-quality butter, which also remained below the limit of acid value established for a high-quality butter. Differently from the results of this work, Koczon *et al.* (2008) observed a significant increase in the acid value of butter samples stored at +5 °C and +20 °C. In their study, the levels of acid value of the samples stored at +5 °C and +20 °C were 5.21 and 7.35 mmol 100 g^{-1} fat, respectively, which are 5 and 7 times higher than the levels in butter stored at the same temperatures in current work. Koczon *et al.* (2008) observed the traits of hydrolysis of the unsalted sweet cream butter already on the third week of storage at +5 °C and on the second week of storage at +20 °C. In present study, none of the samples exceeded the established maximum limit of acid value (1.2 mmol 100 g^{-1} fat) at any storage temperatures during the six months of storage.

The chemical quality of butter has been evaluated on national level in Estonia only with regard to intervention buying-in. All butter samples (n = 429) that were analysed during national buying-in at a period of 2005 to 2009 met the requirements for free fatty acids (Raie, 2016).

The peroxide value is one of the most important indicators of oil quality and this indicates the amount of hydroperoxides formed in the oils and fats as a result of the oxidation process. Similar to the acid value, the peroxide value of USB and SB was determined immediately from fresh butter and during storage at different temperatures according to the plan of analyses (Table 1). During storage at different temperatures, the peroxide value remained at levels of 0.017 to 0.050 meq kg^{-1} fat (Fig. 2), which is in average almost 10 times lower than the allowed limit for intervention buying-in (0.3 meq kg^{-1} fat, EU Regulation No. 1272/2009). There were no significant changes in peroxide value within 16 weeks of storage at –20 °C; the levels were significantly higher only since week 20. When butter was stored at +5 °C, the peroxide values increased until week 6, after which a statistically significant decrease (Fig. 2) was observed. The reasons of that phenomenon are not clear. The peroxide values of USB and SB samples stored at +20 °C were highest on week 6. There was a statistically significant difference only between peroxide values of USB and SB on week 24 (stored at –20 °C) and week 8 (stored at +20 °C). No significant differences were found between the peroxide values at different storage temperatures. Similarly, no significant differences were observed between USB and SB, and no definite temporal trends in the dynamics of peroxide values. The peroxide values of three batches of unsalted and salted butter stored for six months at +5 °C were 0.033 and 0.047 meq kg^{-1} fat, respectively.

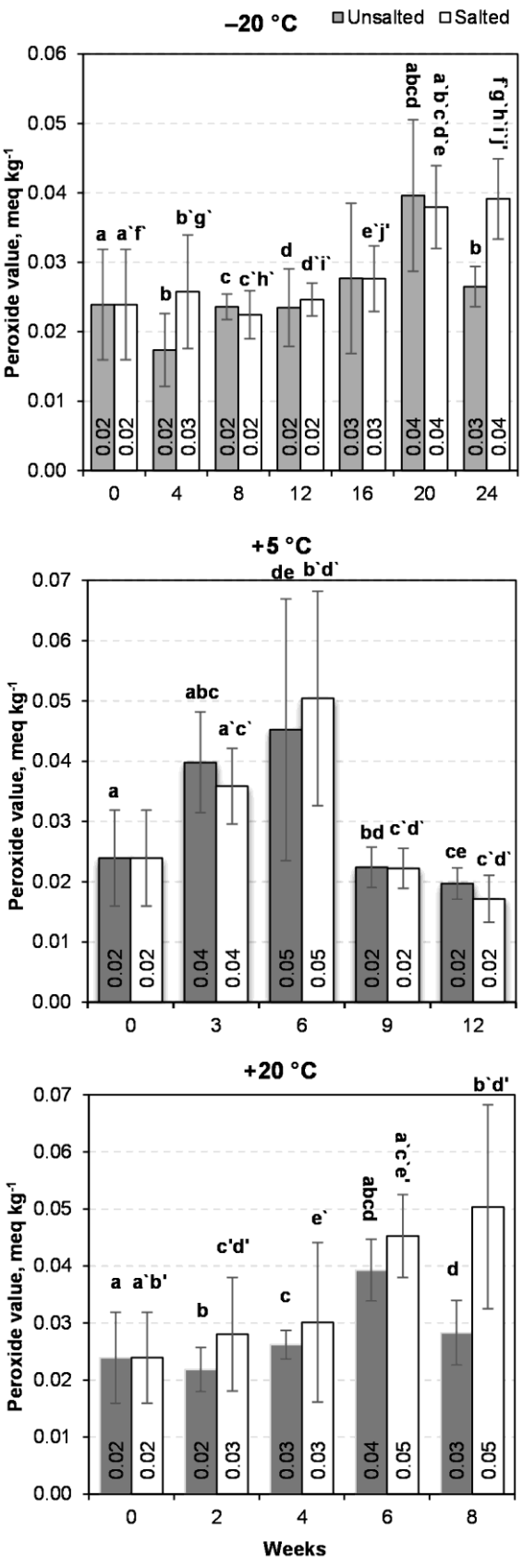

Figure 2. Peroxide values of unsalted and salted butter stored at –20; +5; +20 °C. The mean values with different letters (a, b, c, d, e) within unsalted butter and letters (a', b', c', d', e', f', g', h', i', j') within salted butter are significantly different (P < 0.05) (Teder, 2016)

The storage for the same period at +20 °C resulted in the peroxide value of 0.048 meq kg^{-1} fat for USB and of 0.109 meq kg^{-1} fat for SB. These levels were also lower than the permitted limit established for intervention buying-in.

As the content free fatty acids was low and only free fatty acids can be oxidized, the peroxide value of butter samples remained low. All butter samples (n = 430) that were analysed during national buying-in at a period of 2005 to 2009 met the requirements for peroxide value (Raie, 2016).

Generation of distinguished taste and aroma problems was not observed after storage of USB at three different temperatures for 4 weeks (score of 4.9 points; Fig. 3). Some assessors found unclean and slightly rancid taste in SB after a 4-week storage period, but the highest organoleptic quality, *i.e.* fresh aroma and taste, was attributed to USB and SB samples stored at –20 °C. After 4 weeks of storage, the scores for aroma of USB and SB did not depend statistically significantly (P > 0.05) on storage temperature.

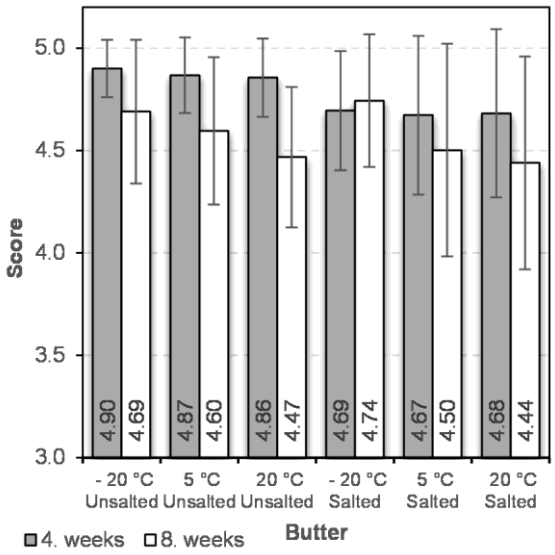

Figure 3. Mean scores for taste and aroma of unsalted butter (USB) and salted butter (SB) stored at –20 °C; +5 °C; +20 °C for four weeks and eight weeks

Sensory analysis of USB after the 8-week period of storage resulted in lower scores for taste and aroma compared to evaluation result after week 4 (Fig. 3) and statistically significant (P > 0.05) differences were between samples stored at –20 °C and +20 °C. USB stored at room temperature gained 4.5 points as assessors described slightly rancid, metallic and bitter taste. After 8-week storage SB kept at +5 °C and +20 °C had lower taste and aroma scores compared to week 4 scores, difference was statistically significant (P > 0.05) at +20 °C stored samples. The scores for taste and aroma of SB stored at –20 °C for 8 weeks were statistically significantly higher than stored at 5 and +20 °C for 8 weeks. Assessors found pronounced mistakes in samples stored in refrigerator and room temperature but not in butter stored in deep freezer. The defects were described as impure, rancid, metallic and bitter tastes.

Assessors found no defects of appearance of USB stored at –20 °C and +5 °C for 4 weeks and 8 weeks (Fig. 4). In warmer storage conditions (+20 °C) some released fat was noticed on the surface of USB after 4 weeks of storage, amount of released fat increased with prolongation of storage. This was a clearly expressed defect but was not reflected in scores as every assessor evaluated small sample of product and could not recognize the defect.

The main defect of SB was non-uniform colour. The main reason of appearance defects is perhaps the uneven distribution of salt in mass. Since during preparation the distribution of salt was assessed visually, it was not possible to fully assure that the salt was uniformly dissolved.

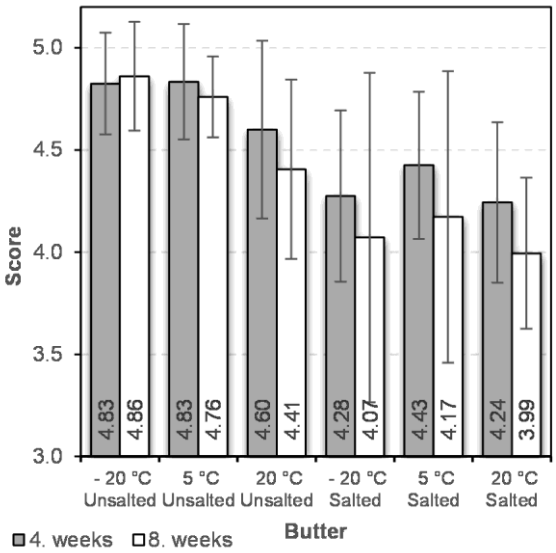

Figure 4. Mean scores for appearance of unsalted butter (USB) and salted butter (SB) stored at –20 °C; +5 °C; +20 °C for four weeks and eight weeks

Good organoleptic properties were confirmed by chemical analyses; both the acid value and the peroxide value were lower than the established maximum limits. Rancid and impure taste can be perceived by a trained expert if free fatty acid content is 1.2 to 1.5 mmol per 100 g fat and by regular consumer at a concentration of 2.0 to 2.2 mmol 100 g^{-1} fat (Henno, 2005). As free fatty acid content was lower than 1.2 mmol per 100 grams of fat, assessors did not perceive unclean and rancid taste caused by excess of free fatty acids. That was reflected in scores higher than 4 points.

Conclusions

The purpose of this work was to evaluate the quality of USB and SB during different storage regimes. For this, three batches of unsalted and salted butter were prepared and stored at 3 different temperatures: –20 °C for 24 weeks, +5 °C for 12 weeks and +20 °C for 8 weeks. Quality of butter was evaluated by determination of acid value, peroxide value and organoleptic parameters.

Based on results and analysis of data collected, the following conclusions can be drawn:

• storage temperature and salt content did not affect significantly the content of free fatty acids or the level of oxidation of sweet cream butter produced in continuous butter machine;

• no certain temporal trends were observed in acid and peroxide values;

• acid value (maximum value 0.81 mmol 100 g^{-1} fat) remained below the established limit for high-quality butter (1.2 mmol 100 g^{-1} fat) during whole observation period at all storage temperatures and salt contents;

• peroxide value (maximum value 0.050 meq kg^{-1} fat) remained below the limit for a high-quality butter (0.3 meq kg^{-1} fat) during whole period of storage at all temperatures and salt contents;

• all butter samples received at least 4 points or higher score for appearance, taste and aroma. This was confirmed by chemical analyses: both the acid value and peroxide value, which remained significantly lower than the limits established in the regulation, proved good organoleptic properties of butter samples;

• butter producers have no need to add salt for achieving better quality during storage. Chemical parameters of salted butter did not differ significantly from the parameters of unsalted butter, but too high salt content may be not favourable or healthy for consumers;

• storage temperature did not have significant impact on chemical characteristics of butter. The storage at room temperature resulted in fat separation.

Conflict of interests

Authors declare that there is no conflict of interest regarding the publication of this paper.

Author contributions

Study conception and design: IJ, LT, KL.
Acquisition, analysis and interpretation of data: LT, IJ, KL.
Drafting, editing and critical revision of the manuscript: KL, IJ, LT

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