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Some Studies on the Effect of Different Storage Temperatures Upon the Fat Constants and Acidity of Print Creamery Butter

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by

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The observation of butter under different storage temperatures is interesting because of its practical bearing upon the factory phase of the dairy industry. Admittedly butter must be stored. It is consumed steadily. It is produced in varying quantities. It is a perishable product. Economic conditions have created the butter store house whereby uneven supplies may be more evenly distributed.

Under all practical storage conditions butter deteriorates. Chemical changes occur, more or less pronounced, injuring the quality and thereby affecting the market value. One of the chief constituents which undergoes noticeable chemical change is the fat, which is over 80% of the weight of ordinary creamery butter. Because of its complexity this constituent presents some difficulty to the analyst, with accompanying diversity of results under almost identical working conditions.

This thesis is a report of work done on the fat from butter under three different storage temperatures. Three lots of one pound prints, five in each lot, were taken from the same churning for use in this experiment. The butter was made from pasteurized and ripened cream, under ordinary creamery conditions. After being packed in a ninety pound Friday press the prints for this experiment were taken at random.

The first lot, designated as lot "A", was stored in a room having a temperature of about 70° F. Of the five prints, A1, A2 and A3, were wrapped in plain parchment and A4 and A5 were wrapped in parchment and placed in commercial butter cartons. The five prints were placed in an open cardboard box on a shelf. Care was taken so that no prints touched each other.

On the day these lots were stored a sample of butter from this churning was reserved for analysis, the results of this analysis being regarded as the initial analysis for each of the fifteen prints.

The second lot, marked B1,B2,B3,B4 and B5, was wrapped exactly as A, the firt three in plain parchment and the last two in parchment and carton. The five B prints were stored in a cardboard box in a room in the refrigerator which was brine cooled and had a temperature of about 45 °F.

Lot C, containing five prints, marked C1,C2,C3, C4 and C5, the first three in parchment and the last two in parchment and carton, was stored in a cardboard box in a refrigerator room at a temperature of 32 °F.

On the day stored each print was weighed to the gram and all whole prints were weighed every three weeks, thereafter for twelve weeks. Analyses of the composition (water,fat,salt,and ash), acidity, and fat constants (Reichert-Meissl, Saponification and Iodine absorption numbers, melting point and refractive index) were made every three weeks on the prints wrapped in parchment only. A half print was used from each lot for an analysis, the remaining half print being rewrapped and used for the next interval. The data from the print weighings present nothing of interest because all the prints were not carried thru.

In determining the total composition of the butter a half print was used in each analysis made. The sample was cut into several pieces and placed in a wide-mouthed, glass stoppered jar, in which it was melted on the steam bath to a milky consistency. The jar was then cooled in running water with constant shaking. From this cooled mass an approximate 5 gram charge was weighed out in duplicate into a tared porcelain evaporating dish. The

dishes were then heated for four hours in a steam jacketed oven at 98° C, weighed and re-weighed at two hour intervals until constant in weight.

The residue from the water extraction was washed with gasoline into a tared Gooch crucible having an asbestos filter thru a Hirsch funnel. The washing was completed with petroleum ether. The crucible was then dried in oven to constant weight. The casein was now burned off in a muffle furnace. The residue plus crucible minus the weight of crucible represented the ash. The residue plus crucible after the fat washing, minus the crucible plus ash represented the casein. The water free butter plus the crucible minus the weight of crucible and its contents after the fat exeraction yields weight of fat in the charge.

Table No.1. Composition of storage butter.

Constituent	Storage Temperature.	Period.				
		Initial %	3 wks. %	6 wks. %	9 wks. %	12 wks. %
Water	70° F.	13.82	12.37	19.55	9.9o	9.18
	45° F.	13.82	12.6o	13.8o	12.45	12.83
	32° F.	13.82	14.4o	13.37	13.8o	12.72
Fat	70° F.	83.21	84.46	87.2o	86.95	87.5o
	45° F.	83.21	84.45	83.o5	84.54	83.85
	32° F.	83.21	82.o7	83.4o	82.7o	84.o5
Casein	70° F.	.79	.83	.95	.94	.93
	45° F.	.79	.79	.84	.79	.8o
	32° F.	.79	.82	.82	.83	.81
Ash	70° F.	2.14	2.31	2.29	2.31	2.42
	45° F.	2.14	2.o8	2.35	2.19	2.63
	32° F.	2.14	2.56	2.4o	2.65	2.4o

In the foregoing table there is a noticeable decrease in the percentage of water in the butter held at 70° F. during the succeeding periods. This lot was outside in the open air which possibly accounts for most of the loss. The lots held in the refrigerator rooms at 45° F. and 32° F. changed but little in composition. The water per cent figure of 14.40% at the end of the first three weeks in the butter stored at 32° F. probably represents a variation that may occur in the water content of ordinary creamery print butter . The results of the ash analysis are not consistent even tho checking duplicates were obtained. Here both the accuracy of the method and experimental error are no doubt at fault.

In the acidity analyses a ten gram charge was weighed into a white casserole and melted on steam bath. 25 Cc each of ether and alcohol were added with stirring. The free acid was neutralized with decinormal sodium hydroxide using phenolphthalein as an indicator. A blank was run on reagents used.

Table No.2 represents the acidity findings on the three lots of butter at the three week intervals of the experiment.

Table No.2. Acidity of storage butter.

Storage Temperature	ccs n/10 alkali to neutralize free acid in 10 grams by periods.				
	Initial.	3 wks.	6 wks.	9 wks.	12 wks.
70° F.	2.55	2.85	4.30	4.40	5.45
45° F.	2.55	2.45	2.60	3.25	3.00
32° F.	2.55	2.50	2.50	2.50	2.70

The development in acidity was very marked in the butter placed at the highest temperature, much less marked in the lots stored 45° F. and 32° F. The lot held at 32° F. gained on the average less in acid than in the case of that held at 45° F.

For the fat analyses, the sample, from which the composition and acidity analyses were made, was heated on a steam bath until the butter separated into layers with the fat in as clear a condition as possible on the top. A hot water jacketed funnel with filter paper was arranged and the fat portion was poured thru, care being taken not to allow water to enter filter with fat. The fat was placed in a cool place over night, securely stoppered. When prepared for any fat analysis the fat sample was liquified by gentle warming to 50-55° C.

According to Richmond when butter is kept and becomes rancid very pronounced changes take place in the composition of the fat. These may be classed under two heads - hydrolysis and oxidation. If butter fat be kept in the dark and out of contact with the air, it keeps indefinitely without change; but in the presence of light and air it becomes oxidized.

The general course of change may be roughly indicated thus.

1. The fat is partly hydrolysed into fatty acids and glycerol.

2. The glycerol is oxidized to fatty acids of low molecular weight.

3. The unsaturated acids are oxidized forming hydroxy-acids.

The genral effect of these changes are

1. The volatile and soluble acids are increased, the soluble in greater proportion than the volatile.

2. The insoluble acids are decreased.

3. The iodine absorption is lowered.

4. The density and refractive index are increased.

5. The potash absorption is increased.

If the butter has been kept in its natural state, the butter fat obtained on melting may have properties differing materially from thos indicated above, owing to the solubility of some of the products in the water still left in the butter. The soluble and volatile acids in the filtered fat may be lowered from this cause, and the insoluble acids increased.

The change is not very rapid, and in the course of several weeks the changes are often not very pronounced.

Bell has recorded the following figures for the changes in the insoluble fatty acids, the butter in this case was kept for the times indicated.

No. of weeks kept.	12	7	7	6	8	6
Before keeping, per cent,	87.3o	87.8o	85.4o	87.4o	87.72	87.65
After keeping, per cent,	88.97	9o.oo	85.72	87.97	88.4o	88.oo

Veith has made analyses showing the change in the insoluble fatty acids produced when butter fat is kept. In each case about a year had elapsed between the two analyses.

	Per cent	Per cent
Original insoluble fatty acids	87.43,88.38,	87.61,87.72,
Insoluble fatty acids after keeping	85.o7,85.97,	84.41,83.82,

It is seen from the figures quoted in these results that the analysis of butter which has been kept for any length of time is a matter of considerable difficulty. Though in butter fat the volatile acids do not show any diminution, but rather an increase (due possibly to the oxidation of the glycerol) in butter the reverse is usually the case. It is by no means improbable that, besides the solubility of these in the water contained in the butter, a portion is destroyed by the action of micro-organisms. The most reliable datum would seem to be the determination of the volatile acids on the butter itself without separation of the fat, and calculation of the Reichert figure on the actual fat present. The potash absorbtion does not appear to undergo much change.

Cornelinson and Rabild, of the Dairy Division, have stored pure butter fat at 0° F. for periods from one to four months during which monthly analyses were made of the fat constants. These investigators found very little chemical change. It was so apparent that no pronounced change could be expected until a longer time had elapsed than is usually practiced in storing butter that this experiment was discontinued.

These samples of nearly pure butter fat showed no physical alterations even after six months; there was no development of any characteristic flavor, whatsoever.

In the present experiment the Reichert-Meissl number was carried out under the standard method described in Leach. The results are shown in Table No.3.

Table No.3. Reichert-Meissl Numbers of storage butter.

Storage Temperature	Period.				
	Initial.	3 wks.	6 wks.	9 wks.	12 wks.
70° F.	28.05	26.8	27.87	26.75	26.87
45° F.	28.05	27.97	27.27	26.5	27.37
32° F.	28.05	27.97	27.2	28.05	26.85

Very little variation in the Reichert-Meissl figure occured in either of the three storage temperatures during the twelve weeks.

The Iodine Absorption number was run according to Hanus' method. Results are presented in Table No.4.

Table No.4. Iodine Absorption Number of storage butter.

Storage Temperature	Period.				
	Initial	3 wks.	6 wks.	9 wks.	12 wks.
70° F.	33.5	30.3	34.7	34.05	34.65
45° F.	33.5	30.75	35.0	35.25	34.5
32° F.	33.5	31.05	35.0	35.4	34.1

The butter stored at 70° F. showed a steady increase in percent of iodine absorbed, indicative of little change in the unsaturated fatty acids. The results of this number are so uniform that little importance can be attached to the results obtained for so short a period.

Table No.5 shows results obtained upon the saponification number. The method used is the standard one described by Leach p486-7.

Table No.5. Saponification Number of storage butter.

Storage Temperature	Period.				
	Initial.	3 wks.	6 wks.	9 wks.	12 wks.
70° F.	249.3	233.95	236.5	234.0	232.4
45° F.	249.3	233.9	239.5	244.0	232.4
32° F.	249.3	232.85	24o.5	239.5	234.7

Very inconsistent results were obtained on this phase of the experiment. Most authorities have found that the saponification number increases with the age of storage butter.

The initial figure seems excessively high. The fact that all three butters analysed the same practically at the end of three weeks and at twelve weeks would indicate that not much change had taken place in the samples.

Allen and Moor have analyzed butter stored for one, two and three years. There was practically no change in the second and third instances. The first year analysis, however, showed a lower figure than the fresh butter.

The melting point determination was made according to Wiley's (official) method. Following is the data obtained.

Table No.6. Melting Point of storage butter.

Storage Temperatures	Period				
	Initial	3 wks.	6 wks.	9 wks.	12 wks.
70° F.	32.7°C.	33.1°C.	33.8°C.	32.1°C.	32.6°C.
45° F.	32.7°C.	32.35°C.	32.4°C.	31.1°C.	30.75°C.
32° F.	32.7°C.	32.2°C.	32.25°C.	31.4°C.	31.6°C.

Each sample shows a slight increase at the end of the sixth week. In the 70° F. butter the mark of 33.8° is 1.2° over the initial. Aside from this one analysis the melting point in general was found to decrease slightly in this experiment.

The refractive index determinations were made on an Abbe' refractometer at a temperature of 40° C. A very marked sameness of results characterized this experiment on the fat from storage butter.

Table No.7. Refractive Index of storage butter.

Storage Temperatures	Period				
	Initial	3 wks.	6 wks.	9 wks.	12 wks.
70° F.	42.6	42.2	42.3	42.5	42.6
45° F.	42.6	42.2	42.5	42.5	42.6
32° F.	42.6	42.2	42.3	42.5	42.5

Conclusions.

1. In composition butter stored outside at 70° F. lost much in water, with a necessary increase in percentages of remaining constituents.

2. The developments of acidity in butter stored at 70° F. in a room is very marked. Cold temperatures retard acidity development.

3. In this experiment there was not enough consistent change in the Reichert-Meissl, Iodine absorption, or Saponification numbers to warrant weighty or definite conclusions as to certain chemical changes.

4. In general the melting point was lowered.

5. The refraction index remained constant to the limits of experimental error.

6. In a space of only twelve weeks it appears that definite change in the fat of storage butter takes place only slowly and from causes emanating, because of their inconsistencies, from the chemical contact which the different butter ingredients give each other.

BIBLIOGRAPHY.

Gray, C.E. ----- Investigations in the Manufacture and Storage of Butter.
U.S.Animal Industry Bur.Bull. 84(1906).

Rahn,O., Brown, C.M., and Smith,L.M., -- Keeping Qualities of Butter.
Michigan Agric.Exper.Sta. Tech.Bull. 2, 1909.

Richmond, H.D., -- Dairy Chemistry, 1914.

Rogers, L.A., --- Keeping Quality of Butter.
U.S.Bureau Animal Industry Bull,57(1904) Especially pp.10-24. Gives additional references on page 24.

Rogers, L.A. and others. --- Factors influencing the Change in Flavor in Storage Butter.
U.S.Animal Industry Bull. 162 (1913).

Sayer, W.S.,------ Keeping Qualities of Butter.
Mich.Agri.Exp. Sta.Tech.Bull. 1.

Larsen,Lund and Miller ---- Factors affecting Acidity of Creamery Butter.
S.D. Agr. Exp.Sta. Bull. No.122, 1910.

Leach ---------- Food Inspection and Analysis. 1911.