

10% cream was sterilized by UHT treatment. The OD of both canned and uncanned UHT 10% cream increased up to the 6th wk of incubation and maintained this OD for up to 30 wk as shown in Fig. 1. However, these increases were not significant in comparison with the increase in the unsterile 10% cream and autoclaved hemoglobin in the same Fig. 1. Moreover, increases of optical densities of sterile 10% cream and hemoglobin were in accord with results of Nakai et al. (4) at high temperatures of storage, although concentrated milk was used in his study. Since there was no microbial contamination, no coagulation, and no gelation in the canned UHT 10% cream during storage at 23 ± 2 C and 32 ± 1 C, the increases of OD of canned and uncanned UHT 10% creams may have been due to chemical decomposition of milk protein producing certain substances absorbing light at $400 \text{ m}\mu$.

Decomposition of protein molecules during storage, in general, may provide an explanation for changes in stability of UHT 10% cream. From Fig. 2 the protein decomposition was not caused by proteases which had survived. No protease activity was in UHT creams stored at 23 ± 2 C under sterile conditions for 22 mo in our laboratories. The decrease of casein-N in UHT cream which occurred at both 23 ± 2 C and 32 ± 1 C was unlikely to be related to survival or reactivation of native protease. Samel et al. (6) reported that casein N content decreased in UHT milk stored at various temperatures, especially at 37 C. They also reported, on the other hand, that increases of nonprotein N and noncasein N were directly related to storage temperature; the higher the storage temperature the greater the increase of nonprotein N and noncasein N. Our results of protein decomposition appear to agree with those of Samel et al. (6) except that the increase of nonprotein N was insignificant in

cream stored at 23 ± 2 C. The decrease of casein N content and increase of noncasein N and nonprotein N appear to be due to heat induced proteolysis in 10% cream stored under sterile conditions.

Acknowledgments

The authors are grateful to D. B. Emmons and D. R. Arnott for their encouragement and valuable discussions. Sincere appreciation is expressed to D. B. Emmons, M. Kalab, and K. M. Shahani for reviewing the manuscript. This work was supported by a grant under the Industrial Research Assistance Program as administered by the National Research Council of Canada.

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Amino Acid Composition of Granules and Spots in Grana Padano Cheeses

A. BIANCHI, G. BERETTA,
G. CASERIO, and G. GIOLITTI
Istituto Ispezione degli
Alimenti di Origine Animale
University of Milan, Italy

Abstract

Amino acids concentrated in Grana Padano cheese in two different physical forms, granules and spots. The major amino acid in the granules was tyrosine

followed in concentration by phenylalanine and glutamic acid.

Composition of spots was predominantly leucine and iso-leucine with tyrosine essentially absent. Composition of the free amino acids in the granules dif-

ferred from that in the whole cheese. Bacterial populations were much higher in amino acid localization than in cheese as a whole, suggesting that bacterial action is a major contributing factor to the phenomenon of amino acid localization.

Introduction

Hard white granules of tyrosine in some fermented foods were first described in 1880, as quoted by Steinegger (17) for Emmenthal cheese. They then were thought to be deposits of salts. In 1926, Mirri (11) described tyrosine granules in country-cured hams. Zeetti (18) and Piettre (12) attempted to determine their origin in cured meats and concluded that they were not produced by microorganisms. Several erratic observations have been published (9, 16) regarding tyrosine granules in cheeses. Ritter and Schilt (14) also detected other white formations with diameters approximately 5mm, called 'flecken' (spots) by the authors; their composition of free amino acids was given.

From the literature two kinds of formations in cheeses were known; crystalline tyrosine granules, and more amorphous granules containing calcium lactate (8). Further investigation seemed desirable to know more of variation in such formations during ripening and of the mechanism of free amino acid production.

The formation of free amino acids has been detected in all types of cheese independent of the consistency and period of ripening, and also in cheese without rind (3). The mechanism of this formation is not fully understood but it is likely (8) that it arises from the poor solubility of some amino acids, probably produced by some bacteria (14). Flükiger and Schilt (7) and Ritter et al. (13) published the amino acid composition of granules and spots without distinguishing between them. Later, Mascherpa and Giolitti (10) reported on compositions of spots and granules in a type of Italian cheese, Grana Padano, at the end of ripening.

We report results of detailed investigations of spots and granules from their first detection up to long ripening in Grana Padano cheese. In physical characteristics granules appear generally as spherical crystals with a 1.0 to 2.0 mm diameter, spots as small areas of white, soft amorphous material with 5.0 to 6.0 mm diameter in the surface of cheese slices. We studied methods of detection, isolation, variations in chemical composition, and genesis of

the amino acids.

Experimental Procedure

Experiments were on 16 Grana Padano cheese cakes which were made from a stock of milk free from antiseptic agents (formaldehyde).

Isolation of granules. Granules were carefully removed from the surface of thin slices of cheese with suitable needles. Weighed granules were ground, dissolved in .1 HCl and deproteinated with sulfosalicylic acid. Centrifugation gave a clear supernatant which was treated with ethyl ether to remove some fat residues and was analyzed on an Amino Analyzer 3A27 C. Erba. The analysis was on granules taken each time from a different cheese cake from 4 to 25 mo old.

Isolation of spots. The analysis as described was also on spots from whole cheeses 18 and 25 mo old. The covering material of spots was removed before analysis. Cheese where neither tyrosine granules nor spots were visible was also analyzed.

Results and Discussion

Amino acid analyses are in Tables 1 and 2. Our main concern was to follow changes in component amino acids during ripening. Variation in composition of granules and spots was considerable. We were able to detect variation in component amino acids in time by amino-analyzer.

A number of amino acids: asparagine, proline, glutamic acid, valine, methionine, isoleucine, tyrosine, phenylalanine, lysine, and histidine, increased steadily during ripening; threonine, alanine, and ornithine showed no change; and α -amino butyric acid, tryptophane, and arginine increased only after 10 to 12 mo of ripening. Tyrosine gave the following values: 48.2 g/100g initially, 82.5 g/100 g after 18 mo, and 70.2 g/100 g after 25 mo. These refer to samples from the inner part of the cheese where humidity was approximately constant over time.

We noticed a nonhomogenous distribution of granules and spots in the cheese; we handled some pieces of cheese containing considerable amounts of granules and spots. Different crystalline forms were found. The granules in the holes of the cheese were spherical, smooth, and peduncolate. Others in the cheese mass were either starlike, bowls, crystalline needles, rough, or polymorphic. We have no satisfactory explanation of this variation.

Received February 11, 1974.

TABLE 1. Free amino acids in the granules and in the whole cheese; composition data.

Amino acids	4 months		6 months		8 months		9 months		10 months		11 months		12 months		13 months		18 months		25 months	
	A ^a	B ^b	A	B	A	B	A	B	A	B	A	B	A	B	A	B	A	B	A	B
Aspartic acid	.11	.12	.09	.12	.13	.17	.11	.18	.07	.14	.14	.17	.15	.22	.22	.21	.11	.28	.11	.26
Threonine	.13	.19	.15	.25	.14	.30	.17	.28	.15	.25	.20	.34	.19	.33	.35	.39	.19	.47	.18	.52
Serine	.13	.13	.13	.17	.11	.23	.16	.23	.13	.20	.21	.28	.19	.29	.24	.38	.23	.55	.21	.51
Asparagine	.32	.70	.34	.74	.18	.95	.42	.82	.38	.75	.44	1.20	.35	1.03	.25	1.17	.57	2.45	.72	2.84
Proline	.29	.64	.32	.74	.38	.89	.52	.76	.42	.68	.56	1.07	.57	1.14	.53	1.30	.45	1.25	.48	1.57
Glutamic acid	.57	.94	.68	1.28	.78	1.60	.76	1.52	.71	1.23	1.10	2.31	1.12	2.14	1.25	2.31	1.02	2.72	1.02	2.88
Citrulline	.09	.20	.11	.16	.11	.39	.20	.36	.12	.27	.17	.35	.18	.32	.22	.37	.11	.56	.14	.51
Glycine	.09	.10	.09	.11	.13	.15	.10	.14	.10	.13	.13	.28	.12	.19	.13	.20	.11	.25	.10	.26
Alanine	.08	.11	.09	.14	.09	.18	.09	.15	.09	.14	.15	.23	.12	.23	.15	.23	.13	.30	.12	.31
α-amino butyr. acid0201020101030301
Valine	.24	.37	.26	.47	.28	.46	.26	.50	.24	.45	.40	.75	.41	.70	.49	.77	.48	.87	.38	.95
Methionine	.12	.16	.11	.18	.12	.21	.14	.20	.14	.17	.18	.25	.16	.32	.24	.28	.34	.32	.21	.35
iso-Leucine	.20	.32	.24	.41	.24	.53	.25	.48	.24	.42	.34	.59	.33	.60	.49	.65	.74	.76	.45	.77
Leucine	.39	.67	.43	.73	.47	.97	.46	.85	.46	.75	.52	.81	.52	.95	.80	.98	1.33	1.03	.73	1.15
Tyrosine	48.20	.18	61.20	.21	47.30	.23	53.00	.20	50.80	.18	54.90	.20	52.20	.21	47.80	.21	82.50	.19	70.20	.23
Phenylalanine	3.28	.36	3.40	.48	2.77	.53	3.03	.45	2.90	.39	4.08	.54	3.35	.53	3.85	.58	8.83	.67	4.60	.72
Ornithine	.07	.07	.07	.13	.06	.09	.05	.06	.10	.11	.06	.13	.08	.17	.06	.05	.04	.16	.03	.09
Lysine	.35	.60	.42	.79	.45	.92	.48	.86	.43	.75	.62	1.15	.61	1.16	.76	1.50	.70	1.80	.52	1.66
Histidine	.07	.12	.15	.25	.09	.24	.14	.24	.16	.24	.14	.19	.13	.13	.19	.34	.15	.37	.12	.41
Tryptophan0304050504050607	.06	.16	.04	.15
Arginine0201010201	.05	.0502

^a Grams of amino acids /100g granules.

^b Grams of amino acids /100g whole cheese (dry weight).

^c Traces.

Composition of spots. Spots in the cheese can be seen only after 1 yr of ripening and only easily in the cheese mass of 16 to 18 mo cheese cake. Composition of spots per 100 g was mainly leucine 9.86 to 7.68 g; iso-leucine 4.96 to 4.13 g; glutamic acid 1.84 to 1.99 g; methionine 1.64 to 1.93 g; valine 1.52 to 1.72 g; and asparagine 1.63 to 1.29 g; which were the minor components of the granules. In the whole cheese the same amino acids per 100 g dry weight were 1.03 to 1.15 g; .76 to .77 g; 2.72 to 2.88 g; .32 to .35 g; .87 to .95 g; and 2.45 to 2.84 g (Tables 1 and 2).

At this stage we are unable to give a satisfactory explanation for later formation of spots and differing component amino acids of spots and granules.

Bacterial analysis. A preliminary topographical analysis of the microflora in different parts of the cheese showed that the number of microorganisms was up to 10 times higher along the boundaries of the tyrosine granules than in the whole cheese. The microorganisms were yeast, streptococci, and lactobacilli.

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TABLE 2. Free amino acid in spots, in whole cheese, and in the cheese without granules and spots; composition data.

	Spots		Whole cheese (dry weight)			Cheese without granules and spots (dry weight)	
	Months		Months			Months	
	18	25	18	25	18	25	
	g/100 g						
Aspartic acid	.21	.19	.28	.26	.35	.32	
Threonine	.37	.34	.47	.52	.62	.62	
Serine	.48	.34	.55	.51	.72	.66	
Asparagine	1.64	1.29	2.45	2.84	2.40	2.62	
Proline	.88	.93	1.25	1.57	1.55	1.86	
Glutamic acid	1.84	1.99	2.72	2.87	3.13	3.60	
Citrulline	.38	.30	.58	.51	.60	.58	
Glycine	.26	.18	.25	.26	.31	.30	
Alanine	.29	.26	.30	.31	.39	.37	
α-amino butyr. Acid ^a	... ^a	... ^a	... ^a	
Valine	1.52	1.72	.87	.95	1.05	1.04	
Methionine	1.64	1.93	.32	.35	.32	.25	
iso-Leucine	4.96	4.13	.76	.77	.78	.74	
Leucine	9.86	7.68	1.03	1.15	.97	.68	
Tyrosine	.08	.08	.19	.23	.14	.13	
Phenylalanine	1.10	1.24	.67	.72	.84	.83	
Ornithine	.09	.08	.16	.09	.10	.10	
Lysine	1.23	1.00	1.80	1.66	2.18	1.70	
Histidine	.28	.24	.37	.41	.48	.41	
Tryptophan	.08	.05	.16	.15	.20	.20	
Arginine	.06	... ^a	.05	.02	.02	.05	

^a Traces.

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In Vitro Digestion of Raw, Roasted, and Pressure-Flaked Corn^{1,2,3}

B. R. TONROY and T. W. PERRY

Department of Animal Sciences
Purdue University, West Lafayette, IN 47907

Abstract

We compared raw, roasted, and pressure-flaked corn in dry matter and starch digestion studies *in vitro*. For dry matter digestion, incubations were 1, 4, 8, 16, 24 and 48 h. Dry matter digestion was increased in hours 1 through 24 by processing. Digestion in the 48-h period was decreased by processing which suggests that processing may increase the ease of utilization but not total utilization. Roasting decreased 48-h dry matter digestion more than pressure flaking. Starch digestion with incubations 1, 4, 24, and 48 h favored processed corn at all hours except 4. The 48-h observations from both studies indicate that while processing enhances starch utilization, it impairs utilization of other components in the grain. Prediction equations for dry matter and starch digestion for each corn treatment from length of incubation period and squared length of incubation period had multiple correlations of .96 or greater.

Introduction

Numerous processing methods have been

Received June 17, 1974.

¹ Journal paper No. 5257 Purdue University Agricultural Experiment Station.

² The authors are grateful to Mrs. Alice Bales for assistance in the chemical analyses.

³ Supported in part by a grant-in-aid plus equipment from Mix Mill, Inc., Bluffton, IN.

developed to improve utilization of feed grains by ruminants. While some of these processes produce reasonably consistent results, those from other similar processes are often varied. Frequently, variations are even within the same process. Examples include work with flaked grain (4, 7, 8), high moisture grain (1, 5, 3), and roasted corn (2). Understanding the mechanism of action not only helps explain variations, but it can be used also to develop a system of quality control, suggest improvements in present processes, and stimulate ideas for new processing techniques. In this study, two *in vitro* experiments compared dry matter and starch digestion in raw, roasted, and pressure-flaked grain.

Experimental Procedure

Raw (Raw 1) and roasted (Roast 1) corn were secured from the Purdue University Livestock Research Farm⁴. Since pressure flaked corn was not available locally, both raw (Raw 2) and pressure flaked (Flake 2) corn of the same origin were purchased commercially. Roast 1 was prepared in an electric powered ROAST-A-TRON.⁵ This machine consists of a cylinder housed within a jacket. Fins on the outside of the cylinder lift the corn through the electric coils. The temperature at which the corn exits the machine is controlled by regulating the rate at which the corn passes through

⁴ West Lafayette, IN.

⁵ Registered trademark, product of Mix Mill, Inc., Bluffton, IN.