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Effect of six months prolonged frozen-storage on changes in organic acid composition of plain soft goat milk cheese

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Abstract

Feasibility of prolonged frozen-storage of goat milk cheeses is extremely important for profitability and sustainability of the dairy goat industry. Extended frozen-storage can be detrimental to the cheese texture and flavour compounds including organic acids. The study was conducted to evaluate the effects of six months of frozen-storage compared to fresh control on organic acid profiles of soft goat cheese. Three lots of plain soft goat cheeses were purchased and each lot of the cheeses was subdivided into four treatment groups as fresh-unfrozen control (UFC), frozen-thaw control (FZC), 3 and 6 months frozen-storage (3FZ and 6FZ). All samples were subjected to ageing at 4 °C for 0, 14, 28 days. Organic acid concentrations for all known standards ranged 0.01 - 13.0 mg/g cheese. Significant effects were observed for most of the known acids, indicating that some variation in manufacturing parameters might have occurred during cheesemaking. Effects of storage treatments (UFC, FZC, 3FZ and 6FZ) were highly significant for most organic acids, except for orotic and a few unknown acids. Ageing at 4 °C for 4 weeks had little influence on all organic acids except butyric acid. Concentrations of butyric, lactic, propionic, tartaric and uric acids were significantly elevated as the frozen-storage period advanced. The UFC cheese had the highest malic and unknown-11 acids, compared to the three frozen groups. A companion study of sensory properties on the same cheeses revealed that practically no differences existed in sensory values among different storage treatments at 0 day of ageing at 4 °C. Prolonged frozen-storage up to six months may be feasible since no apparent deterioration occurred in sensory scores of the goat cheeses although elevations occurred in several organic acid contents.

Keywords: Froze storage, organic acid, goat milk cheese
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Introduction

Organic acids are important flavour compounds of most aged cheeses, which are formed as a result of hydrolysis of milk fat during lipolysis, normal ruminant metabolic processes, bacterial growth or addition of acidulants during cheesemaking (Adda *et al*., 1982; Bevilacqua & Califano, 1989; Akalin *et al.*, 2002; Izco *et al.*, 2002). Organic acids are also the major products of carbohydrate catabolism by lactic acid bacteria.

Quantitative determination of organic acids is an important tool for studying flavour and nutritional quality as well as an indicator of bacterial activity of ageing cheeses, since the total aroma intensity was correlated with organic acid levels in grating cheeses (Akalin *et al.*, 2002). Freezing cheeses is not a common industrial practice (Kosikowski, 1977). However, the seasonality of goat milk production necessitates certain alternative methods of the milk preservation for year-round marketing including frozen-storage of goat cheeses. The effect of freezing on food quality of goat cheeses has little been studied. The purposes of this study were to determine the effect of frozen-storage on organic acid profiles of plain soft goat cheeses, and to study feasibility of prolonged frozen-storage of the goat cheese for later marketing.

Materials and Methods

Three batches of commercial soft goat milk cheeses were purchased from a grade A goat dairy in Georgia. The cheese was manufactured using a modified method of Le Jaouen (1987). Goat milk was pasteurized at 63 °C for 120 minutes and by slow coagulation and natural draining, then hanging the cheese in cheesecloth for three days at cool room temperature (22 °C) before packaging. The cheese was packaged

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in 454 g rod shapes with polyolefin shrink wrap, then shipped to the analytical laboratory in an ice pack box via overnight delivery. A 3.5 g of frozen powdered cheese samples and 20 mL of 0.5% (wt/vol) $(NH_4)_2HPO_4$ were added to a 50 mL Erlenmeyer flask. Organic acids were extracted for 1 h on a shaker at 400 rpm (New Brunswick Scientific, Edison, NJ), then the extracts were centrifuged at 6000 x g for 10 min. The supernatant was filtered through (Supelco Inc.) 0.45 μm membrane filter twice, then 50 μL sample was injected to HPLC. Organic acids of the cheese samples were analyzed using a Hewlett Packard Liquid Chromatography (LC-1100 Series) equipped with auto sampler, quaternary pump, vacuum degasser, and the fluorescence detector which was set at 214 nm. The column used was a ODS Hypersil 5 μm (125 x 4 mm), and the mobile phase was 0.5% (wt/vol) $(NH_4)_2HPO_4$. Column flow rate was 0.3 mL/min. Organic acid standards were purchased from Sigma Chemical Co. (St. Louis, MO), and individual organic acid was quantified on the basis of the external standard method.

Experimental data were analyzed using analysis of variance, correlations between parameters, and least squares mean comparison of organic acids among treated goat cheeses, as described by Steel & Torrie (1960). All data were also analyzed using the General Linear Model of SAS program (SAS, 1990).

Results and Discussion

Six months frozen-storage caused significant ($P < 0.05$ or 0.01) increases in tartaric, lactic, propionic, uric and butyric acid concentrations in the plain soft goat cheese (Table 1 and 2). Acetic acid concentration in FZC cheese was decreased by ageing for 4 weeks at 4 °C, but increased by ageing in fresh cheese (UFC) (Table 1). Soft cheeses are not usually aged, thereby the goat cheeses aged at 4 °C for 4 weeks in this study appeared to be losing acceptable freshness and shelf-life.

Table 1 Comparison of organic acid concentrations among fresh, 0, 3 and 6 month frozen-stored soft goat milk cheeses aged at 4 °C for 4 weeks

Organic acids	UFC			FZC			3FZ			6FZ		
	0 d	14 d	28 d	0 d	14 d	24 d	0 d	14 d	24 d	0 d	14 d	24 d
Tartaric	0.86	0.93	0.93	0.75	0.67	0.70	1.53	1.49	1.42	1.62	1.79	1.92
Formic	2.32	2.23	2.21	2.66	2.60	2.59	1.63	1.74	1.81	2.31	2.37	2.61
Orotic	0.042	0.011	0.012	0.043	0.014	0.013	0.011	0.037	0.036			
Malic	1.13	1.32	1.44	1.22	1.40	1.21	0.42	0.44	0.30			
Lactic	10.04	10.22	10.37	8.96	10.15	9.83	11.19	10.97	10.70	12.47	12.30	12.91
Acetic	2.86	4.20	4.34	5.01	4.03	3.69	3.24	3.32	2.65	3.04	3.41	4.41
Citric	0.69	0.82	0.87	0.88	0.89	0.81	2.12	1.73	1.58	0.58	0.92	1.42
Uric	0.017	0.020	0.015	0.029	0.038	0.034	0.037	0.043	0.040	0.083	0.085	0.084
Propionic	0.71	0.79	0.69	1.28	0.60	0.83	1.53	2.13	2.15	5.36	4.25	4.84
Butyric	1.07	1.20	1.29	1.01	1.21	1.62	1.16	1.53	1.83	2.76	2.93	4.93

UFC -fresh-unfrozen control; FZC - frozen-thaw control; 3FZ -3 months frozen-storage; 6FZ - 6 frozen-storage

Hough *et al.* (1996) showed that the flavour descriptors in Reggianito grating cheese, such as total intensity, cheesy, salty, tongue-tingling, hot and residual intensity could be predicted from organic acids. They noticed that propionic acid was a good indicator of flavour development, and total aroma intensity was well correlated by organic acid contents. Propionic acid in this study was also increased ($P < 0.01$) by frozen-storage (Table 2). The longer the frozen-storage, the greater the elevation of propionic acid (Table 2). The same trend of elevations in other organic acids suggests that flavour development in the cheese has occurred through the catabolic activities of lactic acid bacteria as well as lipolysis of cheese fat by the activation of lipolytic enzymes (Akalin *et al.*, 2002; Izco *et al.*, 2002).

Lactic acid was in greatest concentrations among all known organic acids in the soft goat cheese, while orotic acid was the lowest (Tables 1). There was no pyruvic acid in the soft goat cheese, while several unknown large peaks appeared between propionic and butyric acids. Although organic acid concentrations increased, the apparent sensory qualities in the goat cheese appeared to be unaffected by six months

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prolonged frozen-storage. This result is in agreement with the report of Bertola *et al.* (1996) that frozen Mozzarella could be stored at –20 °C without quality loss as long as the final product had been aged from 14 to 21 days before being consumed.

Table 2 Comparison of mean concentrations of individual organic acids for pooled data across ageing period for the four storage treatment groups

Tartaric		Formic		Lactic		Acetic		Citric		Propionic		Butyric	
Treat	Mean	Treat	Mean	Treat	Mean	Treat	Mean	Treat	Mean	Treat	Mean	Treat	Mean
FZ6	1.78[a]	FZC	2.62[a]	FZ6	12.56[a]	FZC	4.17[a]	FZ3	1.81[a]	FZ6	4.82[a]	FZ6	3.54[a]
FZ3	1.48[b]	FZ6	2.43[ab]	FZ3	10.95[b]	UFC	3.88[ab]	FZ6	0.97[b]	FZ3	1.94[b]	FZ3	1.51[b]
UFC	0.93[c]	UFC	2.23[b]	UFC	10.26[bc]	FZ6	3.62[ab]	FZC	0.86[b]	FZC	0.90[c]	FZC	1.28[b]
FZC	0.71[d]	FZ3	1.73[c]	FZC	9.46[c]	FZ3	3.07[b]	UFC	0.80[b]	UFC	0.69[c]	UFC	1.19[b]

UFC -fresh-unfrozen control; FZC - frozen-thaw control; 3FZ -3 months frozen-storage; 6FZ - 6 frozen-storage
Means within columns with different letters differ significantly ($P < 0.05$ or 0.01)

Conclusions

The study confirmed that tartaric and propionic acids were important organic acid predictors for plain soft goat cheese, and the elevation of the several organic acids did not have any negative influences on the acceptability of the frozen-stored product. Six months frozen-stored, thawed and 0 day refrigerated storage showed minimal changes in organoleptic qualities of the cheese product, indicating that the six months prolonged frozen-storage appears to be feasible for the off-season marketing of the soft goat milk cheese.

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