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PREACIDIFICATION TREATMENTS OF MILK USED IN HALLOUMI CHEESE MANUFACTURE

A. KELES*, Y. DOGRUER, G. UCAR and A. GUNER

Department of Food Science and Technology, Faculty of Veterinary Science,
Selcuk University, 42031, Konya. Turkey

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In this study, Halloumi cheese samples were produced from preacidified cow's milk. Glucono-delta-lactone (gdl), lactic, citric and acetic acids were used in preacidification treatments. The cheese samples were vacuum packed and ripened at 7±1 °C for 90 days. The changes in some chemical, microbiological and sensory properties of the Halloumi cheese samples were investigated during the ripening period. Gdl and lactic acid were found more suitable than citric and acetic acids as an acidulant in Halloumi cheese production.

Keywords: preacidification, Halloumi cheese

Halloumi is a semi-hard to hard cheese variety made from mainly sheep's and goat's milk or mixtures of these (ROBINSON, 1991), and in recent years cow's milk is also used in its production (ECONOMIDES et al., 1987). There are some studies on Halloumi cheese made from recombined milk, too (LELIEVRE et al., 1990; 1991). It is a traditional cheese of Cyprus and widely popular in the Middle East (SHAKER et al., 1987; ROBINSON et al., 1991; PAPADEMAS & ROBINSON, 2000).

In cheese making practise, milk is generally acidified by bacterial cultures, which ferment lactose to lactic acid. In some cases, starters are inhibited by bacteriophages or inhibitory substances, thus pH decreasing does not occur or occurs insufficiently or takes more time to reach renneting pH. For this reason direct acidification techniques are in use (EL-SHENAWY & MARTH, 1990; YILDIRIM & URAZ, 1998; FUNAHASHI et al., 2000; METZGER et al., 2000). For this purpose different acids and acidification agents can be used. The most frequently used are lactic acid, gluconic acid, hydrochloric acid, citric acid, glucono-delta-lactone (gdl), acetic acid and propionic acid (EL-SHENAWY & MARTH, 1990; LUCEY & SINGH, 1998; LUCEY et al., 2000; METZGER et al., 2000).

Gluconic acid and gdl are more useful in food and dairy industry. In France the use of gdl in controlling acidification in cheesemaking has been accepted for the production of most French cheeses (esp. Camembert, Emmental, St. Paulin) as an acidulant to standardize the renneting pH (SERPELLONI et al., 1990). METZGER and co-workers (2000) used acetic and citric acids in preacidification of Mozzarella cheese production.

* To whom correspondence sould be addressed.
Phone: +90 0332 241 00 41/2732; fax: +90 0332 241 00 63; e-mail: akeles@selcuk.edu.tr.

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In an other research lactic, citric and hydrochloric acids were used in whey cheese production (TOUZOPOULOU et al., 2000).

In present study gdl, lactic acid, citric acid and acetic acid were used in direct acidification of milk in Halloumi cheese production. The effects of acidification of milk used in the production of Halloumi cheese by different acids on some chemical, microbiological and sensory properties of cheese were determined during 90 days of ripening.

1. Materials and methods

Halloumi cheese samples were produced in five groups and production was repeated four times. Milk used in the production was obtained from the dairy plant of Selcuk University and cheese samples were produced according to the traditional Halloumi cheese production procedure (ANIFANTAKIS & KAMINARIDES, 1983; KAMINARIDES et al., 1984; ECONOMIDES et al., 1987; SHAKER et al., 1987). Totally 200 l of milk was used in each trial. Milk was heated to 65 °C for 30 min and cooled to 40 °C and then 0.02% $CaCl_2$ was added. Then it was divided into 5 groups (40 l for each group). After 5 min, gdl, lactic acid, citric acid, acetic acid were added (0.015%) individually to four groups of milk. The last group was a control group and no acidification was applied. After addition of acids, chymosin (REN-NA, Liquid Animal Rennet, TSI INC., Lakeville, IN, USA) was added at a rate of 0.10 ml l^{-1} of milk. Prior to addition, chymosin was diluted in 200 ml of cold water. After coagulation was completed (nearly 90 min after renneting), curd was cut in size of 1 cm^3 and heated to 45 °C with gentle agitation and was held at this temperature for five min. Then whey was drained and the curd was pressed at a rate of 7 kg kg^{-1} for 1 h. After pressing, the curd was cut into blocks of approx. 10×15×3 cm in size. Whey was heated to 75 °C and the curd blocks were transferred to the hot whey and cooked for 5–10 min until blocks floated to the surface of the whey. After removing from hot whey, curd blocks were put to rest for cooling and then were dry salted with NaCl at a rate of 2.5% (w/w). After resting overnight, the blocks were vacuum packed in polyethylene bags. Cheese samples were then stored at 7±1 °C for ripening.

Cheese samples were examined for chemical and microbiological attributes at 1st, 7, 15, 30, 60 and 90th days of ripening. Sensory analysis were performed at 30, 60 and 90th days of ripening.

Moisture contents of the samples were determined according to the BRITISH STANDARD (1963). Fat contents were determined by the Gerber Method (MARSHALL, 1992), and acidity values by the method of the TURKISH STANDARD (1974). pH values were determined by the MARSHALL method (1992) with a digital pH meter (NEL Mod. 821) at 25±1 °C.

Total viable aerobic mesophilic colony counts were determined in plate count agar (Merck). Plates were incubated at 30±1 °C for 72±1 h (HARRIGAN & MC CANCE, 1976). Coliform counts were determined in violet red bile agar (Merck). Plates were incubated at 30±1 °C for 24±1 h (HARRIGAN & MC CANCE, 1976). Yeast and mould counts were determined in potato dextrose agar (Merck), pH 3.5 set with 10% sterile tartaric acid solution. Plates were incubated at 22±1 °C for five days (MARSHALL, 1992). Staphylococci and *Staphylococcus aureus* counts were determined in Baird Parker egg yolk tellurite agar (Merck). Plates were incubated at 37±1 °C for 48±1 h. Shiny black colonies of surface growth surrounded by an opaque zone were suspected for *St. aureus*, and catalase and coagulase tests were applied to these colonies (HARRIGAN & MC CANCE, 1976). Lactobacilli counts were determined on Rogosa agar (Merck). Plates were incubated at 30±1 °C for five days (HARRIGAN & MC CANCE, 1976).

Sensory properties were determined by a 6-member sensory panel according to the procedure of the INTERNATIONAL DAIRY FEDERATION (1987). Panelists rated the sensory characteristics of 100 points in total.

As statistical calculations, variance analysis was applied by using SPSS-Windows statistical program and Duncan's Multiple Range Test was used for determining the differences (STEEL & TORRIE, 1981).

2. Results and discussion

Experimental Halloumi cheese samples were produced in five groups. Four different acids were used in acidification of milk. These were glucono-delta-lactone (gdl)(I), lactic acid (II), citric acid (III) and acetic acid (IV). The last group was used as a control group (V) and no acidification was applied. In an earlier report (METZGER et al., 2000), preacidification treatments were used in Mozzarella cheese production by using citric and acetic acids with combination of starters, and milk was ripened for 30–60 min after acidification. In the present study ripening was not applied to milk. After addition of acids, especially in 3rd and 4th groups, precipitation began, so that rennet was added immediately after acid addition. Authors could not wait for ripening, probably because the acidification was poorer than in the present study. Acidification was used until pH reached 5.8 and 6.0 in different trials. In the present study pH values of milk were lowered to 5.65, 5.60 and 5.53 in groups II, III and IV, respectively, after addition of acids. Changes in pH and acidity (lactic acid, l.a.%) values were detected from beginning to the 60th min after acidification and renneting. After acid addition, at the 30th min, pH values were detected as 5.97, 5.71, 5.58, 5.54 in groups I, II, III and IV, respectively, and 6.39 in group V (control group). After further incubation the pH values practically did not change. Initial acidity value of milk was 0.21 l.a.%. After acid addition, the values increased to 0.33, 0.39, 0.46 and 0.50 l.a.% in groups I, II, III and IV, respectively. Then acidity values were nearly constant at both the 30th and 60th min. Data obtained are shown in Table 1.

Table 1. Differences in pH and acidity (l.a.%) values of milk after acidification

		Milk after acid addition					
		I	II	III	IV	V	F
Milk	pH	6.41 ± 0.049	6.41 ± 0.049	6.41 ± 0.049	6.41 ± 0.049	6.41 ± 0.049	–
	Acidity	0.21 ± 0.004	0.21 ± 0.004	0.21 ± 0.004	0.21 ± 0.004	0.21 ± 0.004	–
Acid addition	pH	6.13 ± 0.073^{b}	5.65 ± 0.076^{a}	5.60 ± 0.081^{a}	5.53 ± 0.073^{a}	6.36 ± 0.063^{a}	25.186***
	Acidity	0.33 ± 0.006^{d}	0.39 ± 0.007^{a}	0.46 ± 0.009^{b}	0.50 ± 0.017^{a}	0.23 ± 0.010^{a}	98.085***
30th min	pH	5.97 ± 0.012^{b}	5.71 ± 0.010^{bc}	5.58 ± 0.096^{a}	5.54 ± 0.010^{a}	6.39 ± 0.053^{a}	13.140***
	Acidity	0.34 ± 0.013^{b}	0.39 ± 0.019^{a}	0.42 ± 0.020^{a}	0.43 ± 0.014^{a}	0.23 ± 0.065^{a}	31.050***
60th min	pH	5.98 ± 0.112^{b}	5.75 ± 0.125^{bc}	5.62 ± 0.126^{ba}	5.57 ± 0.137^{a}	6.43 ± 0.073^{a}	8.986***
	Acidity	0.33 ± 0.022^{b}	0.39 ± 0.021^{a}	0.43 ± 0.015^{a}	0.42 ± 0.012^{a}	0.23 ± 0.001^{a}	26.355***

^{a,b,c} Means within the same row not sharing common superscripts are different.

*** $P<0.001$

I: Milk with Gdl added; II: milk with lactic acid added; III: milk with citric acid added; IV: milk with acetic acid added; V: control group

Table 2. Chemical properties of Halloumi cheese samples and pH values during ripening period

Ripening time	Property	Cheese samples					
		I	II	III	IV	V	F
1st Day	Moisture	43.08 ± 0.53	42.00 ± 0.88	42.48 ± 1.66	41.52 ± 0.87	45.23 ± 0.78	2.007
	Fat	23.00 ± 2.15	22.25 ± 1.53	20.75 ± 2.87	22.12 ± 2.6	24.87 ± 3.34	0.342
	FDM	40.47 ± 4.00	38.39 ± 2.73	35.74 ± 3.96	37.76 ± 4.26	45.28 ± 5.69	0.735
	Acidity	0.19 ± 0.05	0.17 ± 0.1	0.19 ± 0.03	0.22 ± 0.04	0.14 ± 0.02	0.792
	pH	6.12 ± 0.24	6.06 ± 0.16	6.02 ± 0.12	6.02 ± 0.09	6.53 ± 0.13	1.851
7th Day	Moisture	34.64 ± 1.59	35.48 ± 1.58	37.89 ± 1.18	35.25 ± 2.01	36.49 ± 1.72	0.603
	Fat	28.13 ± 1.78	27.13 ± 1.66	24.75 ± 3.20	24.38 ± 1.97	26.75 ± 2.50	0.489
	FDM	43.30 ± 3.83	41.97 ± 2.04	39.64 ± 4.59	37.52 ± 2.34	42.17 ± 4.05	0.436
	Acidity	0.22 ± 0.03	0.19 ± 0.03	0.22 ± 0.05	0.24 ± 0.05	0.14 ± 0.03	1.013
	pH	6.08 ± 0.19^{ab}	6.06 ± 0.12^{b}	6.04 ± 0.1^{b}	5.92 ± 0.07^{b}	6.43 ± 0.06^{a}	2.705*
15th Day	Moisture	35.80 ± 2.36	35.37 ± 2.15	35.85 ± 1.77	33.52 ± 2.47	37.34 ± 1.07	0.457
	Fat	25.00 ± 1.47	27.25 ± 1.44	27.38 ± 1.70	24.75 ± 2.14	26.63 ± 2.61	0.420
	FDM	39.01 ± 2.17	42.15 ± 1.76	42.63 ± 2.09	37.27 ± 3.18	42.33 ± 3.58	0.823
	Acidity	0.23 ± 0.04	0.28 ± 0.04	0.32 ± 0.06	0.34 ± 0.06	0.16 ± 0.01	2.254
	pH	6.08 ± 0.13^{b}	5.93 ± 0.10^{b}	5.95 ± 0.11^{b}	5.82 ± 0.08^{b}	6.39 ± 0.08^{a}	4.826*
30th Day	Moisture	34.12 ± 2.16	33.37 ± 2.63	35.15 ± 1.21	35.72 ± 2.80	36.47 ± 2.57	0.279
	Fat	28.50 ± 1.57	27.00 ± 1.96	26.13 ± 1.66	23.50 ± 1.43	26.25 ± 1.11	1.338
	FDM	43.28 ± 2.14	40.38 ± 1.52	40.26 ± 2.24	36.57 ± 1.67	41.37 ± 1.32	1.815
	Acidity	0.30 ± 0.02^{a}	0.31 ± 0.01^{a}	0.31 ± 0.04^{a}	0.34 ± 0.03^{a}	0.18 ± 0.02^{b}	5.009**
	pH	5.95 ± 0.20	5.94 ± 0.17	5.82 ± 0.15	5.90 ± 0.08	6.33 ± 0.06	1.964
60th Day	Moisture	34.25 ± 2.18	34.93 ± 1.38	33.33 ± 3.45	34.96 ± 1.59	36.24 ± 2.74	0.200
	Fat	28.13 ± 1.36	26.63 ± 1.34	27.75 ± 1.93	23.38 ± 2.79	26.00 ± 2.04	0.916
	FDM	42.96 ± 2.88	41.01 ± 2.45	41.65 ± 2.02	35.86 ± 4.00	40.90 ± 3.38	0.799
	Acidity	0.27 ± 0.04	0.27 ± 0.02	0.27 ± 0.03	0.33 ± 0.03	0.24 ± 0.07	0.580
	pH	5.97 ± 0.18	5.99 ± 0.07	5.80 ± 0.08	5.84 ± 0.09	6.25 ± 0.11	2.482
90th Day	Moisture	33.14 ± 1.97	33.12 ± 2.29	34.91 ± 2.27	35.82 ± 0.84	38.29 ± 1.91	1.241
	Fat	26.88 ± 2.16	27.25 ± 1.80	24.25 ± 1.98	21.50 ± 1.59	25.50 ± 2.50	1.309
	FDM	40.07 ± 2.37	40.73 ± 2.38	37.28 ± 2.75	33.51 ± 2.54	41.14 ± 3.14	0.438
	Acidity	0.32 ± 0.05	0.31 ± 0.05	0.32 ± 0.08	0.37 ± 0.06	0.26 ± 0.06	1.223
	pH	5.81 ± 0.13	5.94 ± 0.11	5.87 ± 0.18	5.85 ± 0.11	6.19 ± 0.15	1.452

^{a,b,c} Means within the same row not sharing common superscripts are different.
* P<0.05
** P<0.01
FDM: Fat in dry matter
I: Gdl group samples; II: lactic acid group samples; III: citric acid group samples; IV: acetic acid group samples; V: control group samples

Table 3. Microbiological properties of Halloumi cheese samples during ripening period (Log 10 CFU g^{-1})

Ripening time	Microorganism	Cheese samples					
		I	II	III	IV	V	F
1st Day	Total mesophiles	5.60 ± 0.63	5.11 ± 0.76	5.55 ± 0.44	4.99 ± 0.77	5.29 ± 0.64	0.161
	Coliform	4.39 ± 0.60	3.78 ± 0.80	4.24 ± 0.62	4.45 ± 0.52	4.21 ± 0.42	0.182
	Staphylococci	4.52 ± 0.65	4.68 ± 0.61	5.25 ± 0.28	4.41 ± 0.68	4.35 ± 0.59	0.389
	St. aureus	3.35 ± 1.06	3.55 ± 0.67	3.84 ± 0.20	3.46 ± 0.62	3.25 ± 0.31	0.160
	Yeast and moulds	3.25 ± 0.23	2.80 ± 0.10	2.85 ± 0.18	3.45 ± 0.28	3.18 ± 0.58	0.616
	Lactobacilli	2.06 ± 1.33	2.85 ± 1.11	1.27 ± 1.27	1.94 ± 1.17	2.06 ± 1.21	0.213
7th Day	Total mesophiles	6.63 ± 0.77	6.58 ± 0.72	6.24 ± 0.89	5.94 ± 1.02	6.77 ± 0.66	0.172
	Coliform	4.86 ± 0.14	4.02 ± 0.80	4.83 ± 0.44	3.26 ± 0.20	4.64 ± 0.43	1.725
	Staphylococci	5.37 ± 0.87	5.38 ± 0.94	5.23 ± 0.93	5.12 ± 0.90	5.41 ± 0.77	0.020
	St. aureus	4.73 ± 1.22	5.31 ± 0.97	4.39 ± 0.84	4.91 ± 0.79	4.66 ± 0.73	0.140
	Yeast and moulds	3.98 ± 0.60	4.16 ± 0.40	4.08 ± 0.41	4.36 ± 0.40	4.15 ± 0.47	0.093
	Lactobacilli	3.80 ± 0.40	4.61 ± 0.82	3.13 ± 1.16	3.13 ± 1.06	2.99 ± 1.74	0.368
15th Day	Total mesophiles	6.79 ± 0.95	6.75 ± 0.89	6.66 ± 0.77	6.64 ± 1.08	6.91 ± 0.57	0.015
	Coliform	5.58 ± 0.49	5.26 ± 0.72	4.33 ± 0.57	3.85 ± 0.56	4.76 ± 0.53	1.316
	Staphylococci	5.79 ± 0.97	5.70 ± 0.90	5.57 ± 0.80	6.21 ± 0.70	5.83 ± 0.64	0.073
	St. aureus	4.89 ± 1.01	5.02 ± 0.89	4.95 ± 0.82	5.15 ± 0.87	4.74 ± 0.52	0.031
	Yeast and moulds	4.51 ± 0.30	4.74 ± 0.55	4.51 ± 0.18	4.63 ± 0.40	3.87 ± 0.61	0.598
	Lactobacilli	5.06 ± 1.05	3.91 ± 1.49	4.23 ± 0.60	3.87 ± 1.33	4.43 ± 1.50	0.152
30th Day	Total mesophiles	7.18 ± 0.62	7.13 ± 0.57	6.34 ± 0.59	6.46 ± 0.65	8.09 ± 0.42	1.490
	Coliform	5.17 ± 0.21[a]	4.21 ± 0.44[ab]	5.55 ± 0.79[a]	2.99 ± 0.12[b]	4.15 ± 0.53[ab]	3.424*
	Staphylococci	6.04 ± 0.77	5.95 ± 0.85	5.89 ± 0.69	5.52 ± 0.85	6.73 ± 0.64	0.327
	St. aureus	5.23 ± 0.68	4.99 ± 0.83	4.59 ± 0.50	4.16 ± 0.58	5.62 ± 0.53	0.786
	Yeast and moulds	3.68 ± 0.33	4.33 ± 0.37	4.72 ± 0.24	4.76 ± 0.13	4.22 ± 0.62	1.345
	Lactobacilli	6.54 ± 0.42	5.58 ± 0.64	5.01 ± 0.84	6.11 ± 1.02	5.43 ± 1.28	0.452

Table 3 (cont.)

60th Day	Total mesophiles	7.79 ± 0.42	6.82 ± 0.49	7.27 ± 0.21	6.80 ± 0.31	7.64 ± 0.27	1.640
	Coliform	5.85 ± 0.16[a]	4.54 ± 0.71[b]	5.19 ± 0.45[a]	3.14 ± 0.37[b]	5.26 ± 0.42[a]	3.735*
	Staphylococci	6.59 ± 0.39	5.69 ± 0.71	6.38 ± 0.52	5.99 ± 0.71	6.86 ± 0.26	0. 734
	St. aureus	5.32 ± 0.71	4.58 ± 0.63	5.41 ± 0.53	4.39 ± 0.47	5.00 ± 0.56	0.588
	Yeast and moulds	3.90 ± 0.33	4.14 ± 0.46	4.54 ± 0.25	4.70 ± 0.25	4.18 ± 0.35	0.899
	Lactobacilli	5.21 ± 0.23	5.15 ± 0.70	5.49 ± 0.85	4.26 ± 0.48	4.91 ± 0.77	0.518
90th Day	Total mesophiles	7.45 ± 0.62	7.05 ± 0.58	7.17 ± 0.44	6.99 ± 0.55	7.93 ± 0.40	0.538
	Coliform	4.16 ± 0.50	3.78 ± 0.63	3.63 ± 0.29	2.70 ± 0.32	5.18 ± 0.28	1.673
	Staphylococci	6.08 ± 0.30	5.75 ± 0.34	6.17 ± 0.51	6.20 ± 0.26	6.39 ± 0.34	0.413
	St. aureus	5.01 ± 0.34	4.85 ± 0.37	5.74 ± 0.17	5.09 ± 0.41	4.70 ± 0.31	1.267
	Yeast and moulds	3.68 ± 0.70	3.61 ± 0.75	4.31 ± 0.35	4.80 ± 0.38	3.65 ± 0.73	0.753
	Lactobacilli	5.71 ± 0.99	5.42 ± 0.68	4.99 ± 0.69	5.72 ± 0.32	6.58 ± 0.41	0.778

[a,b,c] Means within the same row not sharing common superscripts are different.

* $P<0.05$

I: Gdl group samples; II: lactic acid group samples; III: citric acid group samples; IV: acetic acid group samples; V: control group samples

Table 4. Sensory properties of Halloumi cheese samples during ripening period

Ripening time	Property	Cheese samples					
		I	II	III	IV	V	F
30th Day	Flavour (45)	40.46 ± 0.95^{a}	36.75 ± 1.18^{b}	37.50 ± 1.23^{ab}	33.92 ± 1.12^{b}	37.08 ± 1.43^{ab}	3.803**
	Texture (30)	27.42 ± 0.66^{a}	25.13 ± 0.85^{ab}	25.29 ± 1.04^{a}	22.79 ± 0.69^{b}	25.71 ± 0.94^{a}	3.816**
	Appearance (15)	13.83 ± 0.39^{a}	12.67 ± 0.55^{a}	10.92 ± 0.63^{b}	11.00 ± 0.63^{b}	13.04 ± 0.50^{a}	5.563***
	Colour (10)	9.42 ± 0.21^{a}	8.96 ± 0.29^{a}	7.00 ± 0.40^{b}	6.96 ± 0.42^{b}	8.71 ± 0.36^{a}	11.115***
	Total (100)	91.13 ± 1.51^{a}	83.50 ± 2.05^{b}	80.71 ± 2.73^{b}	74.67 ± 1.91^{c}	84.54 ± 2.37^{b}	7.725***
60th Day	Flavour (45)	39.17 ± 0.90^{ab}	40.92 ± 1.06^{a}	35.04 ± 1.62^{c}	36.79 ± 1.48^{bc}	38.83 ± 1.21^{ab}	3.150*
	Texture (30)	24.54 ± 0.75^{bc}	27.33 ± 0.57^{a}	22.88 ± 1.16^{c}	24.42 ± 1.05^{c}	25.88 ± 0.69^{ab}	3.694**
	Appearance (15)	14.29 ± 0.29^{a}	13.67 ± 0.43^{a}	10.96 ± 0.66^{b}	11.71 ± 0.61^{b}	13.75 ± 0.54^{a}	7.711***
	Colour (10)	9.38 ± 0.25^{a}	9.38 ± 0.22^{a}	6.96 ± 0.37^{b}	6.96 ± 0.35^{b}	9.58 ± 0.16^{a}	23.676***
	Total (100)	87.38 ± 1.54^{a}	91.29 ± 1.84^{a}	75.83 ± 3.24^{b}	79.88 ± 2.67^{b}	88.04 ± 2.15^{a}	7.303***
90th Day	Flavour (45)	40.38 ± 0.73^{a}	40.83 ± 0.72^{a}	37.54 ± 0.94^{b}	37.63 ± 1.11^{b}	41.13 ± 0.85^{a}	4.013**
	Texture (30)	26.46 ± 0.68^{a}	27.29 ± 0.54^{a}	21.46 ± 1.12^{b}	21.79 ± 1.09^{b}	27.50 ± 0.72^{a}	12.276***
	Appearance (15)	13.50 ± 0.35^{a}	13.13 ± 0.44^{a}	10.67 ± 0.66^{b}	10.46 ± 0.61^{b}	13.54 ± 0.41^{a}	9.522***
	Colour (10)	8.92 ± 0.25^{a}	8.71 ± 0.28^{a}	7.46 ± 0.36^{b}	6.38 ± 0.33^{c}	9.17 ± 0.23^{a}	16.002***
	Total (100)	89.25 ± 1.30^{a}	89.96 ± 1.41^{a}	77.13 ± 2.64^{b}	76.25 ± 2.51^{b}	91.33 ± 1.74^{a}	13.849***

[a,b,c] Means within the same row not sharing common superscripts are different.

* $P<0.05$

** $P<0.01$

*** $P<0.001$

I: Gdl group samples; II: lactic acid group samples; III: citric acid group samples; IV: acetic acid group samples; V: control group samples

Moisture contents of the samples from acidified groups were found to be lower than that of the control group during the ripening period (Table 2). Statistically significant differences were found ($P<0.05$) only on the first day. Differences were probably due to the lower coagulation pH values in acidified groups. Decreasing of pH accelarates draining of the whey from the curd. Fat and fat in dry matter ratios were similar in all groups.

Acidity values of acidified groups were found higher than the control group in every stages of ripening but only the 30th day values were significantly ($P<0.05$) different. The highest acidity values during ripening were determined in the acetic acid group. The acidity values were found lower than those of other reports (ATASEVER et al., 1999; KELES et al., 2001) on acidity values of Halloumi cheese. Differences were probably due to the starter culture used. Also the acidity coursed by acid addition in the initial stage was probably reduced by draining of the whey and cooking of the curd in hot whey. In addition, pH values were found higher than in normally ripened cheese. In every stage of ripening the highest pH values were found in control group samples, however, differences were found significant only ($P<0.05$) on the 7th and 15th day of analysis. Obtained data were found higher than those of several investigators (ANIFANTAKIS & KAMINARIDES, 1983; SHAKER et al., 1987; LELIEVRE et al., 1990; 1991; ATASEVER et al., 1999; KELES et al., 2001).

In microbiological analysis, total viable aerobic mesophilic counts were found to increase during the ripening period (Table 3). The highest counts were detected for control group samples, but differences were not significant ($P>0.05$). The lowest counts were found in acetic acid group samples (IV) and the total viable aerobic counts were lower than those reported earlier by other investigators (ANIFANTAKIS & KAMINARIDES, 1983; CHATER, 1984; WILLIAMS & SYSON, 1984; ATASEVER et al., 1999; KELES et al., 2001).

Coliform counts were generally detected in high numbers. Normally, in all cheese varieties, coliform counts generally exhibit a decrease during the ripening period. In the present study, such a distinctive decrease was not determined. The reason for this might be the lower acidity and the higher pH values. The highest counts were detected in gdl group (I), and the lowest counts were found in acetic acid group (IV) samples during the ripening period. Differences among the groups were significant at the 30th and 60th day of ripening ($P<0.05$).

Staphylococci and *St. aureus* counts were higher than coliforms and gradually increased during the ripening. Counts were similar between groups.

Yeast and mould counts were in low at the initial stage and gradually increased during ripening. Differences between groups were not significant ($P>0.05$), but the highest counts were detected in acetic acid group samples (IV) and the lowest counts were detected in gdl group samples (I). Yeast and mould counts were found lower than reported by some papers before (ATASEVER et al., 1999; PAPADEMAS & ROBINSON, 2000; KELES et al., 2001).

Lactobacilli counts were very low at the initial stage of ripening and they gradually increased during the ripening period. There was no significant difference between groups (P>0.05). Increases were fairly similar to the increases of acidity values. The reason of lower numbers is probably the absence of starter culture.

In sensory analysis, significant differences were detected in every stages of ripening (P<0.05, 0.01, 0.001) (Table 4). Generally, gdl (I) and lactic acid (II) group samples were given the highest scores, while acetic acid (IV) group samples the lowest ones. At the 30th day of analysis, gdl group (I) samples, at the 60th day, lactic acid group (II) samples and finally at the 90th day control group (V) samples were more preferred. This can probably be explained that the maturation process in gdl (I) and lactic acid (II) groups occurred more rapidly.

3. Conclusions

According to results of the study, gdl and lactic acids are more suitable than citric and acetic acids as acidulants in Halloumi cheese production. Microbiological quality was found lower than some cheese varieties produced by using starter cultures. Because of lower acidity and higher pH values and the effects on microbiological quality of cheese (esp. high numbers of coliforms and Staphylococci and low numbers of lactobacilli counts), suitable starter culture combinations could be used with acidulants. Further investigations on the subject should be performed.

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