International Journal of Dairy Technology SDT SOCIETY OF DAIRY TECHNOLOGY

doi: 10.1111/1471-0307.12250

ORIGINAL RESEARCH

Changes in the texture, physicochemical properties and volatile compound profiles of fresh Kashar cheese (<90 days) during ripening

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In this study, four different fresh Kashar cheese samples were ripened for 90 days. The physicochemical properties, texture attributes, fatty acid composition and volatile compound profiles of the samples were measured every 30 days of ripening. The texture properties of the cheese samples were significantly affected by the duration of the ripening period. The results of this study highlighted that texture parameters as a function of ripening period should be considered for both fresh and aged Kashar cheeses to determine the ripening period as they are very important for consumer acceptability and consumption of the end product.

Keywords Fresh Kashar cheese, Ripening, Texture, Volatile compound.

INTRODUCTION

In Turkey, more than fifty different cheese varieties have been produced (Hayaloglu *et al.* 2002) and Kashar is one of the most widely consumed traditional cheese varieties. Kashar cheese is classified based on ripening period and texture characteristics as fresh Kashar (semihard) and old Kashar (hard). The fresh Kashar cheese is produced from pasteurised milk (sheep or cow's milk) and it is sold as unripened, while old Kashar is matured for at least 90 days (TSE 1995). Ripening is a very important process leading to alterations in the content of aromatic compounds, texture and sensory properties (Sousa *et al.* 2001).

The texture of the cheese depends upon the chemical composition of the cheese and biochemical changes occurring throughout ripening (Awad 2006). Textural parameters of the cheese samples are also influenced during the ripening period by pH, moisture and salt content of the sample (Lane *et al.* 1997). Moreover, proteolysis causing to form creates peptides having different molecular weights by breakdown of casein, which plays an important role in changes of the textural parameters of the cheese (Gunasekaran and Ak 2003). Cheese texture has an important role in determining consumer acceptability and the end use of the sample (Everard *et al.* 2006); therefore, observation of these properties as a function of ripening period is very important for producers to determine the ripening time necessary for obtaining cheese with the desired textural properties. Researchers have sought to correlate the textural and sensory characteristics of the cheese samples due to its importance for consumers. To determine textural properties of the samples, texture profile analysis (TPA) is widely used in the food industry. TPA simulates the compressing action of the teeth during mastication (Bourne 1978) and provides information about the hardness, adhesiveness, springiness, cohesiveness, gumminess, chewiness and resilience of the samples. All of these parameters should be considered during production. For example, springiness refers to the recoverability of the sample after it is exposed to forces applied during storage, handling and transportation. Therefore, the springiness value could be taken into consideration during the determination of ageing period for obtaining cheeses with this desired quality.

During cheese ripening, many biochemical and chemical changes occur, which contribute to the formation of the characteristic taste and aroma of the cheese samples (Hayaloglu *et al.* 2002). The coagulant (rennet or rennet substi-

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tute), indigenous milk enzymes, starter, secondary starter and nonstarter bacteria and their enzymes play a very important role throughout ripening (Fox *et al.* 1996). The coagulant is responsible for the formation of the large- and intermediate-sized peptides (Lane and Fox 1997). In addition, enzymes have also a major role in the formation of small peptides and amino acids, which are precursors of the flavour compounds found in cheese (Urbach 1997). Glycolysis of lactose, lipolysis and proteolysis are the principal events occurring during ripening (Fox and McSweeney 1996). Major compounds of the milk such as lactose, milk fat and protein are converted to the flavour compounds throughout ripening (McSweeney 2004). Those events, especially proteolysis, are influenced by pH, salt and moisture content of the curds, ripening temperature and time (Tarakci and Kucukoner 2007). The characteristic aromatic compounds of the cheese samples change during ripening and, therefore, the ripening period is very important for the formation of the desired aroma of the cheeses.

Up to now, we have not encountered any published paper about change in volatile compound profiles and textural characteristics of the fresh Kashar cheese samples during its ripening period. However, observation of the change in these characteristics with respect to ripening period is very important for both producers and researchers. In the consideration of those details, in this study, it was aimed to determine the changes in the physicochemical and texture properties, fatty acid and volatile profile of fresh Kashar cheese samples produced by traditional methods during ripening for up to 90 days.

MATERIALS AND METHODS

Materials

Fresh Kashar cheese samples produced 1 week ago according to the traditional method described by Sert *et al.* (2007) were purchased from four different local producers in Kars, Turkey, and were taken to the laboratory of Erciyes University Food Engineering Department. From each producer, two different samples were obtained from the same production. Wheel-shaped fresh Kashar cheese samples without packaging (approximately 2 kg) were stored at 4 °C during the ripening period. We measured physicochemical and textural properties in 0th, 30th, 60th and 90th days of storage. Volatile compounds and fatty acid composition of the fresh Kashar cheese samples were performed at the beginning and end of the storage.

Methods

Determination of the physicochemical properties of the fresh Kashar cheese samples

The dry matter, ash and protein contents plus pH of the samples were determined according to the methods of the AOAC (2000). Dry matter content of the cheese samples was determined by drying of the samples in a drying oven (Nüve FN 120, Ankara, Turkey) at 105 °C until constant weight was observed. To determine total ash content, cheeses were heated at 550 °C for 5 h in a muffle furnace (Nüve 1000 C, Turkey). The fat content of the samples was determined using the Gerber method. The pH values of the cheeses were measured with a pH meter (Inolab Terminal Level 3, Germany). Protein content of the cheeses was determined using an automatic nitrogen analyser (FP 528 LECO, ABD) based on the Dumas method.

Texture profile analysis

Texture profile analysis (TPA) of the fresh Kashar cheeses was performed based on the method described by Eroglu *et al*. (2015) using a TA-XT2 texture analyzer (Stable Micro Systems, Surrey, UK). Prior to the textural analysis, samples were diced to 20 mm cubes. A cylindrical aluminium probe (aluminium cylinder probe P/50, 50 mm diameter; Stable Micro Systems LTD, Godalming, UK) was used for TPA analysis. Strain value applied during analysis was set to 50% and the test speed was set to 5 mm/s. Pretest speed was selected as 1 mm/s. Texture measurements were performed at 25 °C, and the samples were stored for 30 min at 25 °C for achieving temperature equilibrium. The textural parameters of hardness, adhesiveness, cohesiveness, gumminess, springiness, chewiness and resilience were calculated using the force–deformation curves (Bourne 1978). Analyses were repeated 10 times for each sample.

Fatty acid composition

To determine fatty acid composition of the fresh Kashar cheese samples, fat was extracted from the cheeses using the modified method described by Seçkin *et al.* (2005). Each 100 g cheese sample was mixed with 10 g of kieselguhr and 250 mL hexane. The cheese–hexane mixture was vigorously mixed and filtered manually. The filtered solution (hexane–lipid extracts) was concentrated using a rotary evaporator (Heidolph, Germany) at 25 °C until virtually all of the hexane was evaporated. Extracted lipid was taken and used for further analysis. To determine fatty acid composition of the fresh Kashar cheese, 100 mg of extracted fat was saponified with 100 μL 2 mol/L KOH, and 3 mL *n*-hexane was added to the mixture. The mixture was vigorously shaken with a vortex (Nüve NM 110, Turkey) for 1 min and then centrifuged at 2516 × *g* for 5 min at 25 °C. One millilitre of the supernatant was put into gas chromatograph (GC) vials and injected immediately into a GC (Agilent 6890) equipped with a flame ionisation detector (FID) and a 100 m × 0.25 mm ID HP-88 column. Injection block temperature was set to 250 °C. The oven temperature was kept at 103 °C for 1 min, then programmed from 103 °C to 170 °C at 6.5 °C/min, from 170 °C to 215 °C for 12 min at 2.75 °C/min and finally

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held at 230 °C for 5 min. The carrier gas was helium with a flow rate of 2 mL/min; split rate was 1/50 (Dogan and Akgul 2005). Two replications were performed for the determination of the fatty acid composition of the samples.

Volatile component analysis

Volatile compound composition of the fresh Kashar cheese samples was determined using GC–MS (Agilent 7890A GC system; Agilent, Avondale, USA) according to the modified method of Krist *et al.* (2006). A mass selective detector (Agilent Technologies, Avondale, USA) and HP-5MS column (60 m × 0.250 mm i.d.; film thickness 0.25 μm) were used for this aim. The oven temperature was held at 40 °C for 10 min, increased to 110 °C at 3 °C/min, from 110 °C to 150 °C at 4 °C/min, from 150 °C to 210 °C at 10 °C/min and finally increased to 210 °C/min and held at this temperature for 15 min. The carrier gas was helium with a flow rate of 0.5 mL/min. The voltage of the electron ionisation detector was 70 eV. The compounds adsorbed by the fibres were desorbed from the injection port for 40 min at 50 °C in the splitless mode. The compounds were identified by comparison with spectra from the libraries Flavor 2, NIST 05a and Wiley 7n.

Statistical analysis

All of the results were expressed as mean ± standard deviation calculated using SPSS (SPSS Statistics 17.0, Armonk, NY, USA). Two-way ANOVA was performed using JMP package software (version 5.0.1a; SAS Institute, Inc., Cary, NC, USA) to determine whether the effect of cheese type and ripening period on physicochemical and textural properties of the fresh Kashar cheese samples analysed is statistically significant or not at a significance level of $\alpha = 0.05$.

RESULTS AND DISCUSSION

Physicochemical properties of the fresh Kashar cheese samples

Physicochemical properties of the different fresh Kashar cheese samples ripened at different periods are shown in Table 1. Dry matter content of the Kashar cheese samples increased during the storage period, which is consistent with the study of Hayaloglu *et al.* 2007 and Dervisoglu and Yazici (2001). The initial dry matter content of the Kashar cheese samples was in the range of 45.3% and 55.9%. Final dry matter content of the samples changed between 59.0% and 70.0%. The loss of the moisture level was highest in the C4 sample, which might be associated with the more porous structure of the sample (Hayaloglu *et al.* 2007).

As the initial fat content of the cheeses in dry matter varied between 38.9% and 52.3%, that of the samples at the end of the 90 days of the ripening was in the range of 34.3–45.9%. The differences between the fat content of the Kashar cheese samples might result from the composition of the cheese milk. While the highest fat content was observed in C1 sample (45.9%), the C4 sample had the lowest fat

Table 1 Physicochemical properties of Kashar cheeses during storage period

Property	*Day*	*C1*	*C2*	*C3*	*C4*
Dry Matter (g/100 g)	0	45.3 ± 0.2^{aA}	51.1 ± 0.7^{aC}	55.9 ± 0.1^{aD}	49.7 ± 0.2^{aB}
	30	54.7 ± 0.2^{bAB}	55.7 ± 1.7^{bB}	58.1 ± 1.1^{abC}	53.7 ± 0.5^{bA}
	60	55.9 ± 0.6^{cA}	55.9 ± 1.4^{bA}	59.5 ± 1.2^{bB}	54.2 ± 1.6^{bA}
	90	61.0 ± 0.7^{dA}	59.0 ± 0.7^{cA}	70.0 ± 2.2^{cC}	69.1 ± 0.2^{cB}
Fat in dry matter (%)	0	52.3 ± 1.1^{cD}	45.7 ± 2.8^{aBC}	41.7 ± 2.1^{bB}	38.9 ± 5.0^{bA}
	30	46.3 ± 1.2^{bA}	43.7 ± 1.5^{aB}	45.4 ± 0.9^{cB}	34.8 ± 2.3^{aA}
	60	48.9 ± 1.3^{bB}	45.6 ± 2.0^{aAB}	44.8 ± 1.6^{cA}	41.9 ± 2.9^{Ba}
	90	45.9 ± 0.5^{aC}	45.2 ± 1.0^{aC}	38.6 ± 1.2^{aB}	34.3 ± 1.0^{aA}
Protein (g/100 g)	0	23.9 ± 0.0^{aA}	24.5 ± 0.1^{aB}	25.3 ± 0.0^{aC}	24.2 ± 0.1^{aB}
	30	25.8 ± 0.1^{cB}	26.4 ± 0.2^{cB}	28.6 ± 0.6^{cC}	24.1 ± 0.1^{aA}
	60	25.5 ± 0.0^{bB}	25.3 ± 0.2^{bB}	26.7 ± 0.1^{bC}	24.7 ± 0.2^{bC}
	90	26.3 ± 0.3^{dA}	26.4 ± 0.1^{cA}	26.9 ± 0.2^{aB}	29.4 ± 0.4^{cC}
Ash content (g/100 g)	0	3.56 ± 0.05^{aA}	3.96 ± 0.03^{aB}	3.49 ± 0.04^{aA}	4.10 ± 0.04^{aC}
	30	3.82 ± 0.11^{bA}	4.17 ± 0.12^{aB}	3.86 ± 0.04^{bA}	4.90 ± 0.17^{bC}
	60	4.03 ± 0.06^{bA}	4.75 ± 0.18^{bB}	4.10 ± 0.02^{cA}	4.84 ± 0.13^{bB}
	90	4.52 ± 0.21^{cA}	$5.78 \pm .11^{cB}$	4.44 ± 0.17^{dA}	6.11 ± 0.08^{cC}
pH	0	5.56 ± 0.03^{aA}	5.38 ± 0.02^{aB}	5.39 ± 0.00^{bB}	5.37 ± 0.00^{aB}
	30	5.47 ± 0.01^{bA}	5.39 ± 0.01^{aB}	5.37 ± 0.02^{cC}	5.39 ± 0.01^{aB}
	60	5.43 ± 0.00^{cA}	5.39 ± 0.01^{aB}	5.43 ± 0.01^{aA}	5.32 ± 0.03^{bC}
	90	5.24 ± 0.03^{dB}	5.30 ± 0.02^{bC}	5.17 ± 0.01^{dA}	5.26 ± 0.01^{cB}

a–d: Different superscript lowercase letters denote differences between ripening times within the same sample ($P < 0.05$).

A–D: Different superscript uppercase letters denote differences between the samples within the same ripening time ($P < 0.05$).

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content (34.3%). As C1 and C2 Kashar cheese samples analysed could be classified as full-fat Kashar, which includes at least 45% fat content in dry matter, C3 and C4 were classified as fatty Kashar cheese, which includes fat content ranged between 30 and 45% in dry matter, according to the Kashar regulations (TSE 1995).

The initial protein content of the fresh Kashar cheese samples varied between 23.9% and 25.3%, and after 90 days of ripening, the protein content of the samples was in the range of 26.3–29.4%. Significant differences were observed among cheese samples in terms of protein content during ripening ($P < 0.05$). As seen from Table 1, the protein content of the samples was significantly affected by the ripening time ($P < 0.05$). However, during the ripening period, no proper trend was observed in protein content of the samples and ripening period. For example, the protein content of the C2 sample increased from 24.5% to 26.4% at 30th day of ripening and then decreased to 25.3% at 60th day. The increase in the protein content might be associated with the decrease in moisture content during ripening (Hayaloglu *et al.* 2007).

Ash content of the fresh Kashar cheese samples is also presented in Table 1. The initial ash content of the samples ranged between 3.56% and 4.10%, and at the end of the ripening time, ash content of the samples was in the range of 4.44–6.11%. There were significant differences among the ash content of the samples ($P < 0.05$).

The pH value of the Kashar cheese samples ranged from 5.37 to 5.56 at the start of the ripening time. At the end of the ripening time, the pH varied between 5.17 and 5.30. The pH of the Kashar cheeses decreased during ripening ($P < 0.05$), which might be associated with the metabolism of the lactic acid bacteria which can form lactic acid from lactose (Delgado *et al.* 2011). The findings of the present study are in agreement with the result of the study obtained by Hayaloglu (2009).

Textural properties of the fresh Kashar cheese samples

The average values of the texture profile analysis (TPA) parameters (hardness, adhesiveness, springiness, cohesiveness, gumminess, chewiness and resilience) of the Kashar cheese samples during different ripening period (0th, 30th, 60th and 90th days) are tabulated in Table 2. Hardness of the cheese samples at the beginning of the ripening period was in the range of 7223–8679 g. Hardness values of the C1 and C4 samples were significantly different from each other ($P > 0.05$). The variation between the hardness values of the samples might be associated with the chemical composition of the samples such as fat, protein or dry matter content. The initial hardness value of the C4 sample was lower than that of the C1 sample, which might reflect differences in the dry matter content of the samples. Though the initial protein content of the C4 sample was higher than that of the C1 sample, the C4 sample had lower fat content than in C1 sample. Romeih *et al.* (2002) reported that fat content contributes to smoothness and a softer texture in cheeses, as fat breaks up the protein matrix. In addition, there is a positive correlation between the protein content and hardness of the samples (Koca and Metin 2004). Considering these factors, it was expected that hardness of the C4 sample was higher than that of the C1 sample. The reason for this result might be the porous structure of the C4 sample, resulting from the higher moisture loss from the C4 sample as although the moisture content of the C4 sample was higher than that of the C3 sample, their hardnesses were not significantly different ($P > 0.05$) at the beginning of the ripening period, which might result from the initial lower fat content of the C4 sample. As can be seen from Table 2, magnitudes of the hardness were significantly affected by the ripening period. The hardness value increased up to day 30 for all the Kashar cheese samples, resulting from the loss of the moisture throughout the ripening period (Delgado *et al.* 2011). When considering the relation between moisture content and hardness values of the cheese samples, it was expected that the hardness would increase during ripening; however, that was not observed in C4, which was not significantly different at days 30 and 60 ($P > 0.05$). After day 60 an increase in hardness value was observed in C4 ($P < 0.05$). The poor correlation between the ripening period and hardness might result from several conflicting factors affecting the hardness of the cheese. Karaman and Akalın (2013) reported that increase in the proteolysis of the protein matrix during ripening results in decrease in the hardness.

Table 2 also summarises the change in adhesiveness of the different fresh Kashar cheeses during ripening. Adhesiveness is the force required to remove cheese from the contact surface of the mouth such as palate or tongue (Bryant *et al.* 1995). Adhesiveness values of the samples were very close to each other, especially at beginning and end of ripening. As is seen from Table 2, no direct trend between the adhesiveness values and the ripening period was found. However, when the adhesiveness values of the samples at beginning and end of storage were compared, it can be concluded that the adhesiveness decreased with ripening, which is in agreement with the study of Koca and Metin (2004). Bryant *et al.* (1995) reported that decrease in fat and dry matter content resulted in decrease in adhesiveness of the cheese. However, the opposite result was found in previous study carried out by Karaman and Akalın (2013). The differences between adhesiveness values might have resulted from differences in their chemical composition.

Cohesiveness is a measure of the deformation of cheese before it breaks (Karaman and Akalın 2013). As seen from Table 2, only the cohesiveness of the C4 sample was significantly different from the other Kashar cheeses ($P < 0.05$). Generally, cohesiveness decreased with increase in ripening time. There are both similar and opposite results

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Table 2 Textural properties of Kashar cheese samples during storage

Parameter	*Day*	*C1*	*C2*	*C3*	*C4*
Hardness (g)	0	8679 ± 805^{aB}	7759 ± 216^{aAB}	8470 ± 744^{aAB}	7223 ± 170^{aA}
	30	12679 ± 199^{cB}	11748 ± 1174^{bB}	11011 ± 1099^{bAB}	9573 ± 645^{bA}
	60	11135 ± 131^{bB}	10681 ± 89^{bB}	12039 ± 174^{bC}	9343 ± 749^{bA}
	90	11355 ± 595^{bA}	10719 ± 772^{bA}	12510 ± 463^{bAB}	13066 ± 613^{cB}
Adhesiveness (g.sec)	0	–75.0 ± 2.9^{bB}	–70.8 ± 4.7^{bAB}	–61.0 ± 4.0^{bA}	–75.0 ± 9.3^{bB}
	30	–73.4 ± 14.3^{bA}	–114.0 ± 13.5^{cB}	–88.6 ± 5.0^{cA}	–87.7 ± 1.1^{bA}
	60	–75.0 ± 2.9^{cB}	–54.7 ± 9.6^{bA}	–104.3 ± 24.0^{cB}	–28.5 ± 5.8^{aA}
	90	–6.9 ± 1.7^{aAB}	–10.8 ± 1.7^{aAB}	–5.9 ± 1.0^{aA}	–14.5 ± 6.2^{aB}
Springiness (cm)	0	0.76 ± 0.03^{aA}	0.76 ± 0.02^{aA}	0.72 ± 0.04^{cA}	0.70 ± 0.02^{bA}
	30	0.73 ± 0.01^{aC}	0.64 ± 0.02^{aB}	0.64 ± 0.01^{bB}	0.59 ± 0.02^{abA}
	60	0.72 ± 0.06^{aA}	0.70 ± 0.02^{aA}	0.65 ± 0.03^{bA}	0.63 ± 0.05^{bA}
	90	0.84 ± 0.03^{bB}	0.53 ± 0.01^{aA}	0.54 ± 0.02^{aA}	0.49 ± 0.05^{aA}
Cohesiveness	0	0.69 ± 0.01^{cC}	0.62 ± 0.02 ^{dB}	0.60 ± 0.01^{cB}	0.48 ± 0.03^{bA}
	30	0.61 ± 0.02^{bC}	0.52 ± 0.01^{cB}	0.50 ± 0.03^{bB}	0.40 ± 0.02^{bA}
	60	0.63 ± 0.04^{bB}	0.46 ± 0.01^{bA}	0.48 ± 0.02^{bA}	0.39 ± 0.02^{bA}
	90	0.40 ± 0.02^{aB}	0.36 ± 0.04^{aB}	0.28 ± 0.01^{aA}	0.29 ± 0.03^{aA}
Gumminess (g)	0	6002 ± 613^{bC}	4845 ± 294^{aB}	5071 ± 466^{bBC}	3516 ± 260^{aA}
	30	7817 ± 246^{cC}	6136 ± 555^{bB}	5575 ± 481^{bB}	3836 ± 61^{aA}
	60	7062 ± 540^{cC}	4875 ± 104^{aB}	5782 ± 269^{bB}	3731 ± 446^{aA}
	90	4601 ± 152^{aB}	4040 ± 145^{aAB}	3409 ± 257^{aA}	3748 ± 645^{aA}
Chewiness (g cm)	0	4568 ± 461^{bC}	3708 ± 114^{bB}	3640 ± 149^{bB}	2501 ± 289^{bA}
	30	5749 ± 173^{cC}	3967 ± 322^{bB}	3569 ± 265^{bB}	2275 ± 42^{bA}
	60	5093 ± 163^{bC}	3417 ± 162^{abB}	3772 ± 321^{bB}	2352 ± 74^{bA}
	90	3901 ± 255^{aB}	2568 ± 103^{aA}	1915 ± 80^{aA}	1798 ± 15^{aA}
Resilience	0	0.28 ± 0.01^{cC}	0.25 ± 0.02^{cB}	0.24 ± 0.03^{cB}	0.16 ± 0.02^{cA}
	30	0.24 ± 0.01^{bC}	0.18 ± 0.01^{bB}	0.18 ± 0.02^{bB}	0.12 ± 0.01^{bcA}
	60	0.25 ± 0.02^{bB}	0.16 ± 0.01^{bA}	0.16 ± 0.02^{bA}	0.12 ± 0.01^{abA}
	90	0.15 ± 0.01^{aC}	0.11 ± 0.01^{aB}	0.08 ± 0.00^{aA}	0.08 ± 0.01^{aA}

a–d: Different superscript lowercase letters show differences between ripening times within the same sample ($P < 0.05$).

A–D: Different superscript uppercase letters show differences between the samples within the same ripening time ($P < 0.05$).

in the literature relating to the interaction of cohesiveness and chemical composition. Although a positive correlation was found between the dry matter content and cohesiveness (Koca and Metin 2004), Delgado *et al.* (2011) determined a negative correlation between those two parameters. Moreover, a negative correlation between the fat content and cohesiveness was observed in the other study (Bryant *et al.* 1995).

Springiness is a measure of the ability of a sample to recover when it is deformed during the first compression (Bourne 2002). At the beginning of ripening, as seen from Table 2, springiness values of the cheese samples were very close to each other and, except for C1, springiness values decreased during ripening, which might result from the increase in fat content of the samples. Tunick *et al.* (1993) reported that fat found in the sample decreases the elasticity of the protein network. The result of this study was consistent with the previous studies (Tunick *et al.* 1993; Delgado *et al.* 2011; Karaman and Akalın 2013).

Gumminess is a parameter calculated from the hardness and cohesiveness values of the sample (Bourne 2002), and therefore, factors affecting the hardness and cohesiveness of the sample also affect the gumminess (Goksel *et al.* 2013). As can be seen from Table 2, gumminess was significantly affected by the cheese type and ripening period ($P < 0.05$). A regular trend between gumminess and time was not observed in all Kashar cheese samples, the causes of which were mentioned in the earlier discussion. When the initial and 90 day cheese was compared, it could be concluded that gumminess of the samples, except for C4, increased with ripening time. A similar behaviour was observed in the chewiness, which is equivalent to the energy required for the mastication of the sample before swallowing (Huang *et al.* 2007). The relation between gumminess and chewiness is in agreement with the study of Karaman and Akalın (2013).

The last textural parameter obtained from the TPA analysis is resilience value. There were significant differences

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among the samples ($P < 0.05$). The C1 sample had the highest resilience value and that of the C4 was found as the lowest. As also seen from Table 2, the resilience values increased during the ripening period ($P < 0.05$).

To summarise the textural properties, which are very important for the preference of the consumers, the textural parameters changed depending on the cheese type and ripening time. As seen from the results of the present and previous studies, determination of an accurate relationship between the textural parameters and the physicochemical properties of the cheese samples is very difficult as cheese is a very complex material, which is composed of different compounds (protein, fat, ash) in terms of amount and type (Eroglu *et al.* 2015). In addition, the proteolysis, lipolysis and glycolysis occurring during ripening are also factors affecting the composition and texture of the cheese samples. The establishment of the cause and effect relationship is very difficult since the textural parameters are affected by many factors simultaneously.

Table 3 Fatty acid composition (%) range of the Kashar cheese samples at beginning (0th day) and end (90th day) of the storage period

	Results of present study (%)		*Previous studies (%)*	
Fatty acid	*0th day*	*90th day*	*Fresh (%)*	*Old (%)*
$C_{4:0}$	1.13–1.60	1.93–3.02	1.74–3.91[b,c]	1.84–10.9[a,b,d]
$C_{6:0}$	1.09–1.56	1.53–1.88	1.52–2.10[b,c]	1.50–14.7[a,b,d]
$C_{8:0}$	0.79–1.11	0.94–1.08	0.95–1.92[b,c]	0.92–12.8[a,b,d]
$C_{10:0}$	1.87–2.72	2.10–2.28	2.26–3.04[b,c]	2.90–26.2[a,b,d]
$C_{12:0}$	2.49–3.45	2.46–2.71	2.76–3.30[b,c]	3.20–26.6[a,b,d]
$C_{14:0}$	11.0–11.5	11.1–11.4	11.4–11.8[b,c]	9.31–48.2[a,b,d]
$C_{14:1}$	0.92–1.19	0.95–1.01		0.22–1.06[a]
$C_{15:0}$	1.78–1.94	1.73–1.91	1.23–1.27[c]	0.95–1.54[a]
$C_{16:0}$	35.7–38.1	35.0–36.6	30.9–35.0[b,c]	13.0–30.4[a,b,d]
$C_{16:1}$	1.67–1.81	1.21–1.72		0.64–1.40[a]
$C_{18:0}$	11.8–12.9	10.6–12.3	8.52–10.1[b,c]	9.2–13.1[a,b]
$C_{18:1\ cis}$	23.2–28.0	25.2–27.2	21.3–22.5[b,c]	12.9–29.4[a,b,d]

[a]Kinik *et al.* 2005; [b]Akalin *et al.* 1998; [c]Temiz 2010; [d]Güler 2005.

Fatty acid composition of the fresh Kashar cheese samples

Table 3 presents the fatty acid composition of the fats extracted from the Kashar cheese samples at the 0th day and 90th day of the ripening period. As can be seen from Table 3, fatty acid composition of the Kashar cheese samples ranged in very small limits, which might result from geographical origin of the milk processed to cheese, seasonal variations, initial fatty acid content of the milk, type of the starter cultures, production and ripening process (Werner *et al.* 1992; O'Shea *et al.* 1998). Major fatty acid composition of the fat samples extracted from Kashar cheese samples was found as C16:0 which was followed by C18:1, C18:0 and C14:0, respectively, which is in agreement with the study of Cardak *et al.* (2003). Initial saturated and monounsaturated fatty acid composition of the fats obtained from the cheese samples was in the range of 69.12–73.56% and 26.2–30.6%, respectively. As can be seen from Table 3, generally the fatty acid composition of the samples changed during ripening. Among the saturated fatty acids, as the C16:0 and C18:0 fatty acids decreased, C4:0, C6:0 and C18:1 fatty acid content increased during ripening time. The increase in C4:0, C6:0, C8:0 and C10:0 fatty acid composition during ripening should be explained by the fact that lipolysis of milk fat and breakdown of amino acids could result in formation of fatty acids including four or more carbons (Urbach 1993). The change in fatty acid composition might result from the lipolysis occurred during ripening, in which longer free fatty acid chains are converted to lower ones and methyl ketones (Caglar and Cakmakci 1998). Kinik *et al.* (2005), Akalin *et al.* (1998), Temiz (2010) and Güler (2005) studied the fatty acid composition of different fresh and old Kashar cheese samples. Generally, fatty acid composition of old and fresh cheese samples was found as very different.

Volatile composition of the Kashar cheese samples

Observation of change in volatile compound concentration responsible for flavour and taste of cheeses during ripening is very important for the Kashar producers to determine ripening period and ripening condition. Series of volatile and nonvolatile compounds are formed during ripening due to the breakdown of milk proteins, fat, lactose and citrate occurred by microbial, enzymatic and chemical reactions (Engels *et al*. 1997). The volatile composition range of the fresh Kashar cheese samples at the beginning and end of the ripening time is shown in Table 4. In total, 79 different volatile compounds classified as acid, alcohol, ester, ketone, aldehyde and other compounds were indentified. The volatiles include 12 different acids, 16 alcohols, 15 esters, 11 ketones, 6 aldehydes and 19 other compounds. When comparing volatile compound composition of the fresh Kashar cheese samples with aged ones, many different compounds were reported in previous study (Hayaloglu 2009).

As can be seen from Table 4, initial acid concentration of the fresh Kashar cheese samples was in the range of 12.5–32.2%. In addition to contribution of these compounds to the aroma of cheeses, acids are important in terms of precursors of methyl ketones, alcohols, esters and lactones (McSweeney and Sousa 2000). Butyric and acetic acids are the main acids in the all samples, which is consistent with the study of Yilmaz *et al.* (2005). The volatile acid composition of the samples at the 90th day changed between 34.8% and 61.1%, indicating that the acid concentration

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increased during ripening. Butyric acid, 3-methyl pentanoic acid and acetic acid, concentration of which increased during ripening, were the principal acids at the end of the ripening time. Butyric acid has a rancid taste and it is mainly formed by lipolysis (McSweeney and Sousa 2000). As reported in Table 4, it was seen that acetic and butyric acid concentration of aged (old) Kashar cheese was found as higher than that of fresh Kashar cheese samples, which is very important for aroma of the cheese samples as they can play a significant role in flavour (Urbach 1997). Propionic acid amount of aged Kashar cheese determined in the study of Hayaloglu (2009) was found as higher than that of the fresh ones. High concentration of it caused sweet flavour in cheeses (Steffen *et al.* 1987).

Alcohol concentration of the fresh Kashar cheese samples at the beginning and end of the ripening time changed between 1.56–25.8% and 4.85–8.68%, respectively. As seen from Table 4, there are substantial differences among initial alcohol concentration of the Kashar cheese samples, which might be related to the production process especially pasteurisation affecting the formation of alcohols in cheese. Among the alcohols found in cheese samples, ethanol is the main alcohol, mainly formed by fermentation of lactose and catabolism of Ala (Hayaloglu *et al.* 2007). Ethanol amount in old Kashar cheese sample was found as higher than that of the fresh ones.

Another volatile compound observed in cheese samples are esters that contribute to fruity flavour in cheese (Hayaloglu *et al.* 2007). The ester compositions of the fresh Kashar cheese samples at the beginning and end of the ripening time varied between 2.43–27.2% and 8.49–21.1%, respectively. Ester composition of the cheese samples changed during ripening. As seen from Table 4, many different ester compounds found in the cheeses and they changed during ripening. Esters are formed via esterification, the formation of esters from alcohols and carboxylic acids (Liu *et al.* 2004), and alcoholysis reactions (Hayaloglu *et al.* 2007). Therefore, composition of the milks and reaction conditions such as pH might be an important factor affecting the ester compounds formation. As comparing the findings of Hayaloglu (2009) related to old Kashar cheese samples, it can be concluded that ethyl ester concentration of old Kashar cheese samples was determined as generally higher than that of fresh Kashar cheese samples.

Eleven different ketones were observed in the fresh Kashar cheese samples. The initial and final ketone concentration of the cheeses ranged between 18.7–24.3% and 12.3–20.7%, respectively. Ketone compounds are formed by enzymic oxidation of free fatty acids to β-ketoacids, followed by decarboxylation to ketones (McSweeney and Sousa 2000). As seen from Table 4, ketone composition changed during ripening. 2-pentanone (in C1, C2, C3), 2-heptanone (in all cheese samples), 2-butanone (in C1), 3-hydroxy 2-butanone (in all samples) and acetone (in C1) were the most abundant compounds at the end of the ripening. Diacetyl in mature Kashar cheese was reported by Hayaloglu (2009) as higher than that of fresh Kashar cheese samples. When considering the significance of diacetyl for cheese flavour, mature Kashar cheese could be preferred.

Six different aldehyde compounds were found in the Kashar cheese samples: isovaleraldehyde, benzaldehyde, hexanal, nonanal, pentanal and decanal. The concentration of the aldehyde compounds in Kashar cheese was in the range from N.D. to 6.54% at the beginning of the ripening. Aldehydes originate from the degradation of amino acids (Engels *et al.* 1997). Pentanal was the most abundant aldehyde compound found in the cheese samples. As seen from Table 4, the aldehyde concentration decreased, which was expected since aldehydes do not accumulate in cheeses due to their rapid transformation to alcohols or acids (Hayaloglu *et al.* 2007). 2-methyl propanal, 2-methyl butanal and 3-methyl butanal were observed in different old Kashar cheese samples at different levels (Hayaloglu 2009); however, they were not observed in the fresh ones.

The last class of the volatile compounds was named as other compounds, which include aromatic compounds (phenol) and terpenes (2-pentene, limonene, cymene, xylene). Limonene is the most abundant terpene found in the cheese samples at beginning of the ripening period but was not observed in any of the cheese samples at the end of the ripening period. Type and amount of the terpenes depend mainly on the characteristics of the milk processed to cheese as terpenes in milk originate from plants found in the feed mixture (Curioni and Bosset 2002).

CONCLUSIONS

In this study, textural and physicochemical properties (dry matter, protein, fat and fatty acid content, volatile compound composition) of the fresh Kashar cheese samples were determined. According to the results of this study, all of the properties mentioned were affected by chemical and enzymatic reactions such as lipolysis or proteolysis which occurred during ripening period. Among textural properties, hardness, gumminess and chewiness values increased with ripening period. Observation of the dependency of those properties or compositions on the ripening time is important for the determination of the optimum ripening period, which is crucial for the dairy industry. Further studies need to be carried out to correlate those parameters with the sensory properties of Kashar cheese to determine the optimum ripening time. Change in these parameters as a function of ripening period should be taken into consideration by every producer to determine the ripening period of both fresh and aged Kashar cheese samples since many factors affect these characteristics simultaneously.

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Table 4 Volatile composition range of the Kashar cheese samples (% peak area) at the beginning and end of the ripening period

		Results of present study		*Previous studies*	
Compounds		*0th day*	*90th day*	*Fresh*	*Old*
Acid					
1	Acetic acid	1.18–7.90	4.78–14.3		18.7–19.3[a]
2	Propionic acid	N.D.	N.D.–0.15		2.23–9.68[a]
3	Isobutyric acid	N.D.–1.18	N.D.–0.94		0.89–6.51[a]
4	Butyric acid	4.48–16.2	11.2–29.9		23.1–31.1[a]
5	Isopentanoic acid	N.D.–0.5	N.D.–2.78		2.66–11.7[a]
6	Pentanoic acid	N.D.	N.D.–0.15		2.74–3.41[a]
7	3-methyl pentanoic acid	N.D.–2.91	5.88–19.3		
8	Nonanoic acid	N.D.–9.82	1.12–8.20		
9	Lauric acid	N.D.–0.47	N.D.–2.40		
10	Myristic acid	N.D.	N.D.–0.17		
11	Heptanoic acid	N.D.	N.D.–0.10		
12	Cinnamic acid	1.01–3.25	N.D.		
	Total Acid	12.5–32.2	34.8–61.1	11.6–22.9[b]	
Alcohol					
1	Ethanol	N.D.–7.19	1.82–6.26		11.4–32.6[a]
2	Butanol	N.D.	N.D. –1.55		2.70–4.11[a]
3	Isobutyl alcohol	0.59–0.92	N.D.–1.19		
4	Pentanol	N.D.–17.9	N.D.–2.29		2.25–4.64[a]
5	Octanol	N.D.–0.36	N.D.–0.25		0.47–0.72[a]
6	Phenyl ethyl alcohol	N.D.–0.22	N.D.–0.28		0.99–1.87[a]
7	2,3-Butanediol	1.86–2.96	1.65–9.64		
8	2.6-Dimethyl-4-heptanol	N.D.	N.D.–0.46		
9	2-Butoxyethanol	N.D.–0.36	N.D.–0.10		1.14–2.88[a]
10	Hexanol	N.D.–0.29	N.D.		1.16–5.26[a]
11	2-Ethyl-1-hexanol	0.08–0.51	N.D.		1.61–4.01[a]
12	Furfuryl alcohol	N.D.–0.16	N.D.		
13	Isopropenyl alcohol	N.D.	N.D.–2.40		
14	4-methoxy-1-butanol	N.D.–0.68	N.D.		
15	Benzyl alcohol	N.D.–0.10	N.D.		0.43–1.38[a]
	Total Alcohol	1.56–25.8	4.85–8.68	31.8–55.4[b]	
Esters					
1	Ethyl butanoate	N.D.–3.53	N.D.–3.68		6.49–8.79[a]
2	Isoamyl formate	N.D.–17.97	5.52–18.9		1.78–5.32[a]
3	Ethyl hexanoate	0.32–1.92	N.D.–1.36		4.67–6.52[a]
4	Ethyl octanoate	N.D.–0.23	N.D.–0.08		1.44–1.75[a]
5	Ethyl acetate	N.D. –4.18	N.D.–2.21		7.50–35.7[a]
6	Ethyl laurate	N.D.	N.D.–0.32		
7	Methyl pentaonate	N.D.–0.69	N.D.–0.30		
8	Ethyl myristate	N.D.	N.D.–0.11		
9	Methyl heptanoate	N.D.–0.45	N.D.		
10	Ethyl decanoate	N.D. –0.34	N.D.		
11	Methyl butanoate	N.D.–3.97	N.D.		
12	Methyl laurate	N.D.–0.70	N.D.		
13	Methyl hexanoate	N.D.–3.63	N.D.		
14	Sabinyl acetate	N.D.–3.81	N.D.		
15	Hexyl isobutanoate	N.D. –0.34	N.D.		
	Total Esters	2.43–27.2	8.49–21.1	5.01–14.7[b]	

(continued)

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Table 4 (Continued)

		Results of present study		*Previous studies*	
Compounds		*0th day*	*90th day*	*Fresh*	*Old*
Ketones					
1	2-Pentanone	N.D.–10.2	N.D.–10.6		3.2–29[a]
2	2-Heptanone	2.29–8.36	2.04–4.26		3.6–16[a]
3	2-Butanone	N.D.	N.D.–4.16		5.7–44[a]
4	Nonanone	0.65–1.32	N.D.–0.31		1.6–4.6[a]
5	3-hydroxy 2-butanone	2.20–8.28	4.40–5.86		2.6–26[a]
6	Acetone	N.D.–6.38	N.D.–2.76		
7	2.2-Dymethyl-1.3-dioxane-4.6-dione	N.D.	N.D.–2.48		
8	Methyl ethyl ketone	N.D.–2.48	N.D.		
9	6,10-dimehyl 5,9-dodecadien-2-one	N.D.–0.71	N.D.		
10	Diacetyl	N.D.–2.01	N.D.–3.94		
11	6-Methyl-5-hepten-2-one	N.D.–0.36	N.D.		
	Total Ketone	18.7–24.3	12.3–20.7	15.5–37.1[b]	
Aldehydes					
1	Isovaleraldehyde	N.D.	N.D. –2.87		
2	Benzaldehyde	N.D.–0.64	N.D.–0.18		0.9–1.9[a]
3	Hexanal	N.D.–0.98	N.D.		
4	Nonanal	N.D.–0.25	N.D.		
5	Pentanal	N.D.–6.37	N.D.		
6	Decanal	N.D.–0.15	N.D.		
	Total Aldehyde	0.91–6.54	N.D.–3.05	2.49–34.0[b]	
Other Compounds					
1	2-Pentene	N.D.	N.D. –1.57		
2	Benzothiazole	1.29–1.62	0.69–5.32		
3	Ethane	N.D.	N.D. –0.52		
4	1.3 dimethyl benzene	N.D.–2.30	N.D. –0.84		
5	Chloroform	N.D.–3.63	N.D.		1.2–1.4[a]
6	Ether	N.D.–0.47	N.D.		
7	Limonene	2.91–9.36	N.D.		1.0–1.8[a]
8	Cymene	N.D.–1.93	N.D.		1.6–2.8[a]
9	1.4-dichloro benzene	N.D.–0.70	N.D.		
10	1.3-dichloro benzene	N.D.–0.26	N.D.		
11	Pyrrolidine	N.D.–4.36	N.D. –3.36		0.3–0.4[a]
12	Deuteropropane	N.D.–1.86	N.D.		
13	Methoxy-phenyl oxime	N.D.–0.77	N.D.		
14	Ethyl benzene	N.D.–1.27	N.D.		1.5–2.6[a]
15	Xylene	N.D.–0.86	N.D.		
16	Trimethyl benzene	N.D.–0.30	N.D.		
17	4-octadecyl morpholine	N.D.–0.22	N.D.		
18	Bromodichloromethane	N.D.–0.86	N.D.		
19	1-propene-1-thiol	N.D.–0.29	N.D.		
20	Phenol	N.D.–0.25	N.D.		
	Total Other Compounds	6.89–22.0	2.65–11.7		

[a]Hayaloglu (2009). The results were expressed as peak area/100 000; [b]Temizkan *et al.* 2014. Results were expressed as μg/100 g cheese.

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