

Relation between a temperature-sweep dynamic shear test and functional properties of cheeses

Jean-Michel REPARET^{a*}, Yolande NOËL^b

^a École Nationale d'Industrie Laitière de Besançon-Mamirolle, 25610 Mamirolle, France

^b Laboratoire de Technologie et Analyses Laitières, INRA, BP 89, 39801 Poligny, France

Received 2 October 2001 – Accepted 17 March 2003

Published online 22 May 2003

Abstract – Relationships between functional properties and variables obtained with a temperature-sweep dynamic shear test of twenty different varieties of cheese were evaluated. Flowability was assessed using a modified Schreiber test and stretchability by a method involving vertical traction. The dynamic shear test provides a typical profile for each cheese variety, a profile that might be related to the different cheese-making processes, and especially to the heating and mechanical treatments influencing further the response of the cheese matrix by itself in conditions of cooking use. Principal component analysis (PCA) gave evidence of complex and non-linear relationships between the characteristics of the dynamic shear profile and the stretchability or meltability indices. A significant, but weak, link between pH and stretchability was observed. PCA separated cheeses by their cheese-making technology. The effects of the technological factors for the different types of cheese varieties on their functional properties suggest the possibility either of predicting these properties or of changing the processes in order to create new products with new functionalities.

Functional property / dynamic shear test / Schreiber test / stretchability test / cheese

Résumé – **Relation entre un test de cisaillement dynamique effectué en balayage température et les propriétés fonctionnelles des fromages.** Les relations entre les propriétés fonctionnelles et les variables obtenues par un test de cisaillement dynamique effectué en balayage température ont été évaluées. L'étalement à chaud a été évalué à l'aide d'un test de Schreiber modifié et l'aptitude au filant par une méthode utilisant une traction verticale. Le test de cisaillement dynamique donne un profil caractéristique de chaque variété de fromage, profil qui peut être mis en relation avec les différents procédés de fabrication, notamment avec les traitements thermiques et mécaniques imposés lors de la fabrication dont les effets laisseraient une empreinte dans la matrice fromagère elle-même. Une analyse en composantes principales a mis en évidence des relations complexes et non linéaires entre les variables du test de cisaillement dynamique et les indices d'étalement à chaud et de filant. Une corrélation faible mais significative entre le pH et l'aptitude au filant a été observée. L'analyse en composantes principales a séparé les fromages selon leur technologie de fabrication. Les effets des facteurs technologiques des différentes variétés de fromage sur leurs propriétés fonctionnelles laissent entrevoir la possibilité d'envisager soit de prévoir ces propriétés soit d'orienter les procédés pour créer de nouveaux produits avec de nouvelles fonctionnalités.

Propriété fonctionnelle / test de cisaillement dynamique / test de Schreiber / test d'évaluation du filant / fromage

* Correspondence and reprints

E-mail: jm.reparet@enesad.fr

Present address: ENESAD, Bd O de Serres, 21800 Quétigny, France

1. INTRODUCTION

The role of cheese as an ingredient in the dairy industry has considerably increased during the last few years. Dairy product producers want to adapt the cheese-making process to target products with functional properties for specific culinary applications [14]. Functional properties applied to food ingredients are defined as all non-nutritional properties that influence the behaviour of ingredients in food [10]. Meltability and stretchability are functional properties, which are essential for the use of cheeses in cooking meals.

The dairy industry needs objective methods to assess the melting properties of cheese [4, 7, 22, 23, 29]. These properties are determined by thermal effects of cooking on the cheese structure, resulting in changes in the physico-chemical properties and consequently the rheological or flow properties, which are interdependent [29]. Meltability, a major component of melting properties, is often evaluated in the industry with the Schreiber test [23, 25, 32] and the Arnott test [4, 24]. Stretchability, another component of melting properties, has been assessed manually by using a fork and lifting the cheese from the surface of a pizza [33, 38]. The most serious attempts to measure stretchability with instruments were indirect and empirical [3, 7, 12, 13, 28]. Several authors [21, 22, 33, 35] have attempted to define objective physical magnitudes based on helical viscometry to evaluate meltability and stretchability, but some limits were pointed out.

Dynamic rheological testing, which allows the characterisation of viscoelastic properties of materials [11], has been applied to cheese [2, 26, 36, 39]. As mentioned by Guinee et al. [14], there are few studies reporting on the functional properties of cheeses other than Mozzarella or Cheddar, or even analogue cheeses. Taneya et al. [36] have established the dynamic shear profiles with temperature-sweep over the range $-10\text{ }^{\circ}\text{C}$ to $75\text{ }^{\circ}\text{C}$ from Gouda and Cheddar at different maturation

ages and from two processed cheeses. Ustunol et al. [39] have used a dynamic rheological test with temperature-sweep from 25 to $90\text{ }^{\circ}\text{C}$ to evaluate meltability of Cheddar cheese with varying fat content, and in comparison with the Arnott method. They have found a correlation between the complex modulus and meltability indices. Horne et al. [17] use a dynamic shear test with temperature range from 20 to $80\text{ }^{\circ}\text{C}$ to quantify the viscoelastic properties of a control and Cheddar cheese containing denatured whey protein. They observed significant differences in the values of shear moduli, reflecting the harder, more brittle and crumbly nature of the cheese. Recently, Mounsey and O'Riordan [26] have found a correlation between the maximum $\tan \delta$ values and the meltability indices of imitation cheese. Dynamic rheology may be a useful fundamental method to assess the meltability of cheese products.

This study aimed to evaluate the temperature-sweep dynamic shear test for characterising the functional properties of twenty different cheese varieties, including processed cheeses, and for relating the rheological measurements to meltability and stretchability evaluated with empirical tests. Moreover, the data collection of dynamic shear profiles obtained from a wide range of cheese varieties would support the identification of the most pertinent parameters for characterising functional properties, and can provide references for developing new products.

2. MATERIALS AND METHODS

2.1. The cheeses

Twenty cheese varieties were purchased at a local supermarket. They are listed in Table I with codes used here. They were stored for 24 h at $5\text{ }^{\circ}\text{C}$ prior to testing. Cheeses were selected for their representativeness of the different cheese technologies. Mamirolle is a soft cheese produced

Table 1. Codes of the cheeses, gross composition and proteolysis indices*, stretchability and meltability indices.

Cheeses	Code	pH	DM (%)	Fat (%)	TN (g·kg ⁻¹)	WSN/TN (%)	PTA-SN/TN (%)	Ca/NFDM (%)	Si (mm)	CV% (mm)	Mi 60 °C (mm)	CV% (mm)	Mi 80 °C (mm)	CV% (mm)
Appenzeller	App	6.08	60.5	33.4	2.55	43.2	13.9	3.41	750	-	1.70	3.5	2.76	6.3
Beaufort	Bea	5.74	66.4	35.9	2.71	30.4	12.6	3.50	86	17.6	1.97	4.5	2.31	2.6
Cantal	Can	5.07	57.0	28.2	2.49	35.8	8.1	2.43	65	18.1	1.66	6.3	2.88	4.5
Cheddar	Che	5.22	64.0	37.2	2.42	26.0	7.1	3.29	248	14.0	1.61	8.2	2.17	7.4
Comté	Com	5.74	64.8	43.1	2.83	32.3	7.8	4.77	443	8.9	2.01	2.9	2.30	9.5
Edam	Eda	5.12	55.0	25.6	2.43	27.2	5.9	3.58	17	20.9	2.09	3.4	2.58	6.3
Emmental	Emm	5.65	64.0	32.5	2.98	24.1	8.5	3.74	348	4.0	1.98	5.6	1.88	2.8
Gouda	Gou	5.22	58.7	30.5	2.46	25.7	3.0	3.14	432	8.0	1.98	6.4	2.39	7.2
Miamrolle	Miam	5.54	47.4	22.7	2.30	22.1	4.5	2.45	750	-	1.67	2.3	1.71	5.8
Mimolette	Mim	5.10	54.9	31.0	2.35	24.9	3.5	4.38	14	25.4	2.49	5.1	1.85	3.3
Mozzarella	Moz	6.03	44.0	20.0	2.08	21.3	2.7	2.16	520	7.4	1.23	4.4	1.71	2.9
Parmesan	Par	5.34	65.9	28.7	3.19	51.1	19.8	3.05	21	8.5	1.61	7.6	1.85	8.2
Processed cheese 1	Pr1	5.75	51.8	29.0	1.88	98.8	0.9	2.88	420	14.2	1.97	4.6	2.71	9.1
Processed cheese 2	Pr2	5.68	51.8	25.4	2.00	98.9	1.1	2.71	323	18.4	0.99	4.5	1.28	5.9
Processed cheese 3	Pr3	5.74	48.2	25.2	2.02	90.1	0.8	2.97	550	17.3	2.09	3.5	2.40	5.7
Provolone	Pro	5.15	63.3	34.0	2.41	25.4	6.4	2.52	445	9.1	1.47	8.5	1.98	7.8
Raclette	Rac	5.35	46.6	27.1	2.36	23.3	4.3	3.36	455	12.3	1.65	4.5	2.38	4.6
Reblochon	Reb	5.17	46.0	24.7	1.85	21.2	2.7	2.11	125	23.0	2.15	5.9	2.43	6.3
Swiss Gruyère	Gru	5.55	66.0	33.5	2.75	24.7	9.6	2.61	750	-	1.75	7.1	2.33	6.8
Tomme de Savoie	Tds	5.35	60.0	32.0	2.43	27.4	6.1	2.28	136	15.0	1.56	4.7	1.90	3.7

* See Materials and methods, for the meaning of the codes of the variables. Mi: Meltability index evaluated with Schreiber test at 60 °C (Mi60) and at 80 °C (Mi80); Si: stretchability index.

at the École nationale d'industrie laitière de Mamirolle. It was selected for its textural homogeneity, to provide a typical temperature-sweep dynamic shear profile.

2.2. Physico-chemical analyses

The following analyses were performed: dry matter DM [18] and fat [15] expressed as percent of cheese mass and calcium [30] expressed as percent of non-fat in dry matter (NFDM). The pH of cheeses was determined with a penetrometric electrode (Grosseron, Saint-Herblain, France). Total nitrogen (TN), soluble nitrogen (SN) and phosphotungstic acid soluble nitrogen (PTA-N) were measured by the Kjeldhal method [19]. Soluble fractions were expressed in percent of TN.

2.3. Schreiber test

Meltability of cheeses was determined by using the Schreiber test as described by Kosikowski and Mistry [23]. Cheese samples were removed from refrigerated storage and stored at room temperature for 1 h. Then they were cut into cylindrical specimens (27 mm in diameter and 27 mm in height) with a cork borer and a knife. The specimen, placed in the middle of a special glass dish, was heated for 6 min in a laboratory convection oven for pizza cooking regulated at 270 °C.

In a preliminary trial, the temperature of cheese specimens was measured with a thermocouple plunged into the specimen, in order to evaluate the temperature changes during cooking in the controlled conditions of the test. The temperature change of three different cheese varieties versus heating time in the oven was measured. Two temperature levels were selected, based on the fact that the maximum temperature of the cheese on the top of pizza on the consumer's plate is between 60 and 80 °C. Thus consumers would appreciate functional properties of cheeses preferably around 60 °C. Temperatures above 75 °C would result in a burning of the mouth [8].

The times to reach 60 and 80 °C were almost the same for all of the cheese varieties, so we decided to evaluate the diameter changes of the cheese specimens at these predetermined cooking times. The cheese specimens were taken out of the oven at these times, then cooled to room temperature for 30 min before reading the diameter on a graduated scale on the bottom of the glass dish. Two meltability indices were calculated by averaging the measured diameters of the melted cylinder specimen (unit, mm) at 60 and 80 °C. They were coded Mi60 and Mi80.

2.4. Stretchability test

Stretchability was evaluated by using a Universal Testing Machine (Adamel Lhomargy, Ivry-sur-Seine, France) fitted with a measuring cell, i.e. a rod equipped with 8 square-tipped blades at the end, specially designed and manufactured (Fig. 1). The machine moved the cell up at a speed of 500 mm·min⁻¹. The cheese was removed from refrigerated storage (5 °C) and rapidly cut into small pieces with a knife, then grated in a blender (Osterizer Pulsematic, Oster Corp., Milwaukee, WI, USA) at an ambient temperature. The size of the cheese particles was approximately 5 mm. Two-hundred grams of grated cheese was put in a large diameter glass container, which was plunged into a water-bath previously regulated at 80 °C (± 0.1 °C). A thermocouple was used to control the temperature of the grated cheese. When the cheese sample reached 60 °C, the measuring cell was placed 4 cm above the container bottom then the test started immediately, by pulling the measuring cell vertically, stretching the cheese specimen. A stretchability index was evaluated as the distance covered by the measuring cell until all the cheese strings were broken. Five samples were tested from each cheese.

The temperature of 60 °C was selected to perform the stretchability test after preliminary trials, for the same reasons as described for meltability. This temperature has already been used in previous studies [1, 21].

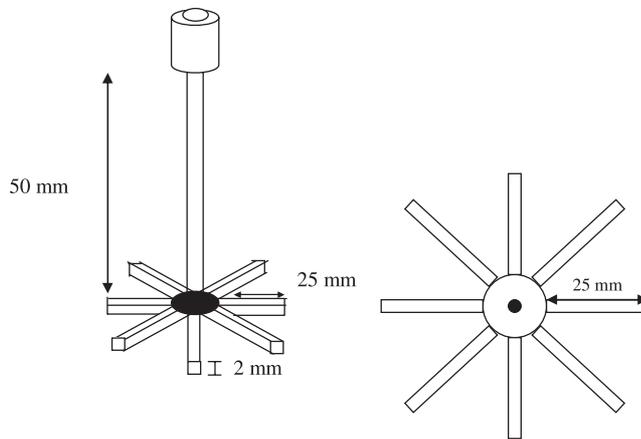


Figure 1. Schematic design of the measuring cell for stretchability test.

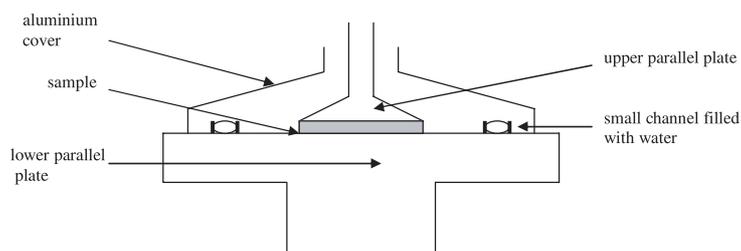


Figure 2. Schematic design of the measuring cell for dynamic shear testing.

2.5. Rheological dynamic shear test

The dynamic shear test was performed using a controlled-stress rheometer (Carri-Med CSL 100, distributed by T.A. Instruments, St-Quentin-en-Yvelines, France) with a parallel plate apparatus (2 cm radius). Data were collected and rheological parameters calculated by using a computer with Carri-Med 50 software. Temperature sweeps were performed from 20 to 90 °C at 1.5 °C·min⁻¹. The test combines low shear with thermal treatments. Measurements were performed at a constant frequency of 2 Hz and a constant strain of 2%. In duplicate, these conditions ensured the samples were tested within the linear viscoelastic range. To prevent dehydration of the sample during the test, 2 mL of water

was put in a special small circular channel near the sample without any contact with it in order to create a special atmosphere. An aluminium cover was put over the entire plate (Fig. 2). The cheeses were sampled at 5 °C by using a meat slicer and a cork borer in order to obtain discs (diameter, 20 mm; height, 1 mm). The height was selected after preliminary trials showing a better reproducibility than a height of 2 mm. The cheese was placed manually on the flat surface, which was then fixed onto the stress-controlled apparatus. Heating of the specimen was performed through the lower plate by Peltier effect.

Preliminary tests confirmed that there was no need to prevent slippage, as already pointed out by Horne et al. [17]. The rheological parameters: G' , the elastic modulus;

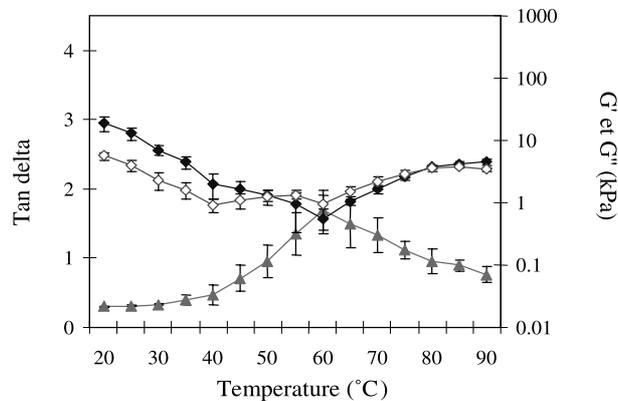


Figure 3. Temperature-sweep dynamic shear profile obtained with Mamirolle cheese ($n=10$); with \blacktriangle $\tan \delta$, \blacklozenge G' , and \circ G'' .

G'' , the viscous modulus; and the loss tangent, $\tan \delta$ (δ), were evaluated.

2.6. Statistical analyses

Principal component analyses (PCA) were carried out with SPAD 3.2 software (Centre International de la Statistique et de l'Informatique Appliquée, Saint Mandé, France).

3. RESULTS AND DISCUSSION

3.1. A typical temperature-sweep dynamic shear profile

Figure 3 shows a typical dynamic shear profile obtained with a soft Mamirolle cheese of 2 weeks as an example. Changes in G' and G'' are represented on a logarithmic scale versus temperature, and $\tan \delta$ on a normal scale. The components G' and G'' decreased steadily between 20 and 65 °C, indicating a solid-like behaviour. Below 50 °C, the elastic component G' values were higher than G'' values. Between 50 and 60 °C, the viscous component G'' became higher than the elastic one, involving an increase in $\tan \delta$ indicating a liquid-like behaviour. The two curves G' and G'' crossed approximately at 50 °C, the crossing point corresponding to the particular value '1' of $\tan \delta$. Above 65 °C, both the

elastic and viscous components increased gradually. Interestingly, the two curves G' and G'' crossed again near 80 °C.

Temperature increase between 20 and 50 °C induced fat liquefaction [24], resulting in both G' and G'' decrease which expressed the melting of cheese. While decreasing, G' remained over G'' , which can be explained by the mechanical resistance of the protein network while cheese fat melts progressively. The lipid fraction near the membrane of a fat globule has a higher melting point than the internal fraction. Thus we can assume that there is a progressive melting of the different lipid fractions in fat globules up to 30 and 40 °C, where the triglycerides of cheese are liquids. The decrease in G' indicated a weakening of the cheese structure, an effect which might be due in large part to liquefaction of the fat phase, fully liquid at about 40 °C [14]. Coalescence of fat globules would follow, reducing the structural support of fat globules to the surrounding protein network, as observed by scanning electron microscopy by Lefevre et al. [24] on Cheddar. Thus, the limited changes in the elastic and viscous components in the range 40–50 °C could be interpreted as a mechanical resistance of the cheese network in relation to the thermal stability of the proteins, while water losses would be prevented by the measuring cell. The cheese protein network is the solid phase,

which entraps fat globules and water. The thermal stability of the protein network results from hydrophobic interactions and polar links determining the secondary and tertiary structures of the proteins. Increasing sample temperature causes both storage and loss moduli decrease as the structure breaks down and the sample becomes less viscoelastic [17], the structure of protein chains loses its mechanical resistance and has a greater conformational mobility, promoting the viscous component [2, 17]. Above 65 °C, casein started to aggregate upon heating, which represents a structural reorganisation, a process that would lead to an increase in the elastic component of the casein matrix and less conformational mobility. For temperatures over 65 °C the phenomenon of aggregation of casein is more developed than the agitation of the network. Thus, 60–65 °C represents a change of phase between agitation and aggregation. The higher values of the viscous component G'' , following the crossing point between G' and G'' , would express these phenomena. After the melting of cheese fat, the changes in protein-protein interactions and water-protein interactions contribute to the collapse of the protein network [24]. Structural rearrangements of proteins due to thermal treatment during testing follow complex processes for caseins and whey proteins, partly described by Holt [16]. At higher temperatures, the high variability of the data might be explained partly by the instability of the product and partly by the difficult measuring conditions. Nevertheless, the increase in $G'(t)$ can be interpreted as a loss of protein structure with a polymerisation and compaction of the protein network due to thermal aggregation [9] in relation to a loss of bound water, which normally contributes to maintaining the original protein structure.

3.2. Dynamic shear profiles of the different cheese varieties

Different rheological profiles are presented in Figure 4. They were selected in

order to represent the diversity of the profiles observed with the 20 cheese varieties tested. The levels of G' and G'' before and after the crossing point, their relative levels, the level of $\tan \delta$ and the temperatures related to its maximum and its particular '1' value characterise different patterns, indicating that each cheese variety has its own rheological signature.

The hard Comté cheese had a dynamic shear-temperature profile of G' , and also G'' , like a 'golf club'. The temperature for $\tan \delta = 1$ occurred around 60 °C, and close to 75 °C for the maximum value of $\tan \delta$, which was higher than 3.

The hard Cheddar cheese had a dynamic-shear temperature profile of G' and G'' like a 'tea-spoon'. The temperature for $\tan \delta = 1$ occurred around 50–55 °C, while it was 70 °C for the maximum value of $\tan \delta$, which reached a peak lower than 3. This temperature profile, $\log G$ versus T , is quite similar to those obtained by Horne et al. [17] with the same cheese, but we could not compare G' and G'' values because the gap used was not the same.

The semi-hard Raclette cheese had a dynamic-shear temperature profile of G' and G'' with rather pronounced variations like a 'ladle', with two crossing-points, associated with $\tan \delta = 1$. The first one occurred around 50 °C and the second one between 80–85 °C. The value of the maximum of $\tan \delta$ was lower than 2. The second crossing-point would indicate a thermal reorganisation of the cheese matrix at high temperatures. Differences in the level of G' and G'' between our work and that of Horne et al. [17] might be explained by the differences in the gap used.

A very hard Parmesan cheese was the only variety with the elastic component G' always higher than the viscous component, resulting in values of $\tan \delta$ always lower than 1, without any crossing-point. Moreover, the values of G' and G'' were the highest among all the studied cheeses.

These results suggest that there was no aggregation of casein upon heating during

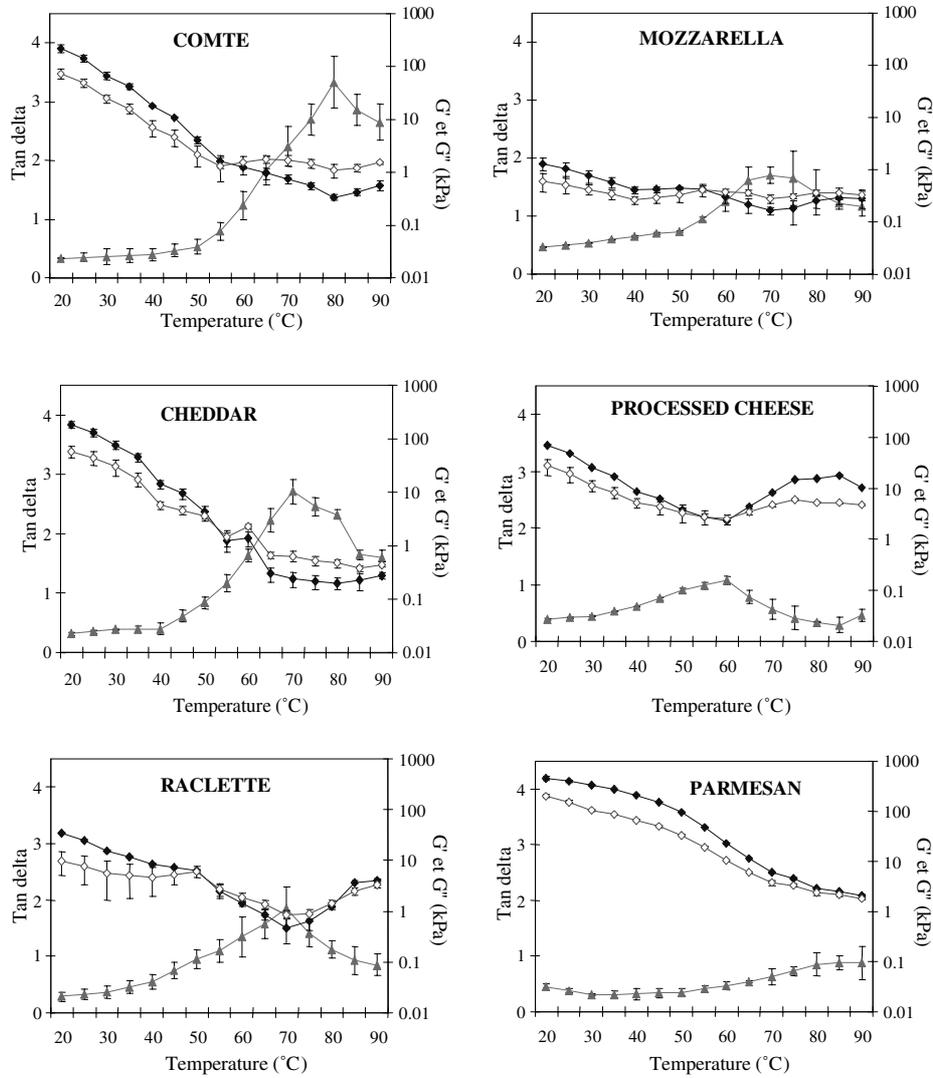


Figure 4. Examples of temperature-sweep dynamic shear profiles obtained from different cheese varieties; with \triangle $\tan \delta$, \bullet G' , and \circ G'' .

the test, as previously described for the other cheeses. This could be explained by the cheese-making and ripening conditions of Parmesan: the strong cooking stage with a rapid temperature increase from clotting temperature to 56 °C within 5 min might change the properties of the protein matrix,

properties which could be expressed during the rheological test. Moreover, Parmesan is submitted to a long ripening time inducing a high level of proteolysis, as shown in Table I, which can contribute to the rheological response, especially at a high temperature.

Parmesan cheese is made from partially skimmed milk, resulting in low fat content (Tab. I), which might reduce the melting of the cheese. As described for Mozzarella by Tunick et al. [37]: when fat content increases, softness and meltability increase.

The overall levels of G' and G'' profiles decreased from Parmesan, Comté, Cheddar to Raclette. In the same cheese order, except Parmesan which had a particular profile, the temperature value for $\tan \delta = 1$ decreased, as well as the value of the maximum of $\tan \delta$. The structural reorganisation, which represents the thermal change from mobility to aggregation of casein appeared more and more pronounced from Parmesan to Raclette, through Comté and Cheddar, with temperature increase during the test. This could be related to the structural properties of the cheese matrix, protein-protein interactions and water-protein interactions and thus to the cheese-making conditions, especially to the cooking treatment during the cheese-making process [9]. Parmesan undergoes the strongest heating stage during the cheese-making process, followed by Comté, while Cheddar is a medium cooked cheese, and Raclette an uncooked cheese. The effects of the heating treatment during the cheese-making process would result in the structural properties of the cheese matrix. Different models based on milk protein, homogenised milk or milk fat systems explain interaction between protein-protein [6, 9], water and protein [5], and the distribution of fat globules entrapped in the protein network [40]. Those interactions would determine the rheological behaviour during heating of the final cheese, and thus its functional properties. In particular, the temperature of the melting of the cheese matrix appears to be strongly dependent on the technological, especially cooking, history of the cheese matrix. We can conclude that the temperature range between the two particular values of $\tan \delta$ (value '1' and maximum value) is highly critical regarding the structural changes related to functional properties.

The variations of G' and G'' with temperature for Mozzarella were much less pronounced than with the previously discussed cheeses, with smaller amplitudes resulting in lower values of $\tan \delta$. This particular behaviour could be related to the structural properties determined by the specific mechanical and cooking treatment during the cheese-making process: protein fibres were aligned when the cheese was stretched and moulded [27]. The casein in the low-moisture Mozzarella was in the form of longitudinally-aligned fibres with entrapped fat columns, consisting of discrete and coalesced fat globules, of similar orientation [13].

The processed cheese had a special profile with an increase in G' and G'' after the unique crossing-point, corresponding to the maximum value of $\tan \delta$ observed at 60 °C. The elastic component G' was always higher than the viscous component G'' . The pattern of viscoelastic changes with temperature is very different than for the other cheeses. We can assume that the processed cheese matrix had very specific structural properties, resulting from the mixture of different ingredients (milk powder, emulsifying salts and milk fat ...) during the process. Poor meltability of processed cheese has been associated with a high degree of fat emulsification, as evidenced by the size distribution of fat globules determined by electron microscopy [34].

Taneya et al. [36] have shown that softening properties of processed cheese can be explained by the distribution of casein in the processed cheese: in soft processed cheeses which have a lower softening point than hard processed cheese, caseins are uniformly dispersed, but those in the hard type are partially in a pearl necklace structure.

3.3. Meltability and stretchability of the cheeses

Table I gives the values of the meltability and stretchability indices obtained with the different cheese varieties. The stretchability

indices ranged from 14 to 750 mm, with the lowest value observed with Mimolette and the highest value with Appenzeller, Swiss Gruyère and Mamirolle. The coefficients of variation were high, ranging from 4.0 to 25.4%. Meltability indices ranged from 0.99 to 2.49 cm at 60 °C, values measured with the processed cheese Pr2 and Mimolette, respectively. They ranged from 1.28 to 2.88 at 80 °C, with Pr2 and Cantal, respectively. Coefficients of variation ranged from 1.28 to 2.88% at 60 °C, and from 2.6 to 9.5% at 80 °C.

3.4. Multivariate analysis

A PCA was performed on the data set including the rheological, physico-chemical data and the indices of meltability and stretchability. The Parmesan-type cheese was excluded from the data set due to its particular behaviour, resulting in missing data for the temperature associated with $\tan \delta = 1$. The first three principal components represent up to 70% of the total variability of the data set (Fig. 5a, b). The first principal component (PC1) was explained mainly by all the physico-chemical variables except pH, and the variables $\tan \delta$ and G' at low temperatures. The meltability indices weakly explained the PC1. Interestingly, they were weakly related to DM, fat and Ca, and even to the proteolysis indices. The second principal component (PC2) was mainly determined by the rheological variables G' and $\tan \delta$ at temperatures between 55 and 65 °C. The rheological variables measured at high temperatures contributed equally to both PC1 and PC2. Two axes of interpretation can complete the analysis of the PC1-PC2 map. The first one opposed the variable G'_{50} to $\tan \delta_{50}$, pH, the stretchability index and $\tan \delta_{30}$. The second one included the variables $\tan \delta$ and G' for the high temperatures 70 and 85 °C.

PC3 represents 12% of the total variability. PC3 was determined by multiple weak contribution of the different rheological variables measured either at low or

high temperatures, as well as the physico-chemical variables. PC3 was essentially determined by the meltability indices and Si in opposition. A weak correlation ($r = -0.36$) was observed between Si and Mi60. This opposition would indicate that a stretchable cheese would have a weaker aptitude to melt, as already observed by the professionals. The results do not confirm those obtained by Richoux et al. [31] who observed a strong correlation between stretchability and meltability indices for one cheese variety: Emmental. A significant correlation ($r = 0.56$) was observed between pH and Si. Kimura et al. [20] have already pointed out the relationship between pH and stretchability of the Japanese String cheese variety. The stretchability index contributed weakly to PC3, as well as G' at 65 °C.

In the PC1/PC2 diagram, the axis 1 separates the cheeses into two main clusters, but the processed cheese Pr2 is isolated in the upper right corner of the diagram. The first cluster gathers in quite a close manner Mozzarella, Mamirolle, Raclette, Reblochon and the two processed cheeses Pr1 and Pr3. All the other cheeses are located in a second loose cluster on the left side of the diagram, where they are mainly separated along PC2. Nevertheless, a sub-cluster with Comté, Cantal, Swiss Gruyère and Emmental was noticeable on this diagram. The PC2/PC3 diagram gives evidence of a much better separation of the cheeses into each of the clusters previously described. Even if PC3 represented a lower level of variability, it contributed well to this separation. We can attribute this contribution especially to the functional properties of the cheeses.

The relationships between the rheological variables measured with the dynamic shear test and the indices of meltability and stretchability appeared to be complex and non-linear. This would explain why no significant correlation, i.e. linear relation, was noticed in the literature. These relationships would need to be explored further by

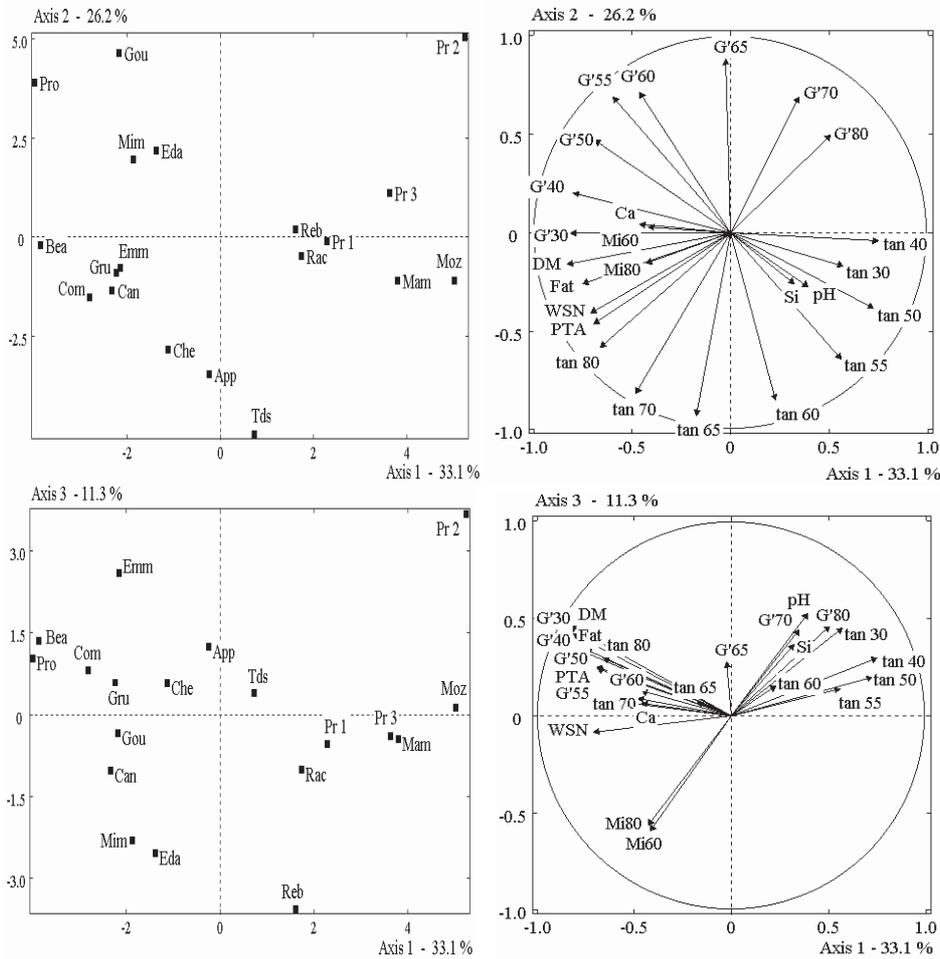


Figure 5. Principal component analysis (maps 1-2 and 1-3) of rheological and physico-chemical variables with indices of meltability and stretchability from different cheeses (Ca means Ca/NFDM, WSN means WSN/NT, PTA means PTA-SN/NT).

a multidimensional approach. The PC1-PC3 diagram shows a real but weak opposition between G'65 and the stretchability index on one side, and the meltability indices on the other side.

4. CONCLUSION

Throughout this study a simple, repeatable rheological test enabled us to establish

a profile representative of each cheese variety. However, given the commercial origin of the cheeses, it is impossible to establish a relationship between the technological factors of their composition and their functional properties. With this in mind, our laboratory used an experimental approach, using factorial design constructed from a cheese model. This study, conducted with the help of bibliographical

data and competences developed at the ENIL of Mamirolle, should enable us to identify and classify according to hierarchy the influence of technological factors and composition on the rheological variables and on the functional properties.

Thus, further studies on other cheese varieties are required to understand better the relationships between cheese-making conditions and functional properties in order to optimise them and even to predict them.

ACKNOWLEDGEMENTS

The authors acknowledge the members of the ENIL of Mamirolle: P. Dieudonné for his expertise in cheese technology, V. Dechartre for his technical assistance, and L. Bego for his initial contribution. J.-M. Reparet gratefully acknowledges financial support from the ENIL of Mamirolle. Y. Noël's work was funded by INRA. The study received financial support from European funds (FEOGA) and the Regional Council of Franche-Comté (France).

REFERENCES

- [1] Ak M.M., Gunasekaran S., Measuring elongational properties of Mozzarella cheese, *J. Text. Stud.* 26 (1995) 147–160.
- [2] Ak M.M., Gunasekaran S., Dynamic rheological properties of Mozzarella cheese during refrigerated storage, *J. Food Sci.* 61 (1996) 566–569.
- [3] Apostolopoulos C., Simple empirical and fundamental methods to determine objectively the stretchability of Mozzarella cheese, *J. Dairy Res.* 61 (1994) 405–413.
- [4] Arnott D.R., Morris H.A., Combs W.B., Effect of certain chemical factors on the melting quality of process cheese, *J. Dairy Sci.* 40 (1957) 957–963.
- [5] Blond D., Lemeste M., Propriétés d'hydratation des macromolécules. Relation avec leurs propriétés fonctionnelles, *Cah. l'ENSBANA* 6 (1988) 11–31.
- [6] Bonomi F., Iametti S., Real-time monitoring of the surface hydrophobicity changes associated with isothermal treatment of milk and milk protein fractions, *Milchwissenschaft* 46 (1991) 71–74.
- [7] Cavella S., Chemin S., Masi P., Objective measurement of the stretchability of Mozzarella cheese, *J. Text. Stud.* 23 (1992) 185–194.
- [8] Cavella S., Chemin S., Masi P., Évaluation objective de la filabilité de quelques fromages à pâte filée, *Ind. Alim. Agric.* 110 (1993) 11–15.
- [9] Cayot P., Lorient D., Effet des traitements thermiques sur la structure des protéines, in: *Structures et Technofonctions des Protéines du Lait*, Ch. 7, Arilait Recherches, Tec. et Doc, Paris, France, 1998, pp. 107–147.
- [10] Cheftel J.C., Cuq J.L., Lorient D., *Protéines alimentaires*, Tec et Doc, Paris, France, 1985.
- [11] Ferry J.D., *Viscoelastic Properties of Polymers*, Wiley and Sons, New York, USA, 1980.
- [12] Guinee T.P., O'Callaghan D.J., The use of a simple empirical method for objective quantification of the stretchability of cheese on cooked pizza pies, *J. Food Eng.* 31 (1997) 147–161.
- [13] Guinee T.P., Auty M.E., Mullins C., Observations on the microstructure and heat-induced changes in the viscoelasticity of commercial cheeses, *Aust. J. Dairy Technol.* 54 (1999) 84–89.
- [14] Guinee T.P., Harrington D., O'Corcoran M., Mulholland E.O., Mullins C., The compositional and functional properties of commercial Mozzarella, Cheddar and analogue pizza cheeses, *Int. J. Dairy Technol.* 53 (2000) 51–56.
- [15] Heiss E., Essai du dosage de la matière grasse dans les fromages par des méthodes rapides, *Deutsch. Molk. Ztg.* 82 (1961) 67–70.
- [16] Holt C., Molecular basis of whey protein food functionalities, *Aust. J. Dairy Technol.* 55 (2000) 53–55.
- [17] Horne D.S., Banks J.M., Leaver J., Law A.J.R., Dynamic mechanical spectroscopy of Cheddar cheese, in: *Cheese yield and factors affecting its control*, Special issue 9402, *Int. Dairy Fed.*, Brussels, Belgium, 1993, pp. 507–512.
- [18] IDF, Cheese and processed cheese: determination of the total solids content, Standard 4A, *Int. Dairy Fed.*, Brussels, Belgium, 1982.
- [19] IDF, Détermination de la teneur en azote (méthode Kjeldhal), Standard 20B, *Int. Dairy Fed.*, Brussels, Belgium, 1993.
- [20] Kimura T., Sagara Y., Fukushima M., Taneya S., Effect of pH on submicroscopic structure of String cheese, *Milchwissenschaft* 47 (1992) 547–552.

- [21] Kindstedt P.S., Kiely L.J., Revised protocol for the analysis of melting properties of Mozzarella cheese by helical viscometry, *J. Dairy Sci.* 75 (1992) 676–682.
- [22] Kindstedt P.S., Ripe J.K., Duthie C.M., Measurement of Mozzarella cheese melting properties by helical viscometry, *J. Dairy Sci.* 72 (1989) 3117–3122.
- [23] Kosikowski F.V., Mistry V.V., Cheese and fermented milk foods, Kosikowski F.V. (Ed.), Westport, CT, USA, 1997.
- [24] Lefevre I., Dewettinck K., Huyghebaert A., Cheese fat as driving force in cheese flow upon melting, *Milchwissenschaft* 55 (2000) 563–565.
- [25] Mc Mahon D.J., Fife R.L., Oberg C.J., Water partitioning in Mozzarella cheese and its relationship to cheese meltability, *J. Dairy Sci.* 82 (2000) 1361–1369.
- [26] Mounsey J.S., O’Riordan E.D., Empirical and dynamic rheological data correlation to characterize melt characteristics of imitation cheese, *J. Food Sci.* 64 (1999) 701–703.
- [27] Oberg C.J., Mc Manus W.R., Mc Mahon D.J., Microstructure of Mozzarella cheese during manufacture, *Food Struct.* 12 (1993) 251–258.
- [28] Olson N.F., Nelson D.L., A new method to test the stretchability of Mozzarella cheese on pizza, Proc. 17th Annu. Marschall Invit. Ital. Cheese Sem., Madison, WI, USA, 1980.
- [29] Park J., Rosenau J.R., Peleg M., Comparison of four procedures of cheese meltability, *J. Food Sci.* 49 (1984) 1158–1161, 1170.
- [30] Pearce K.N., The complexometric determination of calcium in dairy products, *N.Z. J. Dairy Sci. Technol.* 12 (1977) 113–115.
- [31] Richoux R., Roset G., Famelart M.-H., Kerjean J.-R., Diversité de quelques propriétés fonctionnelles à chaud de l’Emmental français, *Lait* 81 (2001) 547–559.
- [32] Rowney M., Roupas P., Hickey M.W., Everett D.W., Factors affecting the functionality of Mozzarella cheese, *Aust. J. Dairy Technol.* 54 (2000) 94–102.
- [33] Savage A.A., Mullan W.M., Evaluation of helical viscometry for assessing the functional properties of Mozzarella cheese, *Int. J. Dairy Technol.* 53 (2000) 57–62.
- [34] Savello P.A., Ernstrom C.A., Kalab M., Microstructure and meltability of model process cheese made with rennet and acid casein, *J. Dairy Sci.* 72 (1989) 1–11.
- [35] Smith C.E., Rosenau J.R., Peleg M., Evaluation of the flowability of melted Mozzarella cheese by capillary rheometry, *J. Food Sci.* 45 (1980) 1142–1145.
- [36] Taneya S., Izustu T., Sone T., Dynamic viscoelasticity of natural and processed cheese, in: Sherman P. (Ed.), *Food Texture and Rheology*, Academic Press, New York, USA, 1979, pp. 369–383.
- [37] Tunick M.H., Mackey K.L., Smith P.W., Holsinger V.H., Effects of composition and storage on the texture of Mozzarella cheese, *Neth. Milk Dairy J.* 45 (1991) 117–125.
- [38] U.S.D.A., Methods of laboratory analysis for meltability, color, stretchability and free fat for types of Mozzarella cheese for pizza making, Washington, DC: Dairy Grading Section, Dairy division, Agriculture Marketing service, Instruction 918-102-2, 1983.
- [39] Ustunol Z., Kawachi K., Steffe J., Arnott test correlates with dynamic rheological properties for determining Cheddar cheese meltability, *J. Food Sci.* 59 (1994) 970–971.
- [40] Walstra P., Physical chemistry of fat milk globules, in: Fox P.F. (Ed.) *Developments in dairy chemistry-2*. Applied science publishers, London, UK 1983, pp. 119–158.