

## Delineation of the texture of Salers cheese by sensory analysis and physical methods

Annick LEBECQUE<sup>a</sup>, Arlette LAGUET<sup>a</sup>,  
Marie Françoise DEVAUX<sup>b</sup>, Éric DUFOUR<sup>a,\*</sup>

<sup>a</sup> U.R. « Typicité des Produits Alimentaires »  
Département Qualité et Économie Alimentaires, ENITA de Clermont Ferrand,  
63370 Lempdes, France

<sup>b</sup> URPOI-INRA, BP 71627, 44316 Nantes Cedex 3, France

(Received 27 July 2000; accepted 13 February 2001)

**Abstract** — The 25 investigated Salers cheeses graded by the professional committee offered a large range of textures. Their texture characteristics were investigated by sensory analysis, rheological measurements and fluorescence spectroscopy. The 8 sensory attributes generated by the panel included the evaluation of mechanical, geometric and surface properties, and allowed the characterisation of the texture of the investigated cheeses. The stress values at 20% compression ranged between 0.073 and 0.290 kPa. The fluorescence spectra of protein tryptophans and vitamin A were recorded directly on the samples. Principal component analysis and canonical correlation analysis were used to evaluate the data and to investigate the relationships between the different sets of data recorded on the same cheeses. All the methods used to analyse the cheeses allowed discrimination of the samples, i.e. the obtained data constituted fingerprints of the cheeses. Moreover, strong correlations were found between the sensory attribute domain and the instrumental domains. It was also suggested that the phenomena observed at molecular and macroscopic levels were related to the texture of the cheeses.

**cheese / texture / sensory analysis / rheology / fluorescence**

**Résumé** — Étude de la diversité de la texture du fromage de Salers par l'analyse sensorielle et les méthodes instrumentales. Les 25 fromages de Salers étudiés, gradés par le comité professionnel, présentaient une large gamme de texture. Leurs caractéristiques de texture ont été évaluées par analyse sensorielle, mesures rhéologiques et spectroscopie de fluorescence. Les 8 descripteurs sensoriels définis par le panel permettaient l'évaluation des propriétés mécaniques, géométriques et de surface; autorisant la description de la texture des fromages étudiés. Les valeurs de la contrainte à 20 % de compression s'échelonnaient entre 0,073 et 0,290 kPa. Les spectres de fluorescence des tryptophanes des protéines et de la vitamine A ont été enregistrés directement sur les échantillons. L'analyse en

---

\*Correspondence and reprints

Tel.: (33) 4 73 98 13 78; fax: (33) 4 73 98 13 90; e-mail: dufour@gentiane.enitac.fr

composantes principales et l'analyse canonique des corrélations ont été utilisées pour analyser les données et évaluer les relations entre les différents jeux de données enregistrés sur les mêmes échantillons de fromage. Toutes les méthodes d'analyse utilisées dans cette étude permettaient de discriminer les fromages : les données enregistrées constituaient des empreintes digitales des fromages. De plus, des corrélations fortes étaient mises en évidence entre le domaine des descripteurs sensoriels et les domaines des mesures instrumentales. Il a été aussi suggéré que les phénomènes observés aux niveaux moléculaire et macroscopique étaient reliés à la texture des fromages.

## **fromage / texture / analyse sensorielle / rhéologie / fluorescence**

### **1. INTRODUCTION**

There is a great interest in defining quality, especially for Protected Denomination of Origin (PDO) cheeses [12] which often present a large variety of tastes and textures. Production of consistently high quality PDO cheeses continues to be a challenge for people involved in the chain of production. In addition, there is an increasing need for cheese characterisation. It is the consequence of the impact of the definition of identity, authenticity and quality on the application of policies of the European Union and of the protection and information of consumers.

Salers is a PDO semi-hard cheese, produced in the centre of France, in the surrounding area of Salers town. It is made twice a day in farms from raw milk coagulated with rennet. After coagulation (renneting time is about 16 min), the curd is cut to obtain grains the size of hazelnuts. After 15–30 min, the whey is drained and the packed curd is lightly pressed. After about 12 h aging, the packed curd is milled to obtain granules the size of maize grains, salted, hooped and pressed for 2 days. The Salers cheese is ripened for at least 3 months. The weight of a ripened cheese is on average 45 kg; its shape is like a cylinder.

Texture is an important criterion used to evaluate the quality of cheeses. Cheese variety and composition influence component distribution which in turn largely deter-

mines structure and texture characteristics [9]. It is generally assumed that at room temperature milk proteins contribute to firmness and milk fats provide smoothness to cheese: the higher the fat content of cheese, the softer the cheese [19]. As a good curd is a prerequisite to obtain a quality cheese, it implies that milk coagulation is a major step of cheese manufacturing largely determining the texture of the product. The main parameters that have an effect on the physical properties of the curd are the milk composition, the acidification kinetic and the amount of rennet. For example, a change in fat content affects protein aggregation and can modify the physical properties of cheese.

Texture is a multivariate attribute (mechanical, geometrical and surface) that can be assessed by various methods. Sensory analysis is widely used by researchers and professionals in order to assess food quality and to predict consumer acceptance. Sensory evaluations require a large panel of trained assessors, are time consuming, and so limited to a restricted number of samples. Instrumental methods may be used to characterise the texture of cheeses. For example, rheological measurements allow access to mechanical attributes of texture. Several studies dealing with the relationships between rheology and sensory properties have been published [13, 22–24]. Alternative methods such as fingerprint methods based on spectroscopic measurements have the ability to give specific

information about structural properties of the product that could be useful for texture characterisation and quality management [5, 14].

Front face fluorescence spectroscopy allows investigation of the fluorescence of powdered, turbid, emulsified and concentrated samples [2, 6, 10–11, 16, 25]. One of the main advantages of this fast method is its ability to record spectra directly on the cheese sample. In addition, the spectrum recorded on a given cheese is a fingerprint of the sample that retains information about its physico-chemical characteristics. The results of canonical correlation analysis applied to soft-cheese sensory profile data and fluorescence spectral collection showed that the 2 groups of variables were highly correlated [14].

Until now, scientific information about rheological and sensory characteristics of Salers cheese has been lacking. The aim of this study was to delineate texture characteristics of Salers cheese by sensory analysis, rheological measurements and fluorescence spectroscopy. Multidimensional statistical methods were used to evaluate the data and to investigate the relationship between sensory data, rheological parameters and spectral collections. In addition, the approach reported in this paper can be considered as a generic approach for the delineation of the diversity of PDO cheeses.

## 2. MATERIALS AND METHODS

### 2.1. Cheeses

Twenty five Salers cheeses of  $3.5 \pm 0.5$  months ripening time were provided by C.I.F. Cantal (Aurillac, France), the professional committee for Cantal and Salers cheeses. The cheese samples graded by the professional committee offered a large range of textures. The coding for the samples is presented in Table I.

Slices were cut in the middle of the cheese radius for physico-chemical, rheological and spectroscopic analysis. Samples were taken midway between the centre and rind of cheeses for sensory analysis.

### 2.2. Physico-chemical analysis

The determination of pH, dry matter, fat content and total nitrogen as protein content for the 25 Salers cheeses was as described by Bouton et al. [3].

### 2.3. Sensory analysis

Twelve subjects (ranging between 21 and 28 years old) were selected (2 sessions) for their sensory ability and trained (12 sessions) for descriptive analysis

**Table I.** Coding of the Salers cheeses.

	Set	1	2	3	4	5
Cheese coding		A1	B1	C1	D2	E1
		A5	B4	C4	D4	E2
		A6	B8	C5	D7	E5
		A8	B9	C8	D8	E6
		A9	B10	C9	D10	E8

according to the guidelines in the ISO 11036:1995 standard and to the method developed by Lavanchy et al. [18]. The eight attributes for texture profiling chosen by the panel included the evaluation of mechanical, geometric and surface properties perceived by hand and mouth: firmness, elasticity, friability, adhesivity, deformability, micro-structure, humidity, cracks. A 0 to 9 scale was used to evaluate the intensity of the attributes. The assessors were trained in the use of definitions of sensory attributes and scales by pre-testing extreme and different cheeses.

Texture profiles of 15 different cheeses (B, D and E sets; see Tab. I) were assessed using the procedure described above. They were evaluated in duplicate over 6 sessions. Room-tempered samples were presented simultaneously on individual plates in randomised order. The mouth was rinsed with mineral water between samples.

## 2.4. Rheological analysis

Samples were taken with a cork borer mounted on a drilling machine stand, driven slowly by hand. The cylinders (18 mm diameter) were cut with a cutting-device to a height of 20 mm. The test pieces were stored in a small airtight box (to avoid dehydration) for 1 h to allow relaxation before testing at 20 °C.

The rheological method used was uniaxial compression at constant displacement rate. An Instron testing machine equipped with parallel plates and a 1 000 N load cell was used. The displacement rate was 50 mm·min<sup>-1</sup>. 6 specimens were tested for each cheese sample. Data on force and displacement were recorded for 25 cheeses. The force and height variation data were computed as stress/strain curves. The full curves were analysed by principal component analysis.

## 2.5. Fluorescence spectroscopy

Fluorescence spectra were recorded using a SLM 4800C spectrofluorimeter (Bioritech, Chamarande, France) mounted with a front-surface accessory. The incidence angle of the excitation radiation was set at 56° to ensure that reflected light, scattered radiation and depolarisation phenomena were minimised. Spectra of cheese samples (3 cm × 1 cm × 0.3 cm) mounted between two quartz slides were recorded at 20 °C with emission and excitation slits set at 4 nm (resolution: 0.5 or 1 nm, averaging: 10). The emission spectra of tryptophan residues (305–400 nm) were recorded with the excitation wavelength set at 290 nm and the excitation spectra of vitamin A (260–350 nm) were recorded with the emission wavelength set at 410 nm. All spectra were corrected for instrumental distortions in excitation using a rhodamine cell in the reference channel.

Slices of 3 cm length and 0.3 cm thickness were cut in the middle of the cheese radius. 25 Salers cheeses were analysed. For each individual cheese, three spectra were recorded using different samples.

## 2.6. Mathematical treatment of data

### 2.6.1. Analysis of the sensory data

The analysis of the sensory data was carried out using Stagraphics 3.1 (Statistical Graphics Corp., Rockville, USA) and Tastel (ABT Informatique, Paris, France) statistical packages.

### 2.6.2. Multidimensional analysis of the data

In order to reduce scattering effects, the fluorescence spectra were normalised by reducing the area under each spectrum to a value of 1 according to Bertrand and Scotter [1]. Principal Component Analysis (PCA) was applied to the physico-chemical

data, to the stress/strain curves and to the normalised spectra in order to investigate changes in the data [14–15]. This statistical multivariate treatment makes it possible to draw similarity maps of the samples and to get spectral patterns [1, 17]. While the similarity maps allow the comparison of the spectra in such a way that two neighbouring points represent two similar spectra, the spectral patterns exhibit the absorption bands that explain the similarities observed on the maps.

### *2.6.3. Correlation between two sets of data*

In a first step, the 8 sensory variables of 15 cheeses were predicted from the tryptophan fluorescence spectra using the principal component regression technique (PCR). In a second step, the chemical data, the rheological data, the fluorescence spectral data and the sensory data were further analysed using canonical correlation analysis (CCA), a multivariate treatment that describes the correlations between two sets of data [26]. CCA was performed on the sensory and instrumental data recorded on 15 cheeses. This method makes it possible to assess new variables, called canonical variates, as linear combinations of the variables of each data set so that these new variables exhibit the highest correlation that can be found between the 2 groups of data. The method has been successfully applied for comparing fluorescence and mid infrared spectra of semi-hard cheeses [5], and confocal microscopy images and sensory data of soft cheeses [4]. In both cases, the authors were able to provide relevant similarity maps of the samples that were not immediately found by principal component analysis.

CCA was carried out with Splus software. PCA and PCR were performed using software written in the C language at INRA Nantes.

## **3. RESULTS AND DISCUSSION**

### **3.1. Physico-chemical characteristics**

As generally observed for PDO products, pH, proteins, dry matter and fat contents presented a large variability for the investigated cheeses (Tab. II). The range of variation for dry matter, fat, fat/dry matter, proteins and pH of cheeses was 2.3%, 6.5%, 4.9%, 4.1% and 2.1%, respectively. Some parameters, such as fat and dry matter, were strongly correlated ( $R = +0.83$ ). Some others were negatively correlated: for proteins and fat,  $R = -0.59$ .

Principal component analysis was applied to these data and the main part of the variability was explained by the first two axes. Principal component 1 (PC1; 60.9% of total inertia) opposed cheeses with the highest fat/dry matter contents (E2, E8, E5 and E1) (correlated with the highest fat contents) to cheeses with the highest protein contents (B1, C1 and C4). PC2 describing 21.9% of the variability appeared to be mainly related to cheese pH: E2, A8 and A1 cheeses with the lowest pH were opposed to B10 and D2 cheeses with the highest pH. This last variable was not correlated with the others (data not shown).

### **3.2. Rheological properties**

The stress values at 20% and 80% compressions for the 25 cheeses are presented in Table II. The stress values at 20% compression ranged between 0.073 and 0.290 kPa. PCA was applied to the collection of stress/strain curves in order to investigate the diversity of the rheological characteristics of the Salers cheeses. The first two principal components took into account 98.6% of the total variation. A discrimination of the samples was essentially observed according to the principal component 1 (93.8% of the inertia): A1, A8, A9 and E2 cheeses, exhibiting the highest stress values at 20% compression, were on the positive side, while C1, C5, D7, E5 and

**Table II a.** Composition and rheological parameters.

Cheese	Chemical composition				Stress (kPa) at compression	
	pH	protein (%)	fat (%)	dry matter (%)	20%	80%
A1	5.42	24.45	29.50	60.75	0.272 ± 0.029	1.116 ± 0.289
A5	5.57	25.07	29.75	60.47	0.226 ± 0.007	1.090 ± 0.048
A6	5.42	23.74	29.25	58.96	0.151 ± 0.011	0.911 ± 0.092
A8	5.41	24.69	30.75	60.88	0.228 ± 0.021	1.158 ± 0.112
A9	5.64	25.45	30.00	61.09	0.200 ± 0.014	1.225 ± 0.099
B1	5.56	26.88	23.25	56.31	0.188 ± 0.020	0.985 ± 0.050
B4	5.64	24.35	31.00	59.82	0.101 ± 0.001	0.750 ± 0.025
B8	5.74	26.06	30.75	59.15	0.124 ± 0.005	0.976 ± 0.096
B9	5.57	24.58	29.50	60.28	0.098 ± 0.009	0.645 ± 0.038
B10	5.83	24.33	29.75	58.94	0.094 ± 0.004	0.666 ± 0.026
C1	5.50	25.49	27.50	57.16	0.087 ± 0.009	0.593 ± 0.009
C4	5.64	26.60	27.50	58.79	0.109 ± 0.007	0.723 ± 0.021
C5	5.58	24.80	32.00	61.39	0.112 ± 0.005	0.554 ± 0.012
C8	5.55	23.53	30.50	59.53	0.091 ± 0.001	0.550 ± 0.029
C9	5.44	24.36	30.50	60.35	0.138 ± 0.003	0.806 ± 0.070
D2	5.79	23.75	28.50	58.41	0.083 ± 0.005	0.723 ± 0.057
D4	5.76	24.46	30.25	60.23	0.106 ± 0.015	0.766 ± 0.065
D7	5.69	24.08	30.75	59.74	0.073 ± 0.005	0.523 ± 0.011
D8	5.70	24.37	28.75	58.84	0.130 ± 0.003	1.560 ± 0.060
D10	5.69	25.16	30.25	61.03	0.136 ± 0.005	0.801 ± 0.036
E1	5.60	23.49	31.50	59.97	0.080 ± 0.006	0.583 ± 0.060
E2	5.43	24.36	33.50	63.05	0.290 ± 0.010	1.276 ± 0.090
E5	5.73	23.10	31.50	59.96	0.090 ± 0.010	0.563 ± 0.040
E6	5.70	24.62	31.00	60.22	0.110 ± 0.010	0.773 ± 0.057
E8	5.57	22.57	31.50	59.97	0.080 ± 0.004	0.576 ± 0.011

**Table II b.** Physico-chemical composition (g·100 g<sup>-1</sup> fresh product) (*n* = 25).

Content	Mean	Minimum	Maximum	SD
pH	5.61	5.41	5.83	0.12
Proteins	24.57	22.57	26.88	1.00
Dry matter	59.81	56.31	63.05	1.37
Fat	29.96	23.25	33.50	1.94
Fat/dry matter	50.05	41.30	53.10	2.45

E8 cheeses with the lowest stress values at 20% compression showed negative scores (data not shown).

### 3.3. Sensory analysis

ANOVA was performed on the sensory data. Significant differences were observed between cheeses, allowing us to class them in five groups characterised by several attributes:

- Group 1 (B1, B5, B8): high adhesivity, high humidity, low friability, low firmness;
- Group 2 (B4, B9, D4): low humidity, fairly friable, low firmness;
- Group 3 (D8, D10, E1, E6): average values for all attributes;
- Group 4 (D2, D7, E8, E5): low friability, fairly firm, fine microstructure;
- Group 5 (E2): firm, dry, significant microstructure.

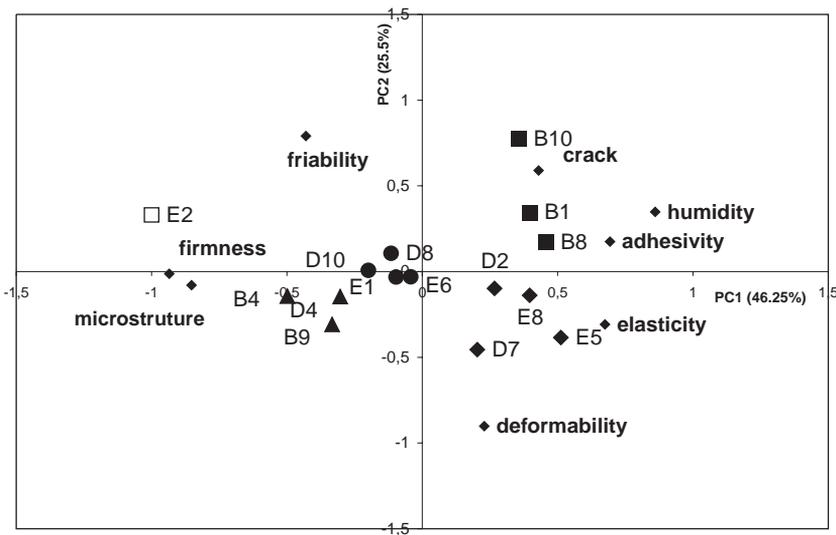
Several sensory attributes describing the texture of the investigated cheeses did not vary independently. The positive and nega-

tive correlations between the sensory attributes are presented in Table III.

PCA was then applied to the sensory data. The first two principal components took into account 71% of the total variation. A discrimination of the samples was essentially observed according to the principal component 1 (46% of the inertia): E5, E8, B8 and B1 cheeses, characterised by humidity, adhesivity and elasticity, were on the positive side, while E2, B4 and D10

**Table III.** Correlation coefficients between the sensory attributes.

Attributes	r
firmness/humidity	-0.86
microstructure/adhesivity	-0.86
firmness/elasticity	-0.76
microstructure/humidity	-0.67
microstructure/firmness	+0.68
adhesivity/humidity	+0.57



**Figure 1.** Principal component analysis similarity map determined by principal components 1 and 2 for sensory data.

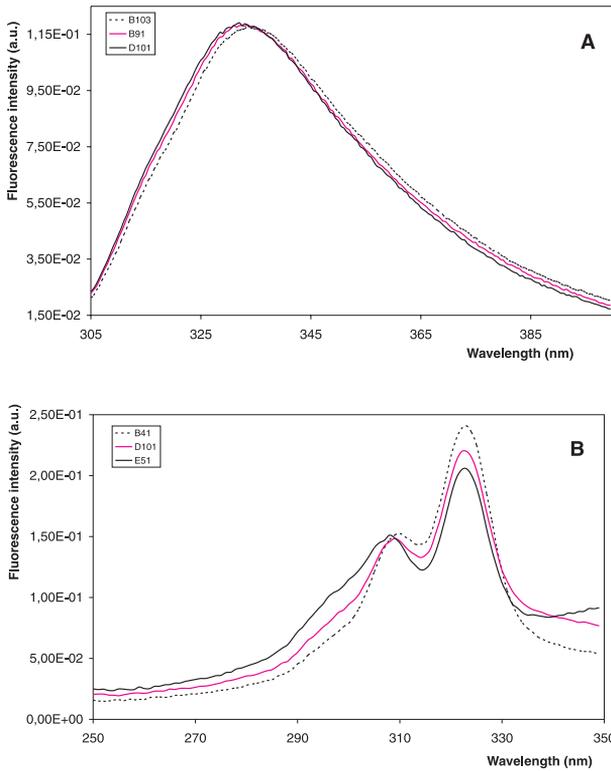
cheeses well described by microstructure, firmness and friability showed negative scores (Fig. 1). Comparing rheological and sensory data, it was observed that cheeses with high scores for humidity, adhesivity and elasticity presented the lowest stress values at 20% compression, whereas cheeses described by microstructure, firmness and friability were correlated with the highest stress values at 20% compression (see Sect. 3.5 for further explanation).

### 3.4. Cheese discrimination from their tryptophan and vitamin A spectra

Fluorescent properties of fluorophores are very sensitive to the changes of their environment [21]. As the 25 Salers cheeses in-

vestigated in this study exhibited relatively different rheological properties, it was assumed that their structures, and as a consequence the environments of the fluorophores, were different [14]. The tryptophan emission spectra of 3 different cheeses are presented in Figure 2a. The tryptophan presented a maximum located at about 332 nm, which location varied slightly from one cheese to another.

The excitation fluorescence spectra of vitamin A located in the fat globules of the cheeses showed a maximum at 322 nm and two shoulders at 305 and 295 nm (Fig. 2b). More interestingly, the shape of the spectra changed with the cheese. The ratio  $F_{322nm}/F_{295nm}$  was different from one cheese to another. The differences observed

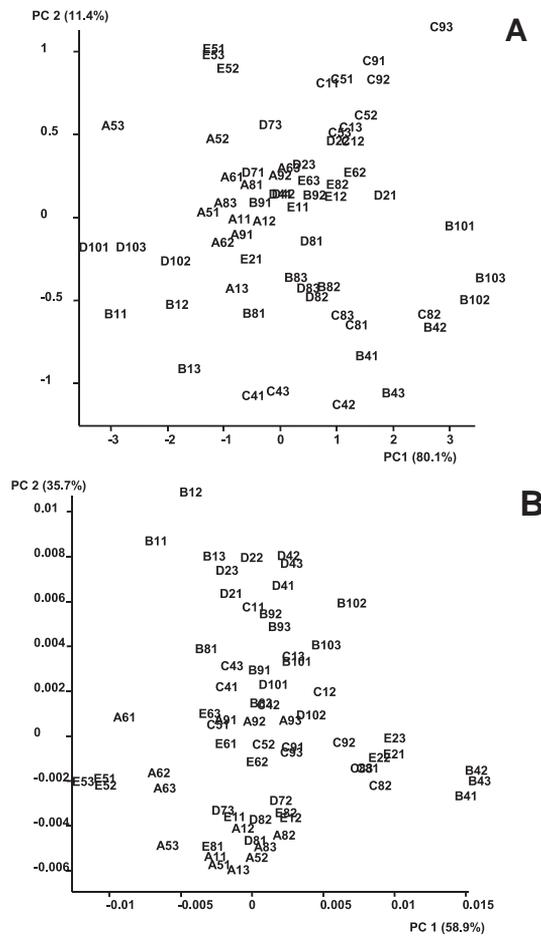


**Figure 2.** Fluorescence spectra of cheeses: (A) Tryptophan emission and (B) Vitamin A excitation.

for vitamin A fluorescence spectra are consistent with changes of lipid structure, but the interpretation at the molecular level is more difficult. Nevertheless, it is well known that the fluorescent properties of fluorophores are very sensitive to the changes of the solvent viscosity [21]. Recently, it has been reported that the shape of the vitamin A excitation spectrum is correlated with the physical state of the triglycerides in the fat globules of an emulsion [7]. In this case, the ratio  $F_{322nm}/F_{295nm}$  decreased during the melting of the triglycer-

ides. On the other hand, the changes in the shape of vitamin A excitation spectra recorded during milk coagulation have been correlated to the changes of fat globule/protein interactions [14]. The development of interactions between the protein network and the fat globules in coagulation kinetics has been demonstrated by fluorescence transfer between tryptophan and vitamin A in the case of the coagulation of reconstituted milks [20].

Although the different cheeses exhibit weakly different fluorescence spectra,



**Figure 3.** Principal component analysis similarity map determined by principal components 1 and 2 for the tryptophan (A) and vitamin A (B) spectra of cheeses. Coding: the letter and the first digit are for cheese coding (see Table I), the last digit is for the repetition.

univariate analysis is not really appropriate for the study of complex spectra. Multivariate analysis techniques such as PCA are more appropriate in this case. For this purpose, 75 tryptophan spectra and 75 vitamin A spectra were collected from the 25 cheeses.

In order to compare the set of fluorescence spectra and to emphasise the similarities and the differences underlined previously, principal component analysis was performed on the spectra to describe the main variations between the different cheeses. Considering the results of PCA performed on the tryptophan spectra, the first two principal components took into account 80.1% and 11.4% of the total vari-

ance, respectively. The map defined by the principal components 1 and 2 showed a discrimination of the cheeses (Fig. 3a).

PCA was applied to the set of vitamin A spectra and the map defined by principal components 1 and 2 is shown in Figure 3b. The first and the second principal components took into account 58.9% and 35.7% of the total variation, respectively, and discriminate the samples.

Considering the results obtained in the present study and in a previous study on soft cheeses [14], it is concluded that tryptophan and vitamin A fluorescence spectra are fingerprints allowing the discrimination of the cheeses.

**Table IV.** Squared correlation coefficients between observed and predicted value for the 8 sensory attributes obtained by Principal Component Regression.

	r <sup>2</sup>		
	Rheology	Tryptophan	Vitamin A
Humidity	0.35	0.1	0.1
Cracks	0.40	0.25	0.35
Elasticity	0.51	0.18	0.19
Firmness	0.33	0.29	0.11
Deformability	0.60	0.49	0.78
Friability	0.67	0.19	0.61
Adhesivity	0.14	0.13	0.52
Microstructure	0.15	0.23	0.52

**Table V.** Squared canonical correlation coefficients between sensory data and the instrumental data (rheology and fluorescence).

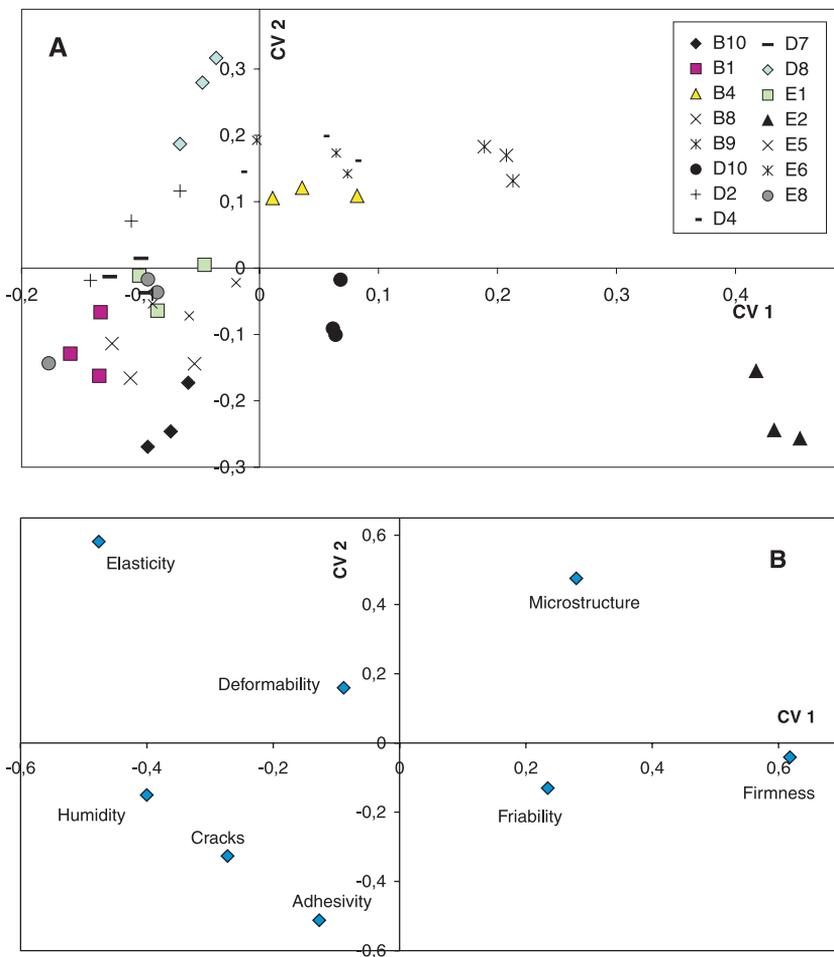
	Canonical variates			
	1	2	3	4
Rheology	0.81	0.78	0.43	0.08
Fluorescence:				
– tryptophan	0.80	0.71	0.19	0.12
– vitamin A	0.85	0.69	0.44	0.10

### 3.5. Correlations between sensory and rheological data

Using the data recorded on B, D and E cheese sets (Tab. I), the 8 sensory variables were predicted from the rheological data using the principal component regression technique (PCR). The method is a multiple regression applied from the principal components of the samples rather than from the raw data.

The correlation coefficients between the predicted and observed values from the rheological data are reported in Table IV. The results showed that the prediction of the sensory attributes with squared correlations higher than 0.5 were obtained for the following sensory attributes: elasticity, deformability and friability.

In a second step, the correlations between the sensory attribute domain and the rheology domain have been taken into



**Figure 4.** A – CCA similarity map defined by the canonical variates 1 and 2 for the rheological data and B – CCA variate map 1–2 for the sensory domain.

account in order to get a better insight into the correlations between the instrumental and sensory characteristics of the investigated cheeses. Canonical correlation analysis can be applied when the same samples have been characterised by two different techniques. The method provides both a global measure of the link between the group of variables and a graphical representation of the correlation revealed.

Four pairs of canonical variates were assessed to describe the correlation between the sensory data and the rheological data. The squared canonical coefficients for canonical variates 1, 2, 3 and 4 were 0.81, 0.78, 0.43 and 0.08 (Tab. V), respectively. The first two canonical variates were found highly correlated confirming that there was a relation between sensory data and rheological data. The similarity map of the canonical variates 1 and 2 assessed from rheological data is shown in Figure 4a. Considering the canonical variate 1, B1 and B8 cheeses had negative scores, whereas E2 cheese had a positive score. In fact the sensory attributes characterising B1 and B8 were humidity and elasticity (Fig. 4b). On the other hand, E2 cheese was well characterised by firmness.

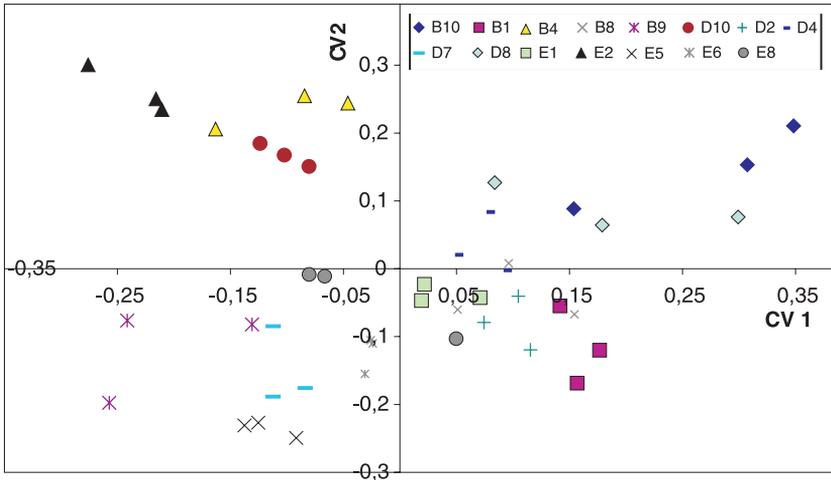
### 3.6. Correlations between sensory and fluorescence data

Using B, D and E cheese sets (Tab. I), the 8 sensory variables were predicted from the tryptophan and vitamin A spectral data using the principal component regression technique (PCR). The correlation coefficients between the predicted and observed values from the fluorescence data are reported in Table IV. The results showed that the sensory attributes were not well predicted from the tryptophan fluorescence spectra since all the squared correlation coefficients were below 0.5. This is quite surprising since it has been shown that the tryptophan fluorescence spectra of soft cheeses were highly correlated with texture

sensory attributes [14]. But the manufacturing of these soft cheeses involved different processes and their physico-chemical characteristics spanned over a large range. Indeed, the investigated set included mesophilic-, thermophilic- and ultrafiltered-processed cheeses exhibiting pH ranging between 5.0 and 7.5 and different fat contents (between 23 and 36 g·100 g<sup>-1</sup> of cheese). It is not the case in this study where the process for the manufacturing of Salers cheese is quite constant and all the physico-chemical parameters, but fat content, do not vary in such a large range for the studied cheeses (Tab. II).

A much better result was obtained from the vitamin A fluorescence spectra. The prediction of the sensory attributes with squared correlations higher than 0.5 were obtained for the following sensory attributes: microstructure, adhesivity, deformability and friability (Tab. IV). Previously, a very high correlation between the sensory attributes and the vitamin A spectra has been observed in the study on soft cheeses [14]. The present study confirms that vitamin A is a good fluorescent intrinsic probe allowing discrimination and characterisation of cheeses. In addition, fat content varies over a relatively large range for the studied cheeses (Tab. II). The milk composition is one of the parameters that have an effect on the physical properties of the curd. A change in fat content affects protein aggregation and can modify the physical properties of the cheese.

In a second step, canonical correlation analyses were applied in order to get a better insight into the relationships between the sensory profiles, and tryptophan and vitamin A fluorescence spectra. Four pairs of canonical variates were assessed to describe correlation between the sensory data and the fluorescence data. Table V reports the squared canonical correlation coefficients obtained for the different analyses. It appeared that tryptophan fluorescence spectra and vitamin A fluorescence spectra



**Figure 5.** CCA similarity map defined by the canonical variates 1 and 2 for the tryptophan fluorescence data.

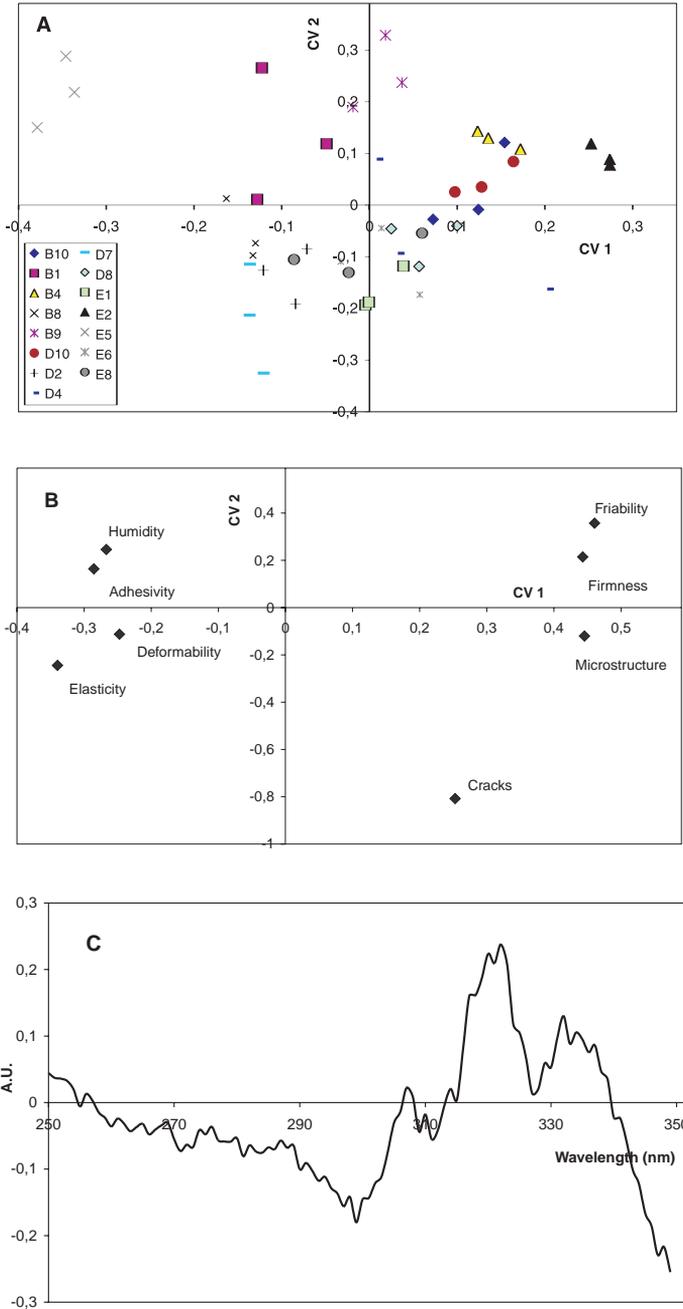
were highly correlated with sensory data. Considering fluorescence of vitamin A and sensory profiles, the first two pairs were correlated with squared canonical correlation coefficients equal to 0.85 and 0.69. These correlations indicated that the canonical variates provided a common description of the samples both from the fluorescence and sensory data. As the two techniques gave similar results, it strongly indicated that fluorescence spectroscopy allowed characterisation of the diversity of the texture properties of Salers cheeses.

The similarity maps 1–2 derived from the canonical correlation analysis performed on the sensory data and 1- the tryptophan spectra and 2- the vitamin A spectra are presented in Figure 5 and Figure 6a, respectively. The similarity maps obtained for the sensory data and vitamin A fluorescence spectra were similar to the similarity maps drawn from the sensory and rheology data (Fig. 4a), and looked like the PCA similarity map for sensory data (Fig. 1). Nevertheless a different trend was observed for the similarity maps derived from the results of the canonical correlation

analysis performed on the sensory data and the tryptophan fluorescence spectra.

Considering the sensory attributes explaining the samples discrimination according to the canonical variates 1, elasticity, humidity, adhesivity and deformability had negative scores, whereas microstructure, friability and firmness had positive scores (Figs. 4b, 6b) for both CCA performed on sensory, rheology and vitamin A fluorescence data. As the three different techniques allowed discrimination of the texture of the investigated cheeses and gave the same trends (Figs. 4a, b and 6a, b), it is suggested that the phenomenon observed at molecular and macroscopic levels are related to the texture of cheeses.

In the region of vitamin A fluorescence, the shape of the spectral pattern associated with the first canonical variate showed a negative peak at 300 nm and two positive peaks at 320 and 332 nm (Fig. 6c). This pattern showed that the cheeses characterised by elasticity, humidity, adhesivity and deformability sensory-attributes displayed a higher fluorescence intensity at 300 nm and lower fluorescence intensities at 320



**Figure 6.** A – CCA similarity map defined by the canonical variates 1 and 2 for the vitamin A fluorescence data, B – CCA variate map 1–2 for the sensory domain and C – spectral pattern associated with the canonical variate 1.

and 332 nm than the cheeses with high scores for microstructure, friability and firmness sensory-attributes.

Considering the semi-hard cheeses such as Salers cheese, the interactions between the protein network and the fat globule vary, depending on the milk composition and on the manufacturing process (acidification rate), and modify in different ways the vitamin A spectra [7, 14, 15]. Crystallisation of the triglycerides during ripening time may also alter the shape of vitamin A fluorescence spectra [8, 20]. However, further experiments are required in order to conclude on this point.

#### 4. CONCLUSION

Salers cheeses were characterised by sensory analysis, rheology measurements and intrinsic fluorescence (tryptophan and vitamin A) spectra. The data tables were analysed by multi-dimensional methods such as principal components analysis and canonical correlation analysis. The obtained similarity maps showed that it was possible to characterise the diversity of the texture of Salers cheese. Sensory analysis, as well as fluorescence spectroscopy and rheology measurements, may be valuable tools to define quality, identity and authenticity of PDO cheeses. The results of the experiments showed that the fluorescence spectra recorded directly on cheeses were fingerprints allowing their identification. In addition, we have demonstrated using PCR and CCA that there are strong correlations between instrumental data and sensory attributes of cheese texture. Using PCR, 4 sensory attributes, over the 8, can be predicted with a good accuracy from the vitamin A fluorescence spectra. Rapid and non-invasive methods such as fluorescence spectroscopy may be useful for the characterisation and the prediction at a young stage of PDO cheese texture.

Spectroscopic methods have been used for a long time to determine the chemical composition of foodstuffs. It is shown in this study that quality parameters of food products such as texture attributes may be derived from fluorescence data. Fluorescence data are spectra which also allow us to derive information on the molecular structure and interactions of the cheese matrix.

#### ACKNOWLEDGMENTS

This research was partly funded by ONILAIT. The authors would like to acknowledge Comité Interprofessionnel des Fromages du Cantal (C. Péchaud, C. Hamerel), Pôle Fromager AOC Massif Central (N. Ballot) and Station de Recherche sur la Viande (J. Culioli, B. Dominguez) of the Institut National de la Recherche Agronomique, Theix, France.

V. Cabordel, E. Forster, C. Caravagno, J. Noble and S. Sordet are gratefully acknowledged for their collaboration on this project. N. Mouhous (UEIMA, INRA Nantes) is thanked for the recording of fluorescence spectra.

#### REFERENCES

- [1] Bertrand D., Scotter C.N.G., Application of multivariate analyses to NIR spectra of gelatinized starch, *Appl. Spectrosc.* 46 (1992) 1420–1425.
- [2] Blumberg W.E., Doleiden F.H., Lamola A.A., Hemoglobin determined in 15  $\mu$ L of whole blood by "front face" fluorometry, *Clin. Chem.* 26 (1980) 409–413.
- [3] Bouton Y., Guyot P., Dasen A., Grappin R., Activité protéolytique de souches de lactobacilles thermophiles isolées de levain et de Comté. II – Applications en sites industriels, *Lait* 75 (1994) 31–44.
- [4] Devaux M.F., Collewet G., Herbert S., Mariette F., Riaublanc A., Bouchet B., Fortier P., Relations entre l'analyse sensorielle de fromage à pâte molle et l'imagerie par résonance magnétique nucléaire et microscopie confocale, in *Compte rendu des 6<sup>e</sup> journées agro-industrie et méthodes statistiques*, 2000, pp 11–20.
- [5] Dufour E., Mazerolles G., Devaux M.F., Duboz G., Duployer M.H., Mouhous Riou N., Phase transition of triglycerides during semi-hard cheese ripening, *Int Dairy J.* 10 (2000) 87–99.

- [6] Dufour E., Dalgalarondo M., Adam L., Conformation of (-lactoglobulin at an oil/water interface as determined from proteolysis and spectroscopic methods, *J. Coll. Interface Sci.* 207 (1998) 264–272.
- [7] Dufour E., Lopez C., Riaublanc A., Mouhous Riou N., La spectroscopie de fluorescence frontale: une approche non invasive de la structure et des interactions entre les constituants des aliments, *Agoral* 10 (1998) 209–215.
- [8] Dufour E., Mazerolles G., Devaux M.F., Duboz G., Duployer M.-H., Mouhous-Riou N., Phase transition of triglycerides in fat globules during semi-hard cheese ripening as studied by mid-infrared and front-face fluorescence spectroscopy, in: Greve J., Puppels G.J., Otto C. (Eds.), *Spectroscopy of Biological Molecules: New Directions*, Kluwer Acad. Publ., Dordrecht, The Netherlands, 1999, pp. 351–352.
- [9] Eck A., Gillis J.C. (Eds.), *Le fromage*. Lavoisier Tec & Doc, 3<sup>e</sup> édition, Paris, France, 1997, 891 p.
- [10] Genot C., Tonetti F., Montenay-Garestier T., Drapon R., Front-face fluorescence applied to structural studies of proteins and lipid-protein interactions of visco-elastic food products. 1- Designing of front-face adaptor and validity of front-face fluorescence measurements, *Sci. Aliments* 12 (1992) 199–212.
- [11] Genot C., Tonetti F., Montenay-Garestier T., Marion D., Drapon R., Front-face fluorescence applied to structural studies of proteins and lipid-protein interactions of visco-elastic food products. 2- Application to wheat gluten, *Sci. Aliments* 12 (1992) 687–704.
- [12] Grappin R., Lefier D., Dasen A., Pochet S., Characterising ripening of Gruyère de Comté: influence of timextemperature and salting conditions on eye and slits formation, *Int Dairy J.* 3 (1993) 313–328.
- [13] Hennequin D., Hardy J., Évaluation instrumentale et sensorielle de fromages à pâte molle, *Int Dairy J.* 3 (1993) 635–647.
- [14] Herbert S., Caractérisation de la structure moléculaire et microscopique de fromages à pâte molle. Analyse multivariée des données structurales en relation avec la texture, Thèse, École Doctorale Chimie Biologie de l'Université de Nantes, France, 1999, 118 p.
- [15] Herbert S., Riaublanc A., Bouchet B., Gallant D.J., Dufour E., Fluorescence spectroscopy investigations of acid- and rennet-induced milk coagulations of milk, *J. Dairy Sci.* 82 (1999) 2056–2062.
- [16] Hirsch R.E., Nagel R.L., Stopped flow front-face fluorometer: a prototype design to measure hemoglobin R-T transition kinetics, *Anal. Biochem.* 176 (1989) 19–21
- [17] Jolliffe I.T., *Principal Component Analysis*, Springer, New York, USA, 1986, 271 p.
- [18] Lavanchy P., Bérodié F., Zannoni M., Noël Y., Adamo C., Squella J., Herrero L., L'évaluation sensorielle de la texture des fromages à pâte dure ou semi-dure. Étude inter-laboratoires, *Lebensm. Wiss. Technol.* 26 (1993) 59–68.
- [19] Lawrence R.C., Gilles J., Creamer L.K., The relation between cheese texture and flavour, *N.Z. J. Dairy Sci. Technol.* 18 (1983) 175–190.
- [20] Lopez C., Influence de la nature de l'interface matière grasse/eau de laits reconstitués sur la cinétique de coagulation et les caractéristiques du coagulum, Stage de DEA, Université de Bordeaux, France, 1997, 42 p.
- [21] Marangoni A.G., Steady-state fluorescence polarization spectroscopy as a tool to determine microviscosity and structural order in food systems, *Food Res. Int.* 25 (1992) 67–80.
- [22] Marshall R.J., Combined instrumental and sensory measurement of the role of fat in food texture, *Food Qual. Preference* 2 (1991) 117–124.
- [23] Noël Y., Zannoni M., Hunter E.A., Texture of Parmigiano Reggiano cheese: statistical relationships between rheological and sensory variables, *Lait* 76 (1996) 243–254.
- [24] Noël Y., Ardö Y., Pochet S., Hunter E.A., Lavanchy P., Luginbuhl W., Le Bars D., Polychroniadou A., Pellegrino L., Characterisation of protected denomination of origin cheeses: relationships between sensory texture and instrumental data, *Lait* 78 (1998) 569–588.
- [25] Parker C.A., Apparatus and experimental methods, in: Parker C.A. (Ed.), *Photoluminescence of solutions with applications to photochemistry and analytical chemistry*, Elsevier, Amsterdam, The Netherlands, 1968, pp. 128–302.
- [26] Saporta G., *Probabilités – Analyse des données et statistique*, Technip edition, Paris, 1990, 165 p.