Mid-infrared spectrometry prediction of the cheese-making properties of raw Montbéliarde milks from herds and cheese dairy vats used for the production of Protected Designation of Origin and Protected Geographical Indication cheeses in Franche-Comté

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ABSTRACT

Franche-Comté is the primary producing region of Protected Designation of Origin cheeses in France. Normally, mid-infrared (MIR) prediction models for cheese-making property (CMP) traits are developed using individual bovine milks. However, considering the requests of all actors in the dairy sector, the present study aimed to assess the feasibility of MIR spectroscopy to develop CMP equations of Montbéliarde herd and dairy vat milks. For this purpose, 22 CMP traits were analyzed on samples collected in 2016 (half in February–March and half in May–June) from 100 commercial herds and 70 dairy vats (55 cheese dairies) located in Franche-Comté. These characteristics included 11 rennet coagulation traits and 8 lactic acidification traits measured in either soft cheese or pressed cooked cheese conditions and 3 laboratory curd yields. Models of MIR prediction for each of the 22 CMP traits were built using partial least squares regression with external validation by dividing the data set into calibration (70%) and validation (30%) sets. We confirmed that the variability of milk traits depends largely on the production scale and is higher for individual milk than for herd milk and even higher for vat milk. The best prediction models were obtained in herd milk samples for curd yields expressed in dry matter or fresh, with a coefficient of determination (R^2) in external validation of 0.78 and 0.77, respectively. As with individual milk, these traits are closely related to the gross composition of the milk

and therefore easier to predict by MIR spectroscopy. However, these curd yield traits were poorly predicted ($R^2 = 0.58$) in vat milk samples due to their lower variability. In herd milk samples, prediction models of other CMP traits were poorly accurate except for the ratio of the time to obtain a standard firmness to the rennet coagulation time in soft cheese or pressed cooked cheese conditions, which showed $R^2 > 0.66$ in external validation. Such trait is important in qualifying the behavior of milk during cheese production. Prediction models of other CMP traits for either herd or vat milk samples had poor accuracy, and further work is needed to improve their performance.

Key words: cheese-making property, mid-infrared spectroscopy prediction, Montbéliarde herd milk, dairy vat milk, Franche-Comté Protected Designation of Origin cheese

INTRODUCTION

Managing cheese-making properties (CMP) of milk is important for cheese-makers to secure quality and regularity in cheese production, especially for Protected Designation Origin (PDO) and Protected Geographical Indication (PGI) cheeses, where treatment of milk (e.g., standardization and heating) is limited or forbidden. The Franche-Comté region, located in eastern France, is the primary producing region of PDO cheeses in France (4 PDO cheeses and 1 PGI cheese). Among the 4 PDO cheeses, Comté, the main French PDO cheese (about 60,000 t), is a cooked pressed cheese, Morbier is an uncooked pressed cheese, Mont d'Or (or Vacherin du Haut-Doubs) is a soft cheese with a washed rind, and Bleu de Gex Haut-Jura is a blue-veined cheese. Gruyère, a PGI cheese related to Comté, is a pressed

Received August 23, 2019.

Accepted February 19, 2020.

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cooked cheese. Like many long-life mountain cheeses, Comté is a large cheese. Approximately 400 L of raw milk is needed to produce a Comté cheese wheel with an average final weight of 40 kg. The PDO cheese sector in Franche-Comté bands together producers, cheese-makers, and cheese ripeners. Local producers pool the milk from their herds in a shared processing unit, a small cooperative called a “fruitière,” to produce PDO and PGI cheeses from whole raw milk according to strict specifications. The average total volume of vats is about 3,500 L in a fruitière. This old cooperative system, dating back to the Middle Ages, to this day makes Comté a cheese with strong roots. Professionals in the sector raise questions about the effects of blending milks with different CMP and storage conditions on the properties of the mixture, which is processed into cheese. Studies on this subject are rare and focus on several specific features, such as the effect of mixing milks of different breeds (Bland et al., 2015a,b), normal and abnormal milks (O’Brien et al., 2001; Frederiksen et al., 2011), or milks from different calving seasons (Lin et al., 2017) as well as the effect of milk storage conditions (Summer et al., 2006; Malacarne et al., 2013; O’Connell et al., 2017). In practice, field studies are difficult, expensive, and time consuming when requiring gold standard methods. Few studies have investigated the variation of milk technological traits at a level other than the individual. These studies were mainly conducted at the herd level (De Marchi et al., 2007; Summer et al., 2014; Penasa et al., 2016) and some at the cheese vat level but in only a small number of commercial cheese factories (Malacarne et al., 2006; Summer et al., 2015).

For some years, mid-infrared (MIR) spectroscopy appeared to be an easy, fast, and cost-effective method for milk phenotyping in the dairy industry (De Marchi et al., 2014, 2018). Since 2015, the laboratory of the Trentingrana-Consorzio dei Caseifici Sociali Trentini s.c.a. (Trento, Italy) has adopted MIR spectroscopy to predict the coagulating ability of bulk milk samples routinely collected from each associated herd (Benedet et al., 2018). Over the past decade, numerous studies have been published on the ability of MIR spectroscopy to assess CMP traits of individual milk (Dal Zotto et al., 2008; De Marchi et al., 2009; Visentin et al., 2015), but studies on bulk milk are recent, rare, and limited. Benedet et al. (2018) used MIR predictions available on MilkoScan FT6000 (Foss, Hillerød, Denmark) to assess technological traits of herd bulk milk in their study, and Manuelian et al. (2019) developed MIR models to predict coagulation and acidity traits of sheep bulk milk. In a previous study (El Jabri et al., 2019), we examined the feasibility of using MIR spectroscopy to predict CMP (rennet coagulation, acidification by lactic bacteria, and laboratory curd yield, **CY**) of individual French

Montbéliarde milks (**I-milk**) from the Franche-Comté cheese production area, comparing partial least squares (PLS) and Bayesian methods. In our case, we obtained the best predictions using PLS regression. Considering the requests of all actors in the dairy sector, we conducted a study at 2 other production scales—herd tank milk (**H-milk**) and dairy vat milk (**V-milk**)—to evaluate the feasibility of developing CMP predictions of Montbéliarde H-milk and V-milk and to compare prediction results at different production scales.

MATERIALS AND METHODS

Sampling Criteria and Protocol

We selected and collected a range of PDO milks (H-milk and V-milk) as wide as possible. A total of 100 commercial herds were selected from an initial data set of 2,100 PDO and PGI herds of Franche-Comté. Herd selection was carried out to hedge the variability of the dairy farming practices in natural geographic location (plain, first plateau, second plateau, and mountain), calving season (autumn, late winter/early spring, summer), and average annual herd protein content based on the previous milking campaign data recording (<3.2, 3.2–3.3, 3.3–3.4, and >3.4%; Supplemental Table S1, <https://doi.org/10.3168/jds.2019-17491>). The milk-cooling tank of these 100 commercial herds was half sampled in February to March 2015 and half sampled in May to June 2015. The average lactation ranks and average calving months of the sampled herds were representative of those in the Franche-Comté PDO zone (Supplemental Figure S1, <https://doi.org/10.3168/jds.2019-17491>). Samples were collected from the tank containing 2 successive milkings, except on farms where the milk is delivered to the dairy after each milking (*lait de coulée*). In this case, the herd tank was sampled twice; namely, 2 samples of 2 consecutive milkings were collected and then mixed in proportion of each milking volume. Seventy dairy vats were sampled (before starter addition) at the same periods as herd tank milks in 57 dairy cooperatives located in the PDO cheese area, including 13 dairies that were collected in both periods. The milk delivered to the dairies was refreshed at 12°C for 12 to 24 h in accordance with the PDO specifications. Dairies were selected to maximize the expected milk composition variability in altitude of the collection area (<400, 400–700, 700–1,000, and >1,000 m) and dairy size (<10, 10–15, 16–25, and >25 milk producers; Supplemental Table S2, <https://doi.org/10.3168/jds.2019-17491>). Cows located in 216 single-breed commercial herds were selected according to their lactoprotein phenotypes and lactation stages to reflect the diversity of Montbéliarde milk composition

in Franche-Comté. A total of 250 I-milk samples were collected in 2016, half in January to March (indoor season) and half in April to June (grazing outdoor season), from the morning milking (2/5), the evening milking (2/5), or the mixture of 2 successive milkings in relative proportion (1/5; El Jabri et al., 2019). Animals, herds, and dairies were selected independently and in a manner that was representative across the 3 departments (Doubs, Jura, and Haute-Saône) of the Franche-Comté region.

Milk Analysis

Milk analyses, MIR spectra acquisition, and PLS calibration equation development were performed as previously described in detail for I-milk (El Jabri et al., 2019). In short, every representative sample of fresh raw whole milk was collected under strict hygienic conditions, immediately cooled to 4°C, divided into 2 subsamples, and analyzed within 24 h. One subsample was preserved with bronopol and analyzed by MIR spectroscopy using a MilkoScan FT6000 (Foss) and by Fossomatic FC counter (Foss) for SCC (Lacolait, Roulans, France). We recorded fat, protein, lactose, and urea contents; MIR spectra were stored. The second subsample was analyzed for total viable counts (cfu/mL), microbial quality (resazurin, natural lactofermentation), titratable acidity (**TA**), and coagulation and acidification properties (Enilbio, Poligny, France) according to the reference methods for soft cheese (**SC**) and pressed cooked cheese (**PCC**) technology as well as CY traits (INRAE, Poligny, France). Milk with SCC >10⁶/mL and cfu >10⁵/mL was discarded. Twenty-two CMP traits were measured, including 11 rennet coagulation traits, 8 lactic acidification traits, and 3 laboratory CY (El Jabri et al., 2019).

Microbial Quality Tests. We performed 2 tests commonly used to characterize the cheese-making value of milk from a microbiological viewpoint. The resazurin test is a colorimetric redox test in which milk and resazurin redox indicator are incubated in the dark at 37°C and reading is done after 4 and 6 h (6 = nondiscolored and 0 = totally discolored). The lactofermentation test is a natural fermentation test in which milk is incubated in the dark at 37°C and milk pH and aspect (e.g., liquid, homogeneous gel, flaky gel, contracted gel, swollen gel, or digested gel) are recorded after 24 h. It allows hypotheses to be made about the prevalence of spoilage microflora and the overall microbial load of milks and the acidification capacity of milks to be measured.

Coagulation Traits. Coagulation properties were measured with the Formoptic, adapted from a Formagraph (Foss Electric) by Chr. Hansen (Horshom, Den-

mark) and Enilbio (Poligny, France) to improve data computerization. In short, raw whole milk samples (10 mL) adjusted in pH (6.45 in SC conditions or 6.60 in PCC conditions) were coagulated at 32°C with Naturen rennet extract (Chr. Hansen, Arpajon, France) in SC conditions or Berthelot rennet extract (ABIA, Beaune, France) in PCC conditions. Both tests were performed in duplicate. With the Formoptic, an optical sensor measures the pendulum movement and converts this information into voltages. These measures are computerized, and a specific software program creates a diagram of firmness (expressed in firmness index, **FI**; FI = volts × 10) versus time (in min). From this diagram, we recorded the following coagulation traits:

1. rennet coagulation time to 0.5 FI for SC (**RCT_{SC}**) and PCC (**RCT_{PCC}**; min),
2. curd firmness after RCT for SC (**a_{SC}**) and PCC (**a_{PCC}**; FI units),
3. curd firmness at 2 times RCT for SC (**a_{2SC}**; FI units),
4. time to obtain 10 FI from RCT for SC (**K10_{SC}**) and PCC (**K10_{PCC}**; min),
5. the ratio of the time to obtain a standard firmness (**K10**) to RCT for SC (**K10/RCT_{SC}**) and PCC (**K10/RCT_{PCC}**), and
6. the curd organization speed (**TG10**; slope of the curve at firmness = 10 FI) for SC (**TG10_{SC}**) and PCC (**TG10_{PCC}**).

Acidity Traits. The CINAC system (Corrieu et al., 1988) was used to assess the acidification properties of milk by continuous measurement of pH for 20 h in either SC or PCC conditions. Flasks of raw whole milk (100 mL) were either inoculated (5.10⁶–10⁷ cfu/mL of milk) with a mesophilic starter (*Lactococcus lactis* ssp. *lactis*) and submitted to a specific SC time–temperature combination or inoculated (5.10⁶–10⁷ cfu/mL of milk) with a thermophilic starter (*Streptococcus thermophilus*) and submitted to a specific PCC time–temperature combination. We conducted the 2 tests in parallel on 2 systems. We recorded the following cheese-specific traits:

1. initial pH for SC (**pH_{0SC}**) and PCC (**pH_{0PCC}**; in pH units, **upH**),
2. acidification rate from 170 to 230 min for PCC (**AR_{170–230PCC}**; upH/h),
3. pH at 10 h for PCC (**pH_{10hPCC}**; upH),
4. time to reach a decrease of 0.08 upH from initial pH for SC (**TΔpH_{0.08SC}**; h),
5. time from pH 6 to pH 4.8 for SC (**TΔpH_{6–4.8SC}**; h),

6. decrease in pH from 16 to 20 h for SC ($\Delta\text{pH}_{16-20\text{hSC}}$; upH), and
7. pH at 20 h for SC ($\text{pH}_{20\text{hSC}}$; upH).

CY Traits. Finally, laboratory CY were measured in duplicate according to a method adapted from Hurtaud et al. (1993). A rennet extract (Berthelot, ABIA) was added to raw whole milk samples (50 mL in conical centrifuge tube at pH 6.6 and 32°C). One hour after rennet addition, the coagulum was cut and then centrifuged ($2,700 \times g$ for 15 min at 32°C). The supernatant (whey) was carefully recovered. Milk, curd, and whey were weighed. We determined the DM of the milk and the whey (NF V04-294 standard, 1996) and the protein content and fat content of the milk (MIR spectroscopy) and calculated the following CY:

1. fresh CY (CY_{FRESH} ; %): $\text{CY}_{\text{FRESH}} = 100 \times (\text{g of curd} / \text{g of milk})$;
2. CY in DM (CY_{DM} ; %): $\text{CY}_{\text{DM}} = 100 \times [1 - (\text{g DM of whey} / \text{g DM of milk})]$;
3. CY in fat and protein ($\text{CY}_{\text{FAT-PROT}}$; g/kg; the amounts of milk fat and protein required to obtain 1 kg of curd): $\text{CY}_{\text{FAT-PROT}} = (\text{protein content} + \text{fat content}) \times (\text{g of milk} / \text{g of curd})$.

MIR Spectra and Prediction Models

Mid-infrared spectra were collected over the spectral range from wavenumbers 5,011 to 925 cm^{-1} , standardized (Grelet et al., 2015), and cleared from spectra regions tainted by the water absorbance following Foss recommendations. Calibration equations were developed on 446 informative wavelengths for each of the 22 CMP traits using a PLS approach with external validation by dividing the data set into calibration (70%) and validation (30%) sets after outlier elimination. Outliers were discarded from reference data using the Grubbs test (Grubbs, 1969). The optimal number of PLS components ($\#L$) to incorporate in the model was defined by evaluating the root mean squared error of prediction based on cross-validation. We carried out PLS regression analyses on the H-milk sample data set ($n = 100$), on the V-milk sample data set ($n = 70$), then on the H-milk + V-milk sample data set ($n = 170$), and finally on the entire data set (all samples: I-milk + H-milk + V-milk samples, $n = 420$). To assess and compare the equations, statistical parameters were calculated: mean, standard deviation (SD), coefficient of determination (R^2), residual standard deviation ($S_{y,x}$), relative error ($\text{RS}_{y,x}$; ratio of the residual standard deviation to the reference mean, in %), bias (mean of differences between predicted and observed values), accuracy ($2 \times S_{y,x}$ corresponds to the accuracy

limits: 95% of residuals are between $\pm 2 \times S_{y,x}$; ISO, 2009), and ratio to performance deviation of external validation (RPD; ratio of SD of the reference data to standard error of prediction; El Jabri et al., 2019). We used R software (version 3.2.3., <https://r-project.org/>) for all computation. Equations with the lowest $\text{RS}_{y,x}$ and the highest R^2 and RPD values were regarded as being the most accurate (Coppa et al., 2010; Ferrand-Calmels et al., 2014). Coppa et al. (2010) used the R^2 value to define 4 classes of robustness: excellent ($R^2 \geq 0.91$), good ($0.82 < R^2 < 0.90$), approximate ($0.66 < R^2 < 0.81$), and poor ($R^2 < 0.66$). Equations with RPD > 2 enable predictions with good accuracy (De Marchi et al., 2013; Gottardo et al., 2015). Viscarra Rossel et al. (2006) defined more specifically 6 classes: excellent (RPD > 2.5), very good ($2 < \text{RPD} < 2.5$), good ($1.8 < \text{RPD} < 2$), fair ($1.4 < \text{RPD} < 1.8$), poor ($1 < \text{RPD} < 1.4$), and very poor (RPD < 1).

RESULTS AND DISCUSSION

Characterization of Milk Samples

Composition Traits. Table 1 summarizes the descriptive statistics for milk composition and CMP traits of I-milk, H-milk, and V-milk samples. The milk's average chemical composition and variability were consistent with the data from routine milk recordings at the same periods and for each production scale in the Franche-Comté region. Not surprisingly, the variability of these traits depended largely on the production scale and was highest for I-milk and lowest for V-milk. The coefficient of variation (CV) of all measured traits decreased rapidly from I-milk to H-milk and V-milk. Mean values for the composition traits were quite similar at all 3 production scales. This was especially true for protein, urea, pH, and TA, which averaged 3.23%, 26.6 mg/100 mL, 6.74, and 16.3°D, respectively. The average TA for H-milk was similar to that routinely collected on herds in Italy and in recent years (3.42–3.62°SH/50 mL equivalent to 15.4–16.3°D; Penasa et al., 2016; Benedit et al., 2018). Concerning PDO cheese V-milk, studies on Parmigiano-Reggiano V-milk from pure-breed herds (Italian Friesian or Italian Brown) reported for pH mean and SD values similar to ours (6.71–6.74, SD = 0.03–0.04) and for TA lower mean values (3.17–3.42°SH/50 mL or 14.3–15.4°D) with intermediate SD (0.11–0.15°SH/50 mL or 0.50–0.68°D; Malacarne et al., 2006; Summer et al., 2015). Differences in TA values between V-milks can be related to the cow breeds or the fact that Parmigiano-Reggiano is made of skim milk from the evening milking and whole milk from the morning milking. Fat and fat/protein ratio were on average slightly lower in H-milk and V-milk (3.74%

and 1.16 for H-milk; 3.67% and 1.14 for V-milk) than in I-milk (3.80% and 1.18). In contrast, SCS and total viable counts were higher in H-milk (3.51, 3.73 log cfu/mL) and V-milk (3.86, 4.44 log cfu/mL) than in I-milk (2.21, 3.38 log cfu/mL) but still at levels low enough to be considered good-quality milk, as confirmed by the resazurin and lactofermentation tests. The average value and SD for SCS and total viable counts in H-milk were of the same order or less than results reported in

milks collected in herds from other PDO cheese areas (Penasa et al., 2016).

CMP Traits. In all data ($n = 420$; not shown in Table 1), the CV for CY_{DM} , a_{SC} , and a_{PCC} averaged 8.0, 14.2, and 14.0%, respectively, but CV in H-milk were less than half those in I-milk (3.5, 7.0, and 7.2% vs. 9.9, 17.5, and 17.5%, respectively) and even lower in V-milk (2.4, 6.2, and 5.0%, respectively). The CY mean values in H-milk or V-milk, regardless of their mode of

Table 1. Descriptive statistics of composition and cheese-making property traits obtained by reference methods of individual ($n = 250$), herd tank ($n = 100$), and dairy vat ($n = 70$) milk samples

Trait ¹	Individual cow				Herd tank				Dairy vat			
	n ²	Mean	SD	CV (%)	n ²	Mean	SD	CV (%)	n ²	Mean	SD	CV (%)
Milk characteristics												
Fat (%)	250	3.80	0.76	19.92	100	3.74	0.21	5.64	70	3.67	0.13	3.56
Protein (%)	250	3.24	0.37	11.33	100	3.23	0.14	4.36	70	3.22	0.09	2.81
Fat:protein ratio	250	1.18	0.23	19.25	100	1.16	0.08	6.96	70	1.14	0.05	4.31
Lactose (%)	250	4.86	0.22	4.43	100	4.91	0.07	1.49	70	4.89	0.05	1.08
Urea (mg/100 mL)	250	26.58	7.88	29.65	100	26.59	7.40	27.84	70	26.64	4.22	15.86
pH	250	6.75	0.06	0.84	100	6.74	0.03	0.43	70	6.74	0.03	0.43
Titratable acidity (°D)	250	16.32	1.42	8.67	100	16.50	0.86	5.24	70	16.17	0.57	3.53
SCS ³	250	2.21	1.72	77.86	100	3.51	0.86	24.52	70	3.86	0.45	11.60
Total viable counts (log cfu/mL)	250	3.38	0.66	19.41	100	3.73	0.56	15.14	70	4.44	0.50	11.15
Resazurin test value 4 h after redox indicator addition	250	5.43	0.50	9.28	100	5.82	0.32	5.42	69	5.44	0.55	10.03
Resazurin test value 6 h after redox indicator addition	250	5.01	0.76	15.09	100	5.47	0.57	10.42	66	4.64	0.77	16.66
Lactofermentation test pH after 24-h raw milk incubation	250	5.82	0.64	11.02	100	5.20	0.51	9.83	70	4.44	0.25	5.58
Coagulation traits for SC at pH 6.45												
RCT _{SC} (min)	250	17.00	3.55	20.90	92	17.28	1.65	9.57	68	17.50	2.09	11.97
K10 _{SC} (min)	247	6.57	2.22	33.77	92	5.73	0.90	16.74	68	5.75	0.94	16.34
K10/RCT _{SC}	249	0.40	0.13	32.02	92	0.33	0.05	14.37	68	0.33	0.04	12.99
a _{SC} (FI)	250	18.79	3.29	17.49	92	19.44	1.37	7.04	68	19.58	1.22	6.23
a2 _{SC} (FI)	250	22.86	2.68	11.74	92	23.12	1.18	5.12	68	23.30	1.01	4.34
TG10 _{SC}	248	14.31	5.54	38.73	92	14.00	2.43	17.39	68	14.06	2.36	16.79
Coagulation traits for PCC at pH 6.60												
RCT _{PCC} (min)	249	31.86	7.29	22.86	100	31.01	2.43	7.83	68	31.80	3.28	10.30
K10 _{PCC} (min)	246	12.65	4.85	38.29	94	10.48	1.78	17.02	68	10.97	1.78	16.23
K10/RCT _{PCC}	248	0.40	0.13	31.79	96	0.34	0.05	14.90	68	0.34	0.04	10.63
a _{PCC} (FI)	249	18.42	3.22	17.47	96	18.98	1.37	7.22	68	18.96	0.95	5.00
TG10 _{PCC}	246	7.32	3.17	43.31	96	7.44	1.43	19.22	68	7.13	1.18	16.49
Acidification traits for SC												
pH _{0SC} (upH)	250	6.58	0.08	1.19	95	6.64	0.04	0.62	67	6.64	0.03	0.52
TΔpH _{0.08SC} (h)	248	3.48	0.28	8.12	95	3.48	0.34	9.79	66	3.45	0.37	10.85
TΔpH _{6-4.8SC} (h)	249	6.08	1.48	24.31	96	5.16	1.03	19.96	65	4.39	0.55	12.41
ΔpH _{16-20hSC} (upH)	250	0.20	0.15	75.34	94	0.13	0.07	49.54	64	0.08	0.02	28.31
pH _{20hSC} (upH)	248	4.49	0.09	2.05	96	4.50	0.07	1.54	65	4.45	0.04	0.88
Acidification traits for PCC												
pH _{0PCC} (upH)	250	6.53	0.06	0.91	91	6.55	0.04	0.55	64	6.58	0.03	0.53
AR _{170-230PCC} (upH)	250	0.12	0.05	37.86	92	0.12	0.03	20.63	62	0.13	0.02	14.39
pH _{10hPCC} (upH)	250	5.58	0.18	3.28	92	5.41	0.11	2.07	62	5.40	0.08	1.51
Curd yield												
Fresh curd yield (%)	250	38.00	9.37	24.67	100	36.06	3.63	10.06	70	34.10	2.43	7.12
Curd yield in DM (%)	250	66.79	6.62	9.91	100	65.71	2.31	3.51	70	64.64	1.57	2.43
Curd yield in fat and protein (g/kg)	250	191.02	26.40	13.82	100	194.90	15.65	8.03	70	202.64	12.25	6.05

¹SC = soft cheese; RCT = rennet coagulation time at 0.5 FI; FI = firmness index ($10 \times$ volts); K10 = time to obtain 10 FI from RCT; a = curd firmness after RCT; a2 = curd firmness at 2 times RCT; TG10 = curd organization speed; PCC = pressed cooked cheese; pH₀ = initial pH; upH = pH units; TΔpH_{0.08} = time to decrease by 0.08 upH from pH₀; TΔpH_{6-4.8} = time from pH 6 to pH 4.8; ΔpH_{16-20h} = decrease in pH from 16 to 20 h; pH_{20h} = pH at 20 h; AR₁₇₀₋₂₃₀ = acidification rate from 170 to 230 min; pH_{10h} = pH at 10 h.

²Number of samples after removing outliers.

³SCS = $\log_2$ (SCC/100,000) + 3.

expression (CY_{FRESH} , CY_{DM} , or $CY_{\text{FAT-PROT}}$), were lower than those in I-milk, although the curd firmness (a_{SC} or a_{PCC}) was slightly improved in H-milk and V-milk. This is most likely related to the small differences in milk fat recorded between production scales, which slightly and indirectly modify the proportion of protein content when analyzing a fixed volume of whole milk samples. We also noted that a_{SC} and a_{PCC} means and CV were similar at a given production scale. Indeed, because of our test conditions (i.e., adjustment of the milk pH to 6.45 for SC or 6.6 for PCC, followed by a measurement of coagulation traits at 32°C with the addition of specific rennet extracts for SC and PCC but an equivalent quantity of chymosin), variations in these traits depend primarily on milk composition (El Jabri et al., 2019). Other coagulation traits (RCT, K10, K10/RCT, and TG10) were half as variable in H-milk or V-milk as in I-milk. Overall, the rennet coagulation traits in H-milk showed less variation than that reported by Toffanin et al. (2012) in 1,570 bulk milk samples collected from 436 Italian dairy herds mainly rearing Holstein-Friesian cows. In this study, the CV of the usual milk coagulation traits [i.e., clotting time, curd firming time (k_{20}), and curd firmness 30 min after rennet addition (a_{30})] ranged from 20 to 30%, and 4% of total samples did not coagulate within 30 min. In our case, the conditions (pH standardization) and traits (curd firmness after RCT) for measuring rennet coagulation ability have contributed to the reduction of the variability of the traits. Moreover, variations in H-milk and V-milk traits are reduced due to the blending of milks from different cows and herds (Toffanin et al., 2012). However, K10 and TG10 were the most variable coagulation traits (CV >15%) regardless of the production scale. Malacarne et al. (2006) also reported large variability of k_{20} (CV = 15–23%) compared with clotting time (CV = 7%) and a_{30} (CV = 13–17%) in 13 comparisons of Parmigiano-Reggiano V-milk composed of a blend of milks from 2 to 5 pure-breed dairy herds (Italian Brown or Italian Friesian). The averages for K10 and K10/RCT were lower in H-milk and V-milk compared with I-milk in either SC or PCC conditions, suggesting that the proportion of poorly coagulating cow milks in bulk milks was very low (Frederiksen et al., 2011). As a whole, coagulation properties of H-milk and V-milk (with the exception of RCT) were slightly better and CY were somewhat poorer than those of I-milk. Average acidification traits in I-milk, H-milk, and V-milk samples were similar in PCC conditions. However, 2 kinetic traits in SC conditions, the time from pH 6 to 4.8 ($T\Delta pH_{6-4.8\text{SC}}$) and the decrease in pH from 16 to 20 h ($\Delta pH_{16-20\text{hSC}}$), had quite different average values depending on the production scales. On average, $T\Delta pH_{6-4.8\text{SC}}$ was 6.1, 5.2, and 4.4 h and $\Delta pH_{16-20\text{hSC}}$

was 0.20, 0.13, and 0.08 upH for I-milk, H-milk, and V-milk samples, respectively. These acidification kinetic traits in SC conditions and the acidification rate from 170 to 230 min ($AR_{170-230\text{PCC}}$) in PCC conditions were also much less variable in H-milk and V-milk samples compared with the individual ones. As indicated by the lactofermentation and resazurin tests, the latter observation seemed related to the presence of a native mesophilic lactic microflora. This one differed in quantity and quality in raw milk samples at the different production scales, whereas the difference in terms of total bacterial counts between I-milk and V-milk did not exceed 1 log unit, and TA was the same. When examining the types of lactofermentation obtained at each production scale, it was found that the proportion of homogeneous gelled milk (regarded as more suitable for cheese-making) was 0.57, 0.18, and 0.08, whereas that of liquid milk was 0.01, 0.66, and 0.79 for V-milk, H-milk, and I-milk, respectively (results not shown). This is partly due to the requirements for milk collection in the PDO area (12°C, 12–24 h), which allow the growth of mesophilic lactic bacteria as shown by the effects of storage conditions on microbial development of raw milk (Malacarne et al., 2013). On the other hand, these PDO requirements also preserve the coagulation traits as shown either by the average values we observed for the curd firmness and RCT traits or by the findings of other studies (Summer et al., 2006).

Performances of PLS Regression in External Validation

Table 2 provides the fitting statistics of the most valuable prediction models for CMP traits. To date, studies on the ability of MIR spectroscopy to predict CMP traits in herd bovine milk and dairy vat milk are scarce. We therefore discuss the results we obtained based on those published, if any, on herd bulk milk of other species or on bovine individual milk samples. Among H-milk traits, the best prediction accuracies were achieved in external validation for CY_{DM} ($R^2 = 0.78$, RPD = 2.07), CY_{FRESH} ($R^2 = 0.77$, RPD = 2.04), K10/RCT_{SC} ($R^2 = 0.73$, RPD = 1.88), and K10/RCT_{PCC} ($R^2 = 0.68$, RPD = 1.73). The calibration equations were built with an optimal number of PLS components lower for the K10/RCT ($\#L = 6$) than for the CY ($\#L = 10$ –11) and with the best $RS_{y,x}$ for CY_{DM} (1.3%). Considering the other H-milk traits, a_{SC} , RCT_{PCC}, a_{PCC} , and $CY_{\text{FAT-PROT}}$ showed R^2 and RPD in external validation close to 0.58 and 1.50, respectively. These accuracies were higher than the findings related to sheep bulk milk samples collected in 140 flocks in central Italy for RCT, k_{20} , and a_{30} ($0.02 < R^2 < 0.28$; $1.01 < \text{RPD} < 1.18$; Manuelian et al., 2019). They were

Table 2. Calibration and external fitting statistics of the best prediction models for the cheese-making properties of herd, dairy vat, and individual milk samples obtained from partial least squares regression^{1,2}

Item and trait ³	Data set ⁴	Calibration				External validation											
		n	#L	S _{y,x}	RS _{y,x} (%)	R ²	n	Mean	SD	Mean pred.	SD pred.	Bias	S _{y,x}	RS _{y,x} (%)	Accuracy	R ²	RPD
Herd milk samples (n = 100)																	
Coagulation SC																	
K10/RCT _{SC}	H	64	6	0.02	5.3	0.87 ⁵	26	0.34	0.05	0.33	0.05	-0.002	0.02	7.4	0.05	0.73	1.88 ⁵
a _{SC} (FI)	H	64	12	0.52	2.7	0.85 ⁵	27	19.47	1.45	19.39	1.35	-0.082	0.95	4.9	1.91	0.59	1.53
Coagulation PCC																	
RCT _{PCC} (min)	H	67	6	1.38	4.4	0.51	29	30.85	2.46	30.70	2.02	-0.147	1.65	5.3	3.30	0.57	1.49
K10/RCT _{PCC}	H	66	12	0.02	6.7	0.72	27	0.35	0.06	0.35	0.04	0.006	0.03	9.4	0.07	0.68	1.73
a _{PCC} (FI)	H	67	8	0.60	3.2	0.75	28	18.94	1.53	18.74	1.19	-0.202	1.02	5.4	2.04	0.57	1.50
Acidification PCC																	
AR _{170-230PCC} (upH)	H	63	7	0.02	13.1	0.64	28	0.12	0.02	0.12	0.02	-0.002	0.02	13.0	0.03	0.52	1.41
Curd yield																	
CY _{FRESH} (%)	H	67	10	1.29	3.6	0.82 ⁵	28	35.22	3.80	35.44	3.06	0.222	1.86	5.3	3.73	0.77	2.04 ⁵
CY _{DM} (%)	H	68	11	0.84	1.3	0.83 ⁵	28	65.26	2.51	65.40	1.82	0.133	1.21	1.9	2.42	0.78	2.07 ⁵
CY _{FAT-PROT} (g/kg)	H	69	7	8.87	4.6	0.62	29	199.57	16.12	195.47	11.45	-4.103	10.62	5.3	21.24	0.58	1.51
Dairy vat milk samples (n = 70)																	
Acidification PCC																	
AR _{170-230PCC} (upH)	V	44	7	0.01	8.4	0.72	17	0.13	0.01	0.13	0.02	0.008	0.01	6.1	0.02	0.71	1.78
Curd yield																	
CY _{FRESH} (%)	V	49	5	1.47	4.3	0.65	19	33.85	2.18	33.93	1.97	0.089	1.46	4.3	2.91	0.58	1.50
CY _{DM} (%)	V	49	4	0.98	1.5	0.64	20	64.57	1.35	64.52	1.47	-0.044	0.90	1.4	1.81	0.58	1.50
Herd milk + dairy vat milk samples (n = 170)																	
Coagulation SC																	
K10/RCT _{SC}	H+V	111	14	0.02	7.2	0.74	49	0.33	0.04	0.33	0.04	0.004	0.03	8.0	0.05	0.64	1.64
a _{SC} (FI)	H+V	111	13	0.73	3.8	0.71	49	19.56	1.20	19.51	1.22	-0.053	0.73	3.7	1.47	0.64	1.64
TG10 _{SC}	H+V	109	11	1.65	11.7	0.57	49	13.87	2.17	14.05	2.10	0.182	1.54	11.1	3.09	0.51	1.41
Coagulation PCC																	
a _{PCC} (FI)	H+V	113	6	0.79	4.2	0.58	49	19.08	1.10	19.03	0.97	-0.056	0.76	4.0	1.53	0.53	1.44
Acidification SC																	
pH _{0SC}	H+V	112	12	0.02	0.3	0.68	48	6.64	0.04	6.64	0.03	0.001	0.03	0.4	0.06	0.50	1.40
Acidification PCC																	
pH _{0PCC}	H+V	108	13	0.02	0.3	0.70	46	6.58	0.05	6.57	0.04	-0.006	0.03	0.5	0.06	0.55	1.47
AR _{170-230PCC} (upH)	H+V	107	11	0.02	9.3	0.73	45	0.12	0.02	0.12	0.02	0.001	0.02	13.0	0.03	0.55	1.47
Curd yield																	
CY _{FRESH} (%)	H+V	117	10	1.68	4.8	0.71	50	34.95	2.81	35.01	3.13	0.062	1.68	4.8	3.36	0.65	1.67
CY _{DM} (%)	H+V	117	10	1.01	1.6	0.75	50	65.04	1.94	65.05	2.00	0.013	1.11	1.7	2.22	0.68	1.75
All milk samples (n = 420)																	
Coagulation SC																	
K10/RCT _{SC}	All	281	14	0.05	13.8	0.76	115	0.37	0.11	0.38	0.09	0.010	0.07	19.1	0.14	0.62	1.61
a _{SC} (FI)	All	283	13	1.14	6.0	0.80	120	19.06	2.88	18.77	2.44	-0.293	1.50	7.9	3.00	0.73	1.92 ⁵
a2 _{SC} (FI)	All	283	13	1.01	4.4	0.76	119	23.06	2.24	22.76	1.91	-0.299	1.34	5.8	2.68	0.64	1.67
Coagulation PCC																	
K10/RCT _{PCC}	All	281	12	0.05	13.8	0.74	118	0.37	0.10	0.39	0.09	0.013	0.06	16.3	0.12	0.62	1.62
a _{PCC} (FI)	All	283	12	1.16	6.2	0.79	121	18.67	2.65	18.40	2.41	-0.275	1.41	7.5	2.81	0.72	1.88 ⁵

Continued

Table 2 (Continued). Calibration and external fitting statistics of the best prediction models for the cheese-making properties of herd, dairy vat, and individual milk samples obtained from partial least squares regression^{1,2}

Item and trait ³	Data set ⁴	Calibration				External validation											
		n	#L	S _{y,x}	RS _{y,x} (%)	R ²	n	Mean	SD	Mean pred.	SD pred.	Bias	S _{y,x}	RS _{y,x} (%)	Accuracy	R ²	RPD
Acidification PCC																	
pH _{0PCC}	All	283	17	0.03	0.5	0.68	117	6.55	0.06	6.55	0.04	−0.001	0.03	0.5	0.07	0.65	1.68
Curd yield																	
CY _{FRESH} (%)	All	283	13	2.96	8.0	0.86 ⁵	122	36.12	7.21	36.31	6.18	0.191	2.74	7.6	5.49	0.86 ⁵	2.63 ⁵
CY _{DM} (%)	All	283	13	1.68	2.5	0.91 ⁵	123	65.74	5.10	65.82	4.69	0.08	1.67	2.5	3.34	0.89 ⁵	3.05 ⁵
CY _{FAT-PROT} (g/kg)	All	283	18	13.50	7.0	0.64	124	194.82	21.24	194.56	17.66	−0.252	14.41	7.4	28.83	0.54	1.47

Acidification PCC																	
pH _{OPCC}	All	283	17	0.03	0.5	0.68	117	6.55	0.06	6.55	0.04	-0.001	0.03	0.5	0.07	0.65	1.68
Curd yield	All	283	13	2.96	8.0	0.86 ⁵	122	36.12	7.21	36.31	6.18	0.191	2.74	7.6	5.49	0.86 ⁵	2.63 ⁵
CY _{FRESH} (%)	All	283	13	1.68	2.5	0.91 ⁵	123	65.74	5.10	65.82	4.69	0.08	1.67	2.5	3.34	0.89 ⁵	3.05 ⁵
CY _{DM} (%)	All	283	18	13.50	7.0	0.64	124	194.82	21.24	194.56	17.66	-0.252	14.41	7.4	28.83	0.54	1.47

¹Not shown: equations with R² < 0.5 in external validation.²n = number of samples after removing outliers; #L = number of partial least squares components; S_{y,x} = residual standard deviation; RS_{y,x} = relative error; R² = coefficient of determination in calibration (or in external validation); Bias = mean of differences between predicted (pred.) and observed values; Accuracy = accuracy limits; RPD = ratio performance deviation.³SC = soft cheese; RCT = rennet coagulation time at 0.5 FI; FI = firmness index (10 × volts); K10 = time to obtain 10 FI from RCT; a = curd firmness after RCT; PCC = pressed cooked cheese; AR₁₇₀₋₂₃₀ = acidification rate from 170 to 230 min; CY_{FRESH} = fresh curd yield; CY_{DM} = curd yield in DM; CY_{FAT-PROT} = curd yield in fat and protein; TG10 = curd organization speed; pH₀ = initial pH; a2 = curd firmness at 2 times RCT.⁴H = herd milk samples; V = dairy vat milk samples; All = all milk samples = individual milk samples + herd milk samples + dairy vat milk samples.⁵Best robustness or accuracy (good to excellent).

much better for K10/RCT_{SC} (R²: +0.25 points; RPD: +0.50 points), K10/RCT_{PCC} (R²: +0.24 points; RPD: +0.40 points), and RCT_{PCC} (R²: +0.28 points; RPD: +0.31 points), with RS_{y,x} values reduced by half or more compared with the predictions we previously obtained on I-milk in identical analytical conditions (El Jabri et al., 2019). An explanatory element could be a greater homogeneity of the fine composition (i.e., minerals, caseins) of H-milk due to the blending of individual milks. For other CY and coagulation traits, accuracies of MIR predictions on H-milk compared with those found on I-milk were slightly better but still poor for CY_{FAT-PROT} and were lower for a_{PCC}, CY_{FRESH}, and CY_{DM} but remaining quite good for both CY (in calibration: R² = 0.82–0.83; in external validation: R² = 0.77–0.78 and RPD > 2.0). In good-quality milk, these traits are mainly correlated with the gross composition of the milk, which is less variable in H-milk and V-milk samples. The best predictions in V-milk traits were achieved for only a few traits: AR_{170-230PCC} (R² = 0.72 with #L = 7 in calibration; R² = 0.71 and RPD = 1.78 in external validation), CY_{DM}, and CY_{FRESH} (R² = 0.64–0.65 with #L = 4–5 in calibration; R² = 0.58 and RPD = 1.50 in external validation). For the first trait, however, the MIR predictive ability was fair. For the last 2 traits, the accuracy of the equations was poor and as low as that of CY_{FAT-PROT} in H-milk. The number of samples collected at the dairy vat scale and their reduced variability explain the difficulty of building MIR predictions. By pooling H-milk and V-milk samples (n = 170), we observe a general loss of accuracy of the equations compared with that obtained with the H-milk samples alone. For milk blends, it therefore appears necessary to take into account their intrinsic characteristics, which are partly related to their thermal history. The pH_{OSC} and pH_{OPCC} traits reached R² values close to 0.7 in calibration as a result of the increased number of data, but R² values in external validation were not better than 0.55 and 0.50, respectively. A rather similar observation can be made for the curd organization speed for SC, a coagulation trait measuring the curd organization rate as K10/RCT_{SC} but with less accuracy. Finally, by pooling all the data (H-milk + V-milk + I-milk, n = 420), we obtained the most robust and accurate equations in external validation for CY_{DM} (R² = 0.89; RPD = 3.05), CY_{FRESH} (R² = 0.86; RPD = 2.63), a_{SC} (R² = 0.73; RPD = 1.92), and a_{PCC} (R² = 0.72; RPD = 1.88). These 4 CMP traits are among those most closely related to the gross milk composition, and their MIR predictions were calibrated with the most reliable reference methods among those used in this study (El Jabri et al., 2019). By increasing the number of data for these particular traits, we obtained prediction performances in external validation very similar to those obtained previously on

250 I-milk samples for CY_{DM} (R^2 : -0.02 point; RPD: -0.22 point) or even a little better for CY_{FRESH} (R^2 : $+0.03$ point; RPD: $+0.27$ point), a_{SC} (R^2 : $+0.06$ point; RPD: $+0.19$ point), and a_{PCC} (R^2 : $+0.09$ point; RPD: $+0.26$ point; El Jabri et al., 2019). Figure 1 shows the scatter plots of CY_{DM} , CY_{FRESH} , a_{SC} , and a_{PCC} and reports the R^2 values obtained with the calibration equation built on all milk samples ($n = 420$). These plots confirmed that the variability in H-milk and V-milk was included within the variability measured in

I-milk. Finally, for several traits, we recalculated MIR predictions using for I-milk the calibration equations based on all milk samples ($n = 420$) and for H-milk or V-milk the calibration equations based on H-milk + V-milk samples ($n = 170$). On I-milk, we obtained for 6 CMP traits (CY_{DM} , CY_{FRESH} , a_{PCC} , a_{SC} , a_{2SC} , and pH_{0PCC}) R^2 between 0.93 and 0.66, with RPD between 3.73 and 1.72. On H-milk, R^2 values were poor for all traits (<0.66), and on V-milk, CY_{DM} was the only trait reaching fair prediction ($R^2 = 0.68$, with RPD = 1.75).

Figure 1. Scatter plots of reference (y-axis) versus predicted (x-axis) values for the best-predicted cheese-making traits using a partial least squares model built on all milk samples. (a) Fresh curd yield (CY_{FRESH} ; %); (b) curd yield in DM (CY_{DM} ; %); (c) curd firmness after rennet coagulation time in pressed cooked cheese conditions of rennet coagulation [a_{PCC} ; firmness index (FI)]; (d) curd firmness after rennet coagulation time in soft cheese conditions of rennet coagulation (a_{SC} ; FI). FI = volts $\times$ 10. Green = individual milk samples; black = herd milk samples; pink = dairy vat milk samples; solid red line = regression line; dashed blue line = first bisector.

The implementation of an equation developed on milk samples with high variability on a population of milk samples with low variability (H-milk, V-milk) leads to a reduction in performance because the variability of the validation set is reduced compared with the performances of the calibration model. For this reason, the equations should be specific to each scale of production. Furthermore, in the specific case of V-milk or H-milk samples, we are not convinced that increasing the number of samples when most of the variability is already trapped can significantly increase the performance of the equations.

CONCLUSIONS

This study is the first to evaluate by MIR spectroscopy the CMP of Montbéliarde milks not only at the individual-scale milk production but also at the herd tank- and dairy vat-scale milk production to fulfil the expectations of cheese-makers. We confirm that the accuracy of the models depends on the number of samples and the variability of their characteristics, which were higher for I-milk samples than for H-milk samples and higher for H-milk samples than for V-milk samples. For most CMP traits, the prediction models developed either on the entire data set (this study) or on the individual data set (El Jabri et al., 2019) have shown similar accuracy, meaning that they could be used equally for genetic purposes. We already successfully used the prediction models developed on all samples of milks ($n = 420$) to assess genetic variability of CMP within the population of Montbéliarde cows, confirm candidate causative variants on CMP, or reveal new genes co-associated with milk CMP and milk composition in this population (Sanchez et al., 2018a,b, 2019). The relationships between the fine physicochemical composition of the milks and their CMP will be studied at each production scale. Another objective of this project is to identify factors contributing to the CMP variation at different scales of the PDO and PGI milk production system (cow, herd, and dairy cooperative) by using MIR spectroscopy prediction methods when their accuracy is acceptable or, alternatively, reference analytical methods.

ACKNOWLEDGMENTS

This study was funded by the French Ministry of Agriculture, Agro-Food and Forest (Paris, France), the French Dairy Interbranch Organization (CNIEL, Paris, France), the Regional Union of Protected Designation cheeses of Franche-Comté (URFAC, Poligny, France), and the Regional Council of Bourgogne Franche-Comté

(Besançon, France) under the project FROM'MIR. The authors have not stated any conflicts of interest.

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